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Hydrodynamic Cavitation and Landfill Leachate Treatment Optimization for the Removal of Organic and Inorganic Contaminants

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I. List of publications

1. Moftakhari Anasori Movahed, S., Calgaro, L., Marcomini, A., 2023. *Trends and characteristics of employing cavitation technology for water and wastewater treatment with a focus on hydrodynamic and ultrasonic cavitation over the past two decades: A Scientometric analysis*. Science of The Total Environment 858, 159802. <https://doi.org/10.1016/j.scitotenv.2022.159802>
2. Moftakhari Anasori Movahed, S., Calgaro, L., Marcomini, A., 2025. *Water-Related Life Cycle Assessment Applications Over the Past Decade: A Scientometric Analysis*. Accepted with minor revisions at Heliyon.

Manuscripts in preparation, submission expected by July 2025.

3. Moftakhari Anasori Movahed S., Calgaro L., Ioannou G., Katsogiannis I., Marcomini A. *Development and testing of a nanobubble-based hydrodynamic cavitation system*.
4. Moftakhari Anasori Movahed S., Calgaro L., Scanferla P., Pelay, R., Marcomini A. *Developing and testing of a pilot scale plant for ammonia and PFAS removal from landfill leachate*.

II. Acronym list

AOP	Advanced Oxidation Processes
API	Active Pharmaceutical Ingredients
BOD	Biological Oxygen Demand
CEC	Contaminants of Emerging Concern
COD	Chemical Oxygen Demand
CR	Congo Red
DoE	Design of Experiment
DLS	Dynamic Light Scattering
EC	Emerging Contaminants
HA	Humic Acid
HC	Hydrodynamic Cavitation
HPSS	High Performance Solidification/Stabilization
ICP-MS	Inductively Coupled Plasma Mass Spectrometry
ICP-OES	Inductively Coupled Plasma Optical Emission Spectrometry
LCA	Life Cycle Assessment
NF	Nanofiltration
PAH	Polycyclic Aromatic Hydrocarbons
PCP	Personal Care Products
PFC	Perfluorinated Compounds
PFAS	Per- and polyfluoroalkyl substances
PMT	Persistent, Mobile, and Toxic
PPP	Plant Protection Products
PS	Persulfate
RR120	Reactive Red 120
RO	Reverse Osmosis
TOC	Total Organic Carbon
TT	Thermal Treatment
UF	Ultrafiltration
UV	Ultraviolet
UV-Vis	Ultraviolet–Visible spectroscopy
VCR	Volumetric Concentration Ratio
vPvM	very Persistent and very Mobile
WWTP	Waste Water Treatment Plant

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IV. Abstract

Several technologies have proven effective for the treatment of water, wastewaters, and leachates, yet various limitations have been identified regarding the removal of specific classes of contaminants and the excessive use of energy and reagents.

The first part of this thesis' experimental work focused on constructing a hydrodynamic cavitation system that utilizes a Venturi tube specifically designed to promote the formation of nanobubbles. Ten prototypes were developed following a Design of Experiment (DoE) approach and tested with a cavitation device connected to a laser particle sizer to measure the bubbles' characteristics. The best one with a throat diameter of 1mm, throat length of 9.85mm, divergence length of 67.15mm and a divergence angle of 1.96° was selected and built in stainless steel. This Venturi tube was able to produce nanobubbles with an average diameter of 151nm, frequency of 89%, and Zeta potential of -15.4mV. The performance of the cavitation system was investigated for the degradation of Humic Acid (HA) and two azo-dyes (i.e., Congo Red (CR) and Reactive Red 120 (RR120)). The results showed that the highest degradation efficiencies were achieved for CR (93%) and RR120 (95%) using persulfate, while HA exhibited the best degradation (66%) with hydrogen peroxide. Additionally, the presence of salt reduced the degradation for all substances by approximately 10–20%.

The second part of the work involved developing a multi-phase process for treating landfill leachate containing ammonia, metals, and PFAS. This process integrated steps of (i) biological oxidation of organic material, (ii) anaerobic biological reduction of nitrogen compounds, (iii) membrane filtration, (iv) physico-chemical treatment of the membrane concentrate, (v) solidification/stabilization and thermal treatment of the produced sludge, and (vi) salt recovery using reverse osmosis and concentration under vacuum. During each phase, the presence of contaminants was studied using various analytical techniques (UV-Vis, ICP-MS, HPLC-MS, IC), showing that i) all PFAS and metals were below the regulatory limits in the final water discharge; ii) the produced salt complied with regulatory standards for use as road salt; and iii) PFAS were still present in the leachate from the stabilized granular material, suggesting that further treatment of this material will be needed.

The third part of this thesis focused on two bibliometric studies regarding research trends and gaps on cavitation applied for water and wastewater treatment and on the application of Life Cycle Assessment in the field of water use and management.

The overall activity led to four manuscripts that are discussed in detail in this thesis.

V. Objectives and thesis structure

The main objective of this thesis work was to investigate the use of several water treatment technologies for the removal of organic and inorganic contaminants from water, wastewater, and landfill leachate. In particular, the work focused on 1) investigating the performance of a hydrodynamic cavitation system using a Venturi tube specifically designed to favor the formation of nanobubbles to treat water and wastewaters containing natural organic matter and two widely used azo-dyes and 2) developing a process for treating landfill leachate containing ammonia, metals, and PFAS by integrating steps of (i) biological oxidation of organic material, (ii) anaerobic biological reduction of nitrogen compounds, (iii) membrane filtration, (iv) physico-chemical treatment of the resulting water, and (v) stabilization and thermal treatment of the produced sludge.

The main questions asked are:

1. What are the research trends and gaps in cavitation applied for water and wastewater treatment?
2. What are the emerging trends and existing gaps in applying life cycle assessment (LCA) to water use and management?
3. What are the optimal Venturi tube design parameters to favor the generation of nanobubbles with the smallest diameter and the lowest Z-potential? How does a hydrodynamic cavitation system employing the optimized Venturi tube perform for the degradation of organic compounds?
4. Which is the best combination of processes to treat a landfill leachate characterized by high levels of ammonia and PFAS, in order to reduce the post-disposal impact of municipal solid waste?

The thesis is made up of six chapters, the first of which is an introduction to the topics covered, while the other chapters explore in detail the investigated processes and the use of scientometric review to investigate research trends and gaps on the selected topics. Specifically:

Chapter 1 briefly describes the main anthropogenic activities and natural phenomena that cause water scarcity worldwide, with a focus on contaminants of emerging concern (CECs) and their main pathways into the environment (e.g., landfill leachate and wastewaters) leading to water resources' contamination. Then, an overview of the main wastewater treatment technologies employed to remove CECs from waters and wastewaters is presented, with a specific focus on hydrodynamic cavitation, its mechanisms, and its potential advantages in removing organic and inorganic

contaminants. This general introduction concludes with an overview of wastewater treatment interferences, especially natural organic matter (NOM).

Chapter 2 reports the materials, instruments, and methods common to the works discussed in this thesis.

Chapter 3 reports research work aimed at investigating the trends and gaps on the topic of employing cavitation in water and wastewater treatment processes by means of scientometric analysis. The work aimed at identifying the most investigated topics and providing insights on future directions in this field which was used to better understand and optimize the hydrodynamic cavitation system employed in the experimental part of the thesis.

Chapter 4 outlines a scientometric study focused on Life Cycle Assessment (LCA) and its implementation on any technology or process for the treatment of water and wastewater, as well as to any water use (or reuse) to produce food, goods, energy, or services. The study outlines the most important innovations of the last decade, as well as the most investigated topics to assess future research directions.

Chapter 5 describes the investigation on the design of a Venturi tube specifically tailored to promote the formation of nanobubbles with low Z-potential and its use in a hydrodynamic cavitation system to degrade organic substances. The selected substances were humic acid as proxy for NOM and two azo-dyes (i.e., Congo Red and Reactive Red 120), and the system was tested with and without the addition of oxidants (i.e., hydrogen peroxide and sodium persulfate) and salt (i.e., NaCl). Furthermore, a neural network model was developed to investigate further configuration options other than those originally tested, with the aim of further optimize the nanobubbles' characteristics (i.e., average diameter, frequency, and Z-potential).

Chapter 6 reports on the development of an integrated, multi-stage process to treat a landfill leachate characterized by high levels of ammonia and PFAS, as well as by the presence of metals and other organic substances. A dedicated process was also developed to address the management of contaminated sludge from the treatment of nanofiltration concentrated fraction using the High-Performance Solidification/Stabilization process, with the aim to obtain a reusable cementitious granular material.

1. Chapter 1: Introduction

1.1 Water pollution and scarcity

Water covers over 75 percent of the earth's surface; however, only a small portion is fit for human consumption. In fact, only 2.5% of the water on Earth is freshwater, with the remaining 97.5% being salty. Furthermore, about 69% of freshwater is trapped in polar glaciers, and about 30% is groundwater, which is difficult and expensive to extract [1]. Finally, only ca.1.2% of freshwater can be found as surface water and is easily accessible.

In addition, the limited amount of accessible water is at risk. Several factors, such as the development of water-intensive industrial sectors (e.g., pharmaceuticals, plastic, and textile production) [2,3], climate change, and increasing urban population have put great strains on water resources [4]. Therefore, maintaining drinking water quality is vital since it is directly correlated with human health, and chemical exposure can cause short- and long-term effects [5]. In particular, the World Health Organization has estimated that approximately 10 percent of the population around the globe is not able to benefit from safe drinking water sources [6]. Simultaneously, it is also fundamental to ensure a safe and stable provision of water for industrial activities [7].

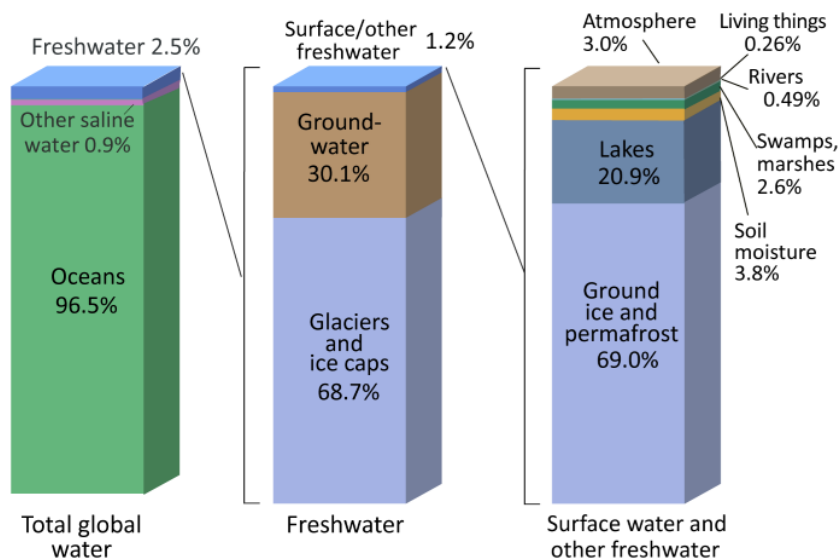


Figure 1.1: Water distribution on earth [1].

The increasing strain on water resources, driven by industrial expansion, urbanization, and the emergence of new contaminants, is further exacerbated by the effects of climate change, which intensify water scarcity, particularly in dry regions.

In detail, climate refers to the typical weather conditions, such as temperature, precipitation, and humidity, averaged over a particular time frame and location [8]. Climate change refers to the challenging-to-reverse alterations in an area's climatic elements when compared to the typical climate patterns observed over an extended period [9,10]. The global population residing in water-scarce regions has grown from 2.04 billion in 2010 to 2.36 billion in 2020, with projections suggesting it

could reach 2.70 billion by 2030 [11]. Research shows that the impacts of climate change and water scarcity vary across nations. In developing countries, where livelihoods heavily rely on natural resources like water, soil, and pastures, communities are particularly sensitive and vulnerable to these challenges compared to those in developed nations [12,13].

The effects of climate change, including abnormal precipitation patterns and rising temperatures, directly contribute to water scarcity and intensify the competition for limited supplies, as shown in Figure 1.2.

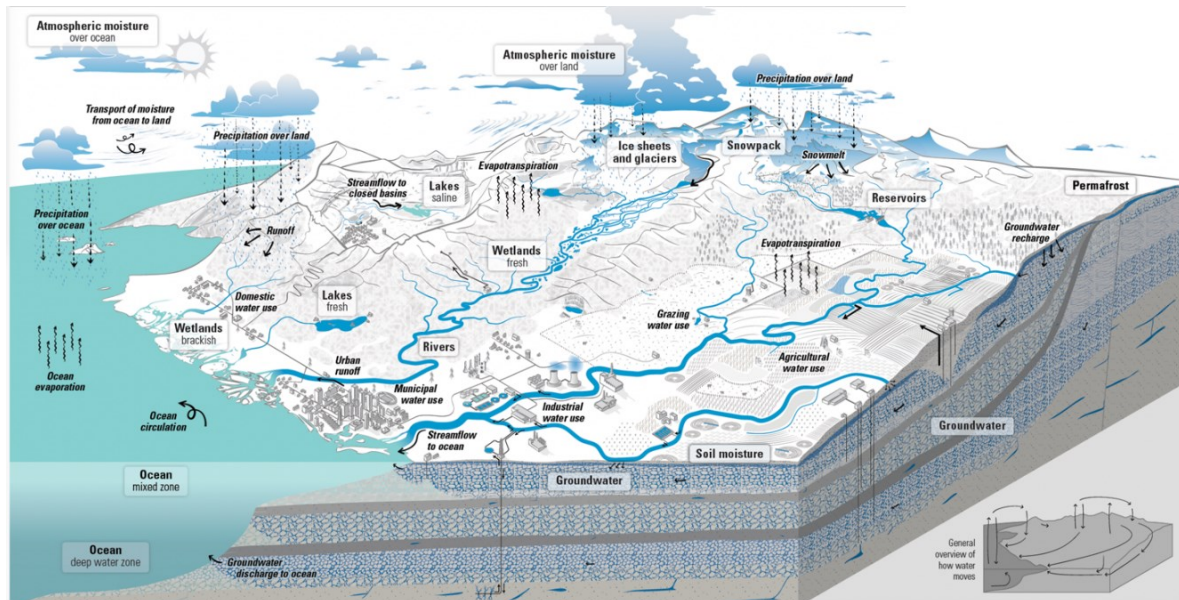


Figure 1.2: Hydrological cycle and its main changes due to climate change [14].

In particular, water scarcity refers to a situation where water demand surpasses the available supply. A region is considered to experience "water scarcity" when clean, natural water availability falls below 1,000m³ per person annually [15,16]. Unlike issues affecting a single individual or location at a specific time, water scarcity impacts larger populations across geographic regions (e.g., countries) over extended periods (months or years). For example, due to the climate change effects, Southern and Western European countries are particularly impacted by persistent droughts, exacerbated by recurrent heat waves during the hottest months of the year [17,18] as shown in Figure 1.3.

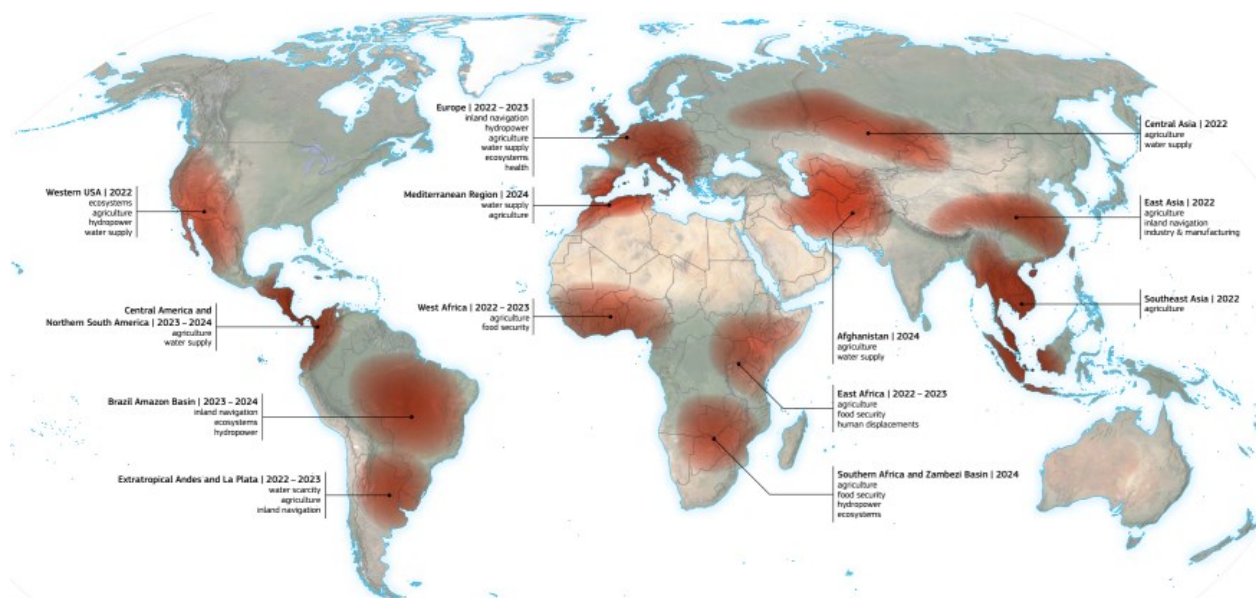


Figure 1.3: World map drought atlas for 2024 considering agriculture, water supply, ecosystems, health, industry and manufacturing, water scarcity, hydro power, inland navigation, and food security as the factors involved [19].

Water scarcity issues can be linked to both some climate-related phenomena and other anthropogenic factors [20], such as:

1. Extreme hydrological events: heavy rainfall and droughts can disrupt agricultural production and damage infrastructures, thus posing a risk to food availability, human lives, and ecosystems.
2. Permafrost thawing: rising temperatures melt frozen ground, releasing methane and accelerating climate change. This destabilizes landscapes, damages infrastructure, and disrupts water flows, worsening water scarcity by altering groundwater availability and freshwater supplies.
3. Increasing urbanization: the development of new urban areas increases clean water demand while at the same time contributing to the further degradation of available water resources due to wastewater discharges.
4. Water loss due to inefficiency: outdated and inefficient water infrastructure (e.g., leaky pipes, outdated systems, and poor maintenance) can lead to the loss of up to 30% of treated and drinking water.
5. Water mismanagement: poor water management, overuse, and inefficiency due to unsupervised practices that waste large quantities of water can cause marked water resource depletion, especially in agricultural, industrial, and domestic activities.
6. Water quality degradation: Excessive use of fertilizers and pesticides, soil depletion, and unregulated waste disposal can contaminate water supplies, thus making them unsafe for consumption and agricultural use.

7. Groundwater depletion: excessive extraction at unsustainable rates is depleting the groundwater resources, creating a hidden and escalating global threat. The groundwater from deep aquifers provides 30% of the world's freshwater for farming, daily consumption, and industrial use. Therefore, excessive use can deplete a vital source of fresh water, leading to more water scarcity.
8. Competing over water resources: when water resources are limited and not enough to meet all needs, it creates a growing competition that leads to higher costs, strained international relations, and, in some cases, conflicts over water rights.

In detail, water scarcity affects the availability of safe drinking water, leading to increased reliance on contaminated sources and increasing the risk of waterborne diseases, as drinking water quality is directly linked to human health [6]. In particular, contaminants such as pathogens, chemicals, and metals can cause various health issues, including gastrointestinal infections, neurological disorders, and cancers [7].

However, the problem of water scarcity is not limited to drinking water use. Water scarcity is a critical issue extending far beyond drinking water, significantly affecting various sectors heavily reliant on water. Agriculture, which accounts for approximately 70% of global freshwater usage [21,22], is among the most vulnerable sectors. In regions such as the Middle East and North Africa, projections suggest that water scarcity could lead to a 60% reduction in agricultural output by 2050, with serious implications for food security [23]. Similarly, the textile industry, particularly cotton production, faces growing risks due to its dependence on large volumes of water. In Pakistan, one of the leading cotton producers, diminishing water resources threaten the sustainability of the textile sector [24]. Livestock farming is also deeply impacted, as water shortages affect the availability of quality feed and pasture, reducing productivity and posing economic challenges for farmers. These examples demonstrate how water scarcity affects essential industries, highlighting the urgent need for integrated water resource management to ensure long-term sustainability and resilience [25].

Within this context, water reuse (e.g., greywater recycling, rainwater harvesting, and irrigation with reclaimed wastewater) has been shown as a viable alternative, using several technologies and management measures to minimize water loss and guarantee the necessary water quality for the intended use. In detail, among the developed technologies, each offers unique advantages while presenting certain limitations; therefore, their challenges must be carefully addressed to ensure sustainable and effective implementation [26–29]. For example, water desalination provides a reliable source of freshwater from seawater, ensuring supply in arid regions and during droughts. However, it is energy-intensive, costly, and poses environmental risks due to brine disposal [30]. Drip irrigation systems enhance water use efficiency by delivering water directly to plant roots, reducing evaporation

losses, but high setup and maintenance costs can be barriers for some farmers. Greywater recycling conserves freshwater by reusing water for non-potable purposes and reduces wastewater discharge, yet it may pose health risks if not treated properly and can face public acceptance challenges. Rainwater harvesting captures runoff, providing an independent water source and mitigating flooding, but its effectiveness can be limited by storage capacity and rainfall variability. Constructed wetlands naturally treat wastewater while supporting biodiversity and requiring low energy inputs, though they need substantial land and time to reach full efficiency. Smart water management systems use sensors and data analytics to optimize water distribution and detect leaks, improving efficiency, but they involve high implementation costs and require technical expertise. Salt-tolerant agriculture expands irrigation possibilities using saline water and enables farming on marginal lands; however, the development and adoption of such crops can be lengthy and subject to regulatory hurdles.

In order to address these challenges, the European Union has taken several legislative measures, such as the Drinking Water Directive (2020/2184/EC), the Groundwater Directive (2006/118/EC), the Water Framework Directive (WFD) (2000/60/EC), and the Marine Strategy Framework directive (MSFD) (2008/56/EC) aiming to ensure both the environment and human health protection. However, while this legislation measures the identity of several pollutants of concern, other chemicals are continuously introduced and detected in the environment, requiring continuous evaluation of the risks they pose.

1.2 Contaminants of emerging concern

Contaminants of emerging concern (CECs) are synthetic or natural chemicals and microorganisms that are rarely monitored but may pose risks to environmental and human health. They are termed "emerging" due to increasing awareness of their harmful effects, new exposure pathways, or the need for novel treatment methods [31–35].

Contaminants of emerging concern (CECs) encompass a broad range of chemical substances, including active pharmaceutical ingredients (APIs), personal care products (PCPs), plant protection products (PPPs), industrial additives, perfluorinated compounds (PFCs), disinfection byproducts, and engineered nanomaterials, as shown in Figure 1.4 [36].

Emerging contaminants reach the environment through multiple pathways, including municipal and industrial wastewater discharge, hospital effluents, landfill leachates, agricultural runoff, accidental leakage, sewer overflows, and direct discharge from agricultural and aquaculture activities, as shown in Figure 1.5. It should be noted that wastewater treatment plants (WWTPs) play a crucial role in managing these pollutants, yet they are not always effective in fully removing them.

These pollutants enter the environment through various pathways and interact with different environmental media, including soil, water, and air. These contaminants enter surface and

groundwater systems, potentially contaminating drinking water supplies and posing risks to human and ecosystem health. Pharmaceutical residues, veterinary drugs, pesticides, and industrial byproducts are among the most commonly detected pollutants in these environmental compartments [37,38]. Due to their high mobility and persistence, they can also infiltrate the soil, which can lead to long-term contamination, affecting microbial communities and plant life, while their presence in water raises concerns over aquatic toxicity and bioaccumulation in living organisms [39]. Additionally, sediments can act as a secondary pollution source, leading to the resuspension and reintroduction of these pollutants into water systems over time [40,41].

Once introduced into the environment, emerging contaminants undergo various physicochemical and biological processes that determine their persistence and distribution, allowing them to disperse through natural cycles and impact water resources, as well as terrestrial and atmospheric ecosystems. In detail, Figure 1.6 illustrates the fate of CECs, showing how they are transported through water bodies, deposited onto sediments, or volatilized into the atmosphere. Key processes such as hydrolysis, biodegradation, and photolysis can alter the chemical structure of these contaminants, sometimes producing even more harmful byproducts [42].

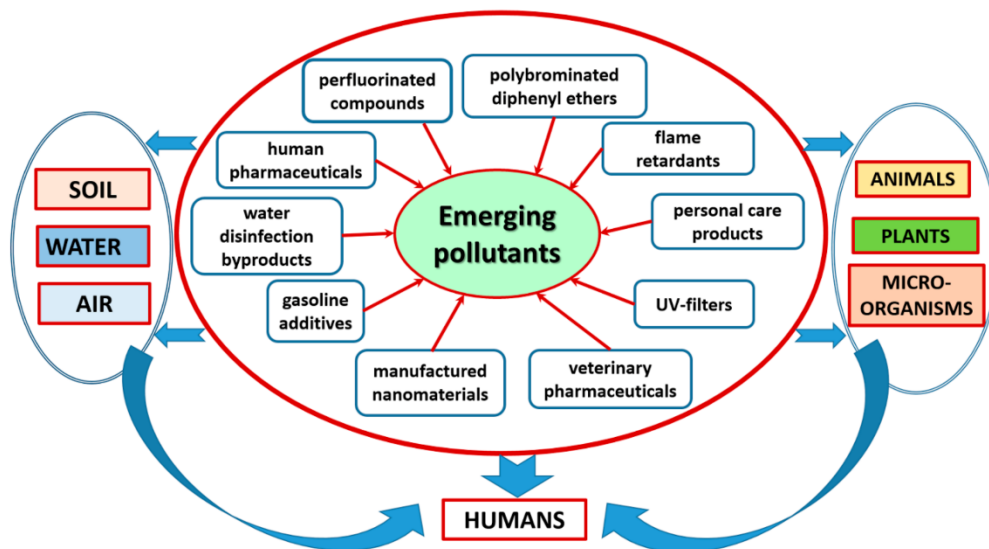


Figure 1.4: CECs overview and distribution [36].

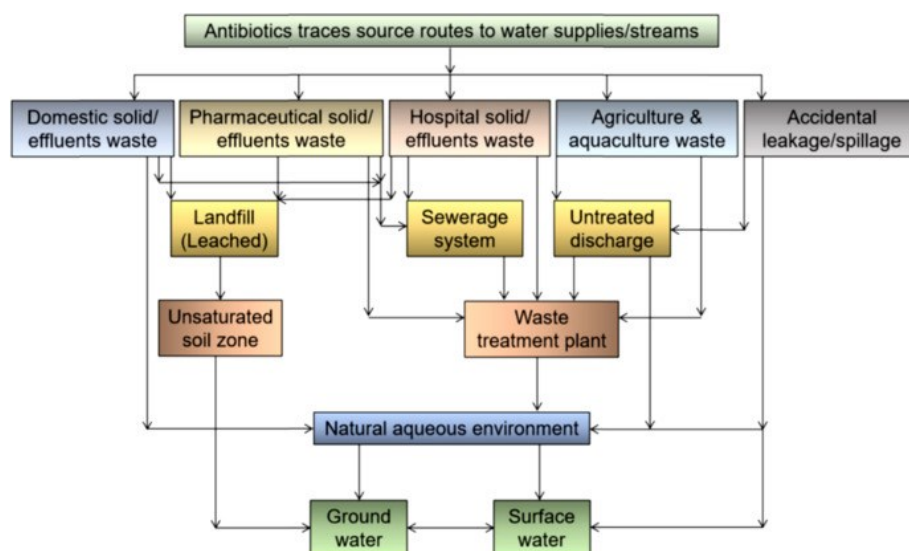


Figure 1.5: Emerging contaminants pathway from source to the environment [43].

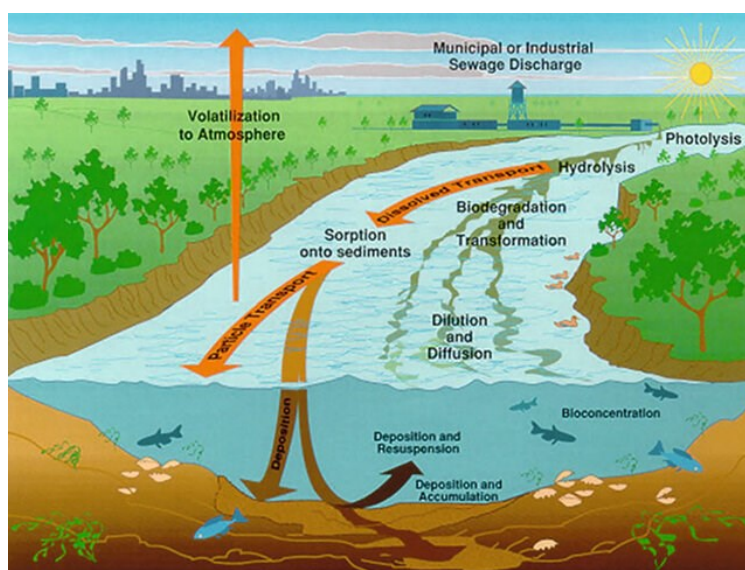


Figure 1.6: Fate of CECs in aquatic and terrestrial environments [44].

Despite their potential hazards, many emerging contaminants are not currently regulated under existing EU water management frameworks (e.g., Directive 2000/60/EC, Directive 2008/56/EC, Directive 2013/39/EU). As a result, conventional WWTPs are not fully equipped to eliminate these substances, leading to their continuous release into the environment [45]. Furthermore, growing attention is being given to persistent, mobile, and toxic (PMT) substances, as well as very persistent and very mobile (vPvM) contaminants, due to their ability to remain in environmental systems for extended periods. Understanding the characteristics, transformation mechanisms, and environmental persistence of CECs is essential for developing effective mitigation strategies and improving water quality management [46–49]. Therefore, understanding the characteristics of these contaminants is crucial in understanding their fate and potential risk in the environment.

1.3 Persistent, mobile, and toxic (PMT) and very persistent, very mobile (vPvM) substances

Compounds that are highly mobile and persistent (PMT/vPvM) can pose significant risks to aquatic ecosystems and human health since they can migrate across soil layers, contaminate groundwater and drinking water resources, and are not removed by conventional water treatment methods., leading to long-term environmental and human health concerns [50–52]. Moreover, ceasing emissions does not guarantee an immediate reduction in environmental concentrations, imposing long-term economic and resource burdens on society [53]. In fact, they are specifically addressed and managed under the REACH (Registration, Evaluation, Authorization, and Restriction of Chemicals) regulation by the European Union. Manufacturers are required to assess and manage risks associated with the use of these substances in order to limit their movement and accumulation in the environment. In detail, REACH is required to evaluate all substances based on persistence, toxicity, mobility, and bioaccumulation criteria, as shown in Figure 1.7.

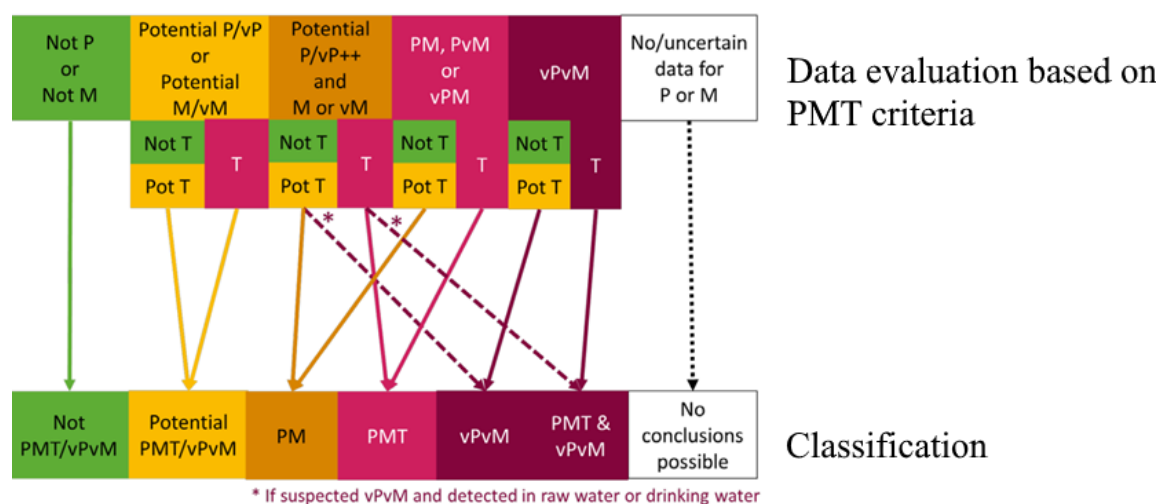


Figure 1.7: Summary of REACH classification guidelines for PMT substances [54].

1.3.1 Occurrences and Environmental Impacts of PMT/vPvM

The presence of PMT/vPvM substances has been detected globally. For example, Huang and co-workers (2021) [51] found that among the 432 pharmaceuticals and personal care products (PPCPs) monitored in Chinese water resources, 138 were detected in surface water, with 52 (37.7%) meeting or being suspected of meeting the PMT/vPvM (persistent, mobile, and toxic/very persistent, very mobile) criteria. In drinking water and groundwater, the percentage of APIs and PCPs meeting PMT criteria was higher, reaching ca. 44.3% (Figure 1.8). As shown in Figure 1.9, antibiotics were the most present class of compounds, with fluoroquinolones and sulfonamides being the most frequently

detected. The Yangtze, Pearl, and Haihe River basins, being the most industrialized and populated, had the highest concentrations of these pollutants.

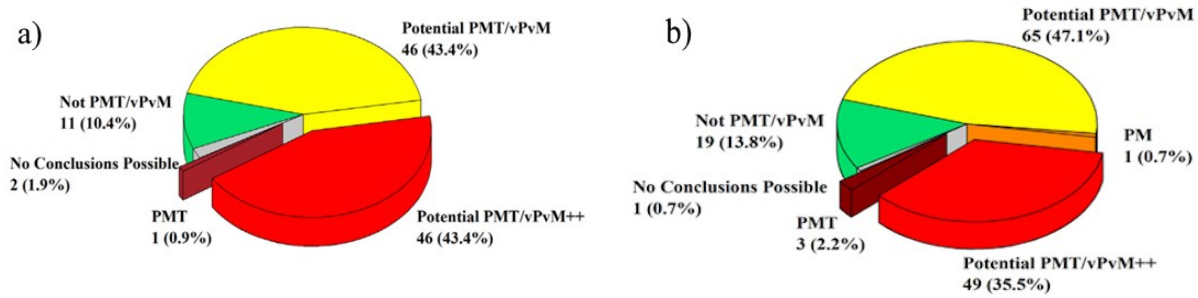


Figure 1.8: PMT/vPvM assessment of PPCPs detected in drinking water and groundwater in China (a), and PMT/vPvM assessment of PPCPs detected in Chinese surface water (b) [51].

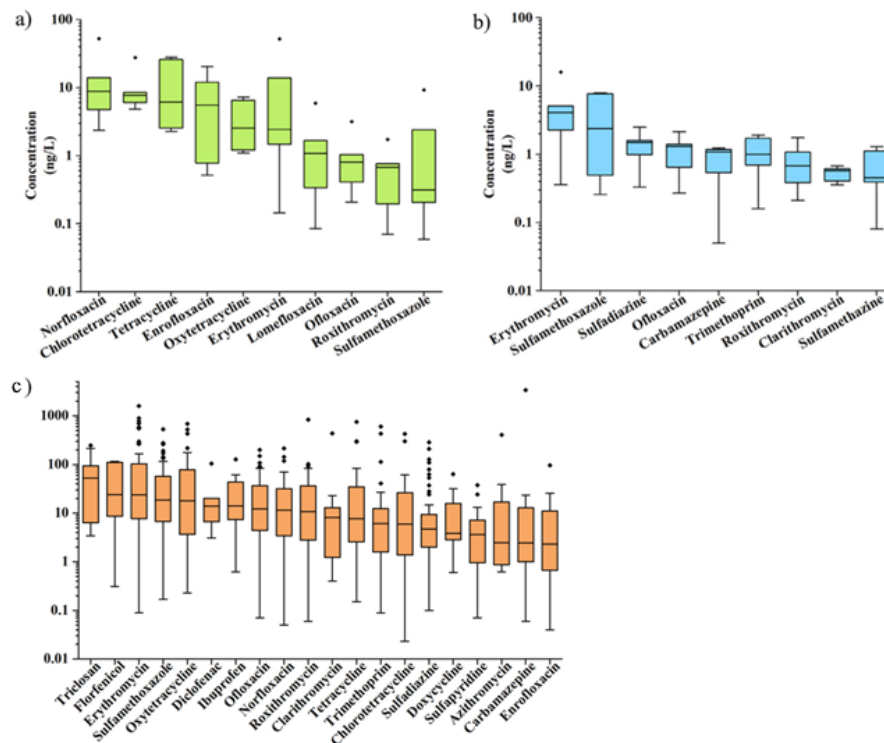


Figure 1.9: Concentration of the most abundant PPCP-PMT/vPvM substances detected in groundwater (a), drinking water (b), and surface water (c) as reported for China [51]

Furthermore, a study in Germany by [55] Neuwald et al. (2022) carried out suspect screening, which prioritized 34 PMT/vPvM substances for the analysis in 46 water samples from various drinking water sources across Germany. The samples revealed significant contamination by several substances, including 1H-benzotriazole, melamine, cyanuric acid, and 1,4-dioxane. These four substances accounted for over 95% of the total detected concentrations, with 1H-benzotriazole reaching a peak concentration of 56 $\mu\text{g/L}$ in surface water near industrial point sources, as shown in Figure 1.10. The study also identified other emerging contaminants, including 2-acrylamido-2-methylpropanesulfonic

acid (AMPS) and diphenylguanidine (DPG), with DPG concentrations varying widely due to stormwater runoff. Statistical analyses showed that PMT/vPvM substances from consumer products exhibited a higher detection frequency than those from industrial sources, suggesting widespread urban wastewater contributions. The results further confirmed that most PMT/vPvM substances passed through bank filtration without significant reduction in concentration, raising concerns about their persistence in drinking water sources.

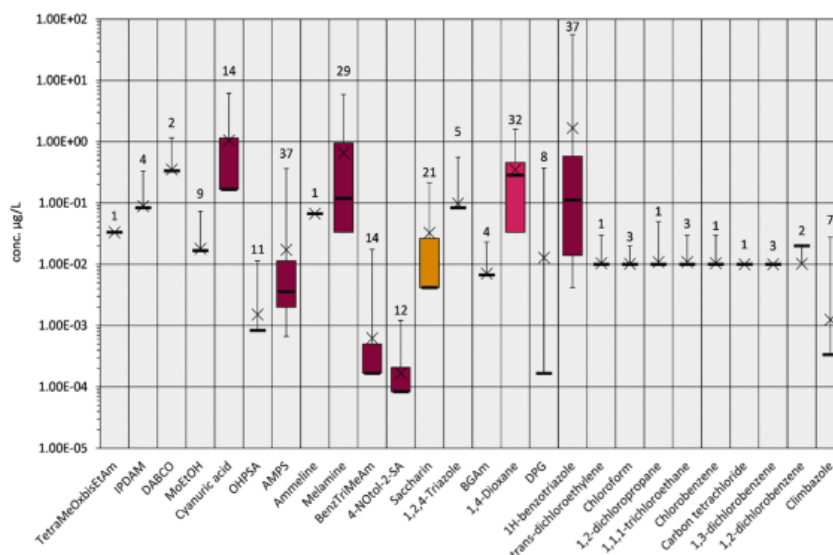


Figure 1.10: Boxplots of PMT/vPvM substance concentrations across all samples. Mean, median, minimum, maximum, and interquartile ranges [55].

1.4 Main sources of water contamination

Over the past decades, water resources have been polluted by human activities. In this regard, industrial and civil discharges, and agricultural activities have been shown to be among the main sources of water pollution. More specifically, industrial effluents could be characterized by the presence of high-concentration metals (leather tanning and smelting industries) and organic pollutants (e.g., dyes in textile industries [56]). Meanwhile, agricultural runoffs can contain fertilizers, pesticides, and organic waste that can lead to the degradation of water resources. Moreover, in countries with sufficient land for landfilling, surface and groundwater can be contaminated due to unsanitary landfilling practices via leachate infiltration through the soil [57,58]. In detail, landfill leachate for classification is commonly referred to by age: young (<5 years), intermediate (5-10 years), and mature (>10 years), each having a different chemical composition. The types of leachates are influenced by landfill design features such as waste types, waste disposal methods, and engineering design aspects such as liners and drainage systems. For instance, municipal solid waste landfills will commonly generate leachate with high organic carbon and ammoniacal nitrogen, and hazardous waste landfills will produce leachate that contain heavy metals and persistent organic

pollutants. Therefore, efficient leachate management requires treatment technologies that are optimized to leachate characteristics, which can include biological treatment processes for biodegradable organics, and advanced oxidation processes and membrane filtration for more persistent chemical compounds [59–61]. Based on the focus of this thesis, landfill leachate, and textile industry wastewater will be discussed in more depth.

1.4.1 Landfill leachate

Landfill leachate is a liquid byproduct of the physical, biological, and chemical breakdown of solid waste occurring in landfills [62], and it generally contains several types of contaminants including dissolved organic matter (e.g., volatile fatty acids, proteins, and carbohydrates), macro inorganic compounds (e.g., Ca^{2+} , Mg^{2+} , Na^+ , K^+ , NH_4^+ , Fe^{2+} , Mn^{2+} , and HCO_3^-), metals (e.g., Cd^{2+} , Cr^{3+} , Cu^{2+} , Pb^{2+} , and Ni^{2+}) and organic pollutants (e.g., polyaromatic hydrocarbons, phenols, and pesticides) (Table 1-1).

Table 1-1: Leachate composition in terms of average values and ranges for parameters with differences between acid and methanogenic phase (all values in mg/l except pH and BOD₅/COD) [63].

Parameters	Acid phase		Methanogenic phase		Average
	Average	Range	Average	Range	
pH	6.1	4.5–7.5	8	7.5–9	
BOD ₅	13,000	4000–40000	180	20–550	
COD	2000	6000–60000	3000	500–4500	
BOD ₅ /COD ratio	0.58		0.06		
Sulfate	500	170–1750	80	10–420	
Calcium	1200	10–1200	60	20–600	
Magnesium	470	50–1150	180	40–350	
Iron	780	20–2100	15	3–280	
Manganese	25	0.3–65	0.7	0.03–45	
Ammonia-N					740
Chloride					2120
Potassium					1080
Sodium					1340
Cadmium					0.005
Chromium					0.28
Copper					0.065
Lead					0.09
Zinc	5	0.1–120	0.6	0.03–4	

Young leachate has a high concentration of BOD (between 4000 and 13000 mg/L) and COD (between 30000 and 60000 mg/L) [64], with a low pH (less than 6.5), high biodegradability potential, and a TOC/COD ratio of 0.2-0.5 and BOD₅/COD ratio of 0.5-1.0. Mature landfill leachate is characterized by a high proportion of large organic molecules, low concentrations of biodegradable substances (COD<3000 mg/L), BOD₅/COD ratio of less than 0.1, and high ammonia concentrations (more than 1000mg/L). In contrast, intermediate-age landfill leachate has a COD/TN ratio of 3.0-6.0 and moderate biodegradability [65].

Leachate from landfills can contaminate various environmental compartments, posing significant risks to ecosystems and human health. In detail, if improperly constructed landfill liners will allow landfill leachate to seep out and create a leachate pond. The leachate from the pond can migrate to nearby water resources such as rivers, lakes, and streams and contaminate them [66]. Additionally, leachate percolates through soil layers, leading to groundwater pollution and endangering drinking water sources, presenting risk in both active and closed landfill sites, as illustrated in Figure 1.11 [67].

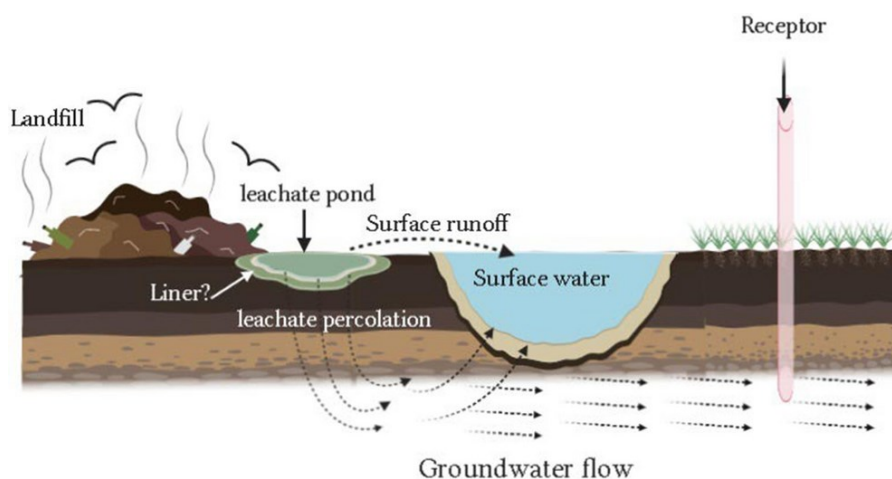


Figure 1.11: Landfill leachate contaminating surface water, groundwater, and soil [66].

1.4.2 Textile industry discharges

Textile industries are among the major industries worldwide, and while much progress has been made to reduce its environmental impact [68], this industry is still struggling in many developing countries to adopt source-level waste minimization measures [69]. Textile industries use harmful chemicals such as metals and organic pollutants. In fact, the major pollutants present in textile wastewater are spent dye and Cr⁺⁶ [70]. In particular, synthetic dyes have been extensively used due to their low cost, ease of preparation, and color intensity. Among these compounds, Azo dyes are the most commonly produced chemical dyes (i.e., 60% of the total dyes) in the world, and their use is expected to grow in the future [71]. Aside from the textile industries, these dyes are also used in paper, food, printing, cosmetics, and leather industries [72,73].

Investigating the elimination of azo dyes from wastewater is important since about 60-70% of them have been shown to have toxic, carcinogenic, and mutagenic effects [74]. Among the dyes available for industrial use, some dyes are used more pervasively. These dyes include Rhodamine B, Reactive Red, Reactive Black, Reactive Blue, Reactive Yellow, Reactive Orange, Congo Red, Acid Red, Acid Blue, Direct Red, Methylene Blue, and Methyl Orange [72,75,76]. Textile plant effluents have been characterized by high chemical oxygen demand (COD), biochemical oxygen demand (BOD), high salinity, total suspended solids (TSS), total dissolved solids (TDS), total nitrogen (TN), alkaline pH (Table 1-2). Salinity, in particular, has been reported to range from 2 to 12.5 g/L in effluents from tannery and textile plants. The high levels of salinity can cause interference with water and wastewater treatment processes and should be considered in investigating the water and wastewater treatment processes [77,78].

In this thesis, the degradation of two prominent azo dyes in the textile industry (i.e., Congo Red (CR) and Reactive Red 120 (RR120)) using hydrodynamic cavitation were investigated under various testing conditions (i.e., the presence of oxidants, salt). Moreover, the most common practices for treating textile wastewater include a series of treatment units that can be used based on the characteristics of the discharge. In detail, biological processes, such as the Upflow Anaerobic Sludge Blanket (UASB) reactor followed by aerobic treatment, have demonstrated high efficacy in removing organic loads and color from textile wastewater [79]. After the biological component of the leachate is treated, AOPs, including Fenton and photocatalysis, are used in degrading complex dye molecules. These processes generate highly reactive hydroxyl radicals that can mineralize organic pollutants. However, their efficiency can be influenced by factors such as pH and the presence of other substances in the wastewater [72]. Then membrane technologies (e.g., ultrafiltration, nanofiltration, etc.) are employed to stop the contaminants that were not removed in the previous steps [80]. At the end of the treatment train, if there are any residual dyes or organic contaminants, adsorption via activated carbon is used to eliminate the contaminants [81].

Table 1-2: Characteristics of conventional textile industry wastewater [82]

Parameters	Values
pH	6-10
Temperature (°C)	35-45
Biochemical oxygen demand (mg/L)	80-6000
Chemical oxygen demand (mg/L)	150-12000
Oil and Grease (mg/L)	10-30
Total suspended solids (mg/L)	15-8000
Free ammonia	<10

Total dissolved solids (mg/L)	2900-3100
Chloride (mg/L)	1000-1600
Sodium (mg/L)	70
Trace elements (mg/L)	<10
Silica (mg/L)	<15
Total Kjeldahl nitrogen (mg/L)	70-80
Color (Pt-Co)	50-2500
Salinity (mg/L)	2000-12500

1.5 Water and wastewater treatment technologies

Water and wastewater treatment is mainly dependent on the types of contaminants involved and in order to ensure high quality of water, each class of contaminants should be addressed individually. This creates the need for a comprehensive treatment system that meets the specific treatment requirements for each contaminant type. However, water and wastewater treatment technologies are influenced by socio-economic aspects and the institutional capacity of a society, as shown in Figure 1.12. In detail, basic treatments often rely on low-cost methods such as coagulation/precipitation, filtration of colloidal and other suspended matter, followed by disinfection. Advanced technologies (e.g., membrane filtration, adsorption, and catalytic or electrochemical oxidation) are increasingly used as secondary steps to remove chemicals that may bypass traditional treatment processes such as volatile organic compounds (VOCs), personal care products (PCPs) and cyanotoxins. Moreover, for contaminants that are more difficult to treat (e.g., PFAS and APIs), membrane filtration (i.e., nanofiltration and reverse osmosis) and advanced oxidation processes (AOPs) have been shown to be very effective [83].

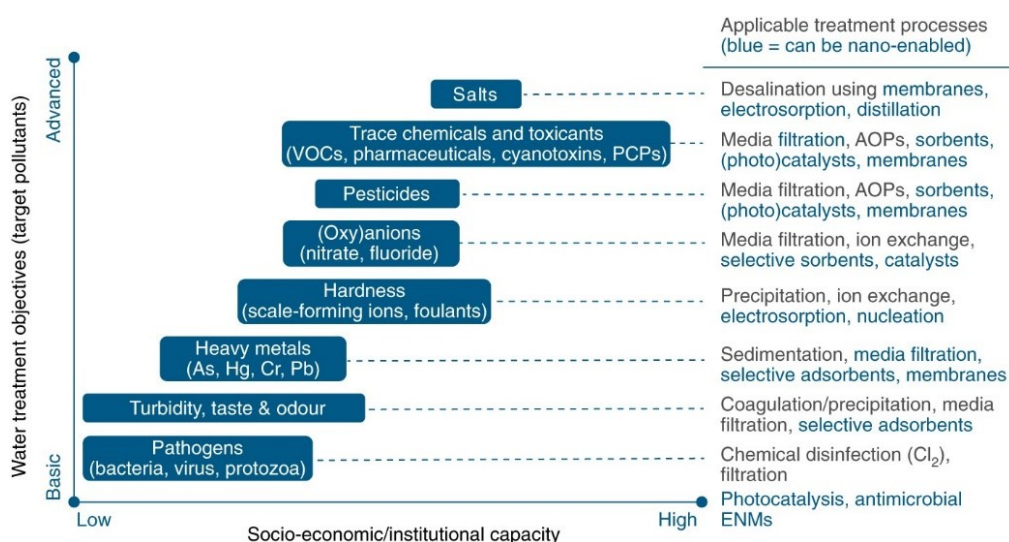


Figure 1.12: Socio-economic water/wastewater treatment aspects based on contaminant class [84].

1.5.1 Wastewater treatment technologies for CECs

The removal efficiency of CECs in WWTPs depends on several factors, such as their persistence, physicochemical properties, used treatment technologies, and operating conditions. For this reason, WWTPs usually use primary, secondary, and sometimes tertiary treatment steps. Although the primary treatment phases are designed to remove suspended and colloidal solids, CECs are also removed to some extent, mostly through sorption onto the primary sludge (i.e., 20-50% removal), as shown in Figure 1.13 [85]. The secondary treatment is employed to remove nutrients and/or organics via biological degradations (i.e., 30-70% removal). The CECs undergo biodegradation, sorption,

dispersion, dilution, photodegradation, and volatilization in this phase. However, the main removal mechanisms are biodegradation and sorption [86,87]. The tertiary treatment phase is designed to degrade or remove pathogens, suspended solids, and nutrients, but it also shows a significant CEC removal percentage. In fact, some of the most recalcitrant CECs are removed effectively using advanced oxidation processes (AOPs) [88].

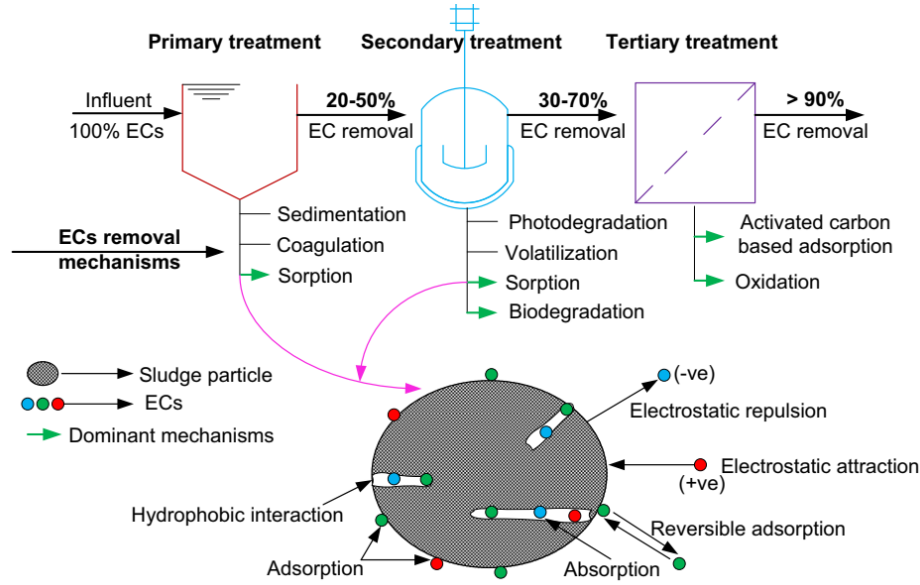


Figure 1.13: CECs removal mechanism in a typical wastewater treatment plant [85].

1.5.1.1 Primary treatment technologies for CECs

Sorption is the key process responsible for CECs elimination during primary treatments, including both absorption onto the organic component of the primary sludge through hydrophobic interactions and adsorption on sludge particles' surface sites via electrostatic interactions, as expressed by Equation 1:

$$K_d = \frac{C_{solid} \cdot \rho_{solid}}{C_{aq}} \quad \text{Equation 1}$$

Where K_d is the ratio of contaminant concentration in the sludge to the concentration in water, C_{solid} is the concentration of contaminant on solid phase (sorbed concentration) ($\mu\text{g/g d.w.}$); ρ_{solid} is the density of sludge particles (g/L), and C_{aq} is the dissolved concentration of the contaminant ($\mu\text{g/L}$).

1.5.1.2 Secondary treatment technologies for CECs

In secondary treatments, the main mechanisms involved are biodegradation and sorption, while other mechanisms, such as photodegradation and volatilization, have negligible effects [89]. For this reason, secondary biological treatment technologies are the most prominent technologies that are employed to remove CECs [90]. Specifically, Activated Sludge Processes (ASP) is the most practiced method in traditional WWTPs. In addition, there are other highly effective biological methods such

as biological aerated filters (BAF), constructed wetlands, membrane bioreactors (MBRs), rotating biological contactors, moving bed biological reactors (MBBR), microalgal bioreactors, oxidation ditches, that are used in unconventional WWTPs that are able to optimize their treatment plants to incorporate these technologies [91]. The most common secondary treatment methods are discussed briefly in the next section.

1.5.1.2.1 Activated sludge processes

The activated sludge process (ASP) utilizes the microorganisms available in the sludge to degrade and convert CECs found in the wastewater. Such organic pollutants can be consumed and metabolized by these microorganisms into other substances, and they can also be incorporated into the biomass within the sludge through sorption. However, the efficiency of each of the processes is determined by the nature of each contaminant as CECs undergo varying degrees of biodegradation during this treatment process, ranging from full mineralization (conversion to carbon dioxide and water) to partial degradation that produces several byproducts [92]. For example, methoxy-triclosan can biodegrade up to 81% in ASP; but, when exposed to laccase enzyme during ASP, it is converted into a more refractory form, 2,4-dichlorophenol, to a lower extent (56.5%).

1.5.1.2.2 CECs removal in membrane bioreactor

Membrane filtration is based on hydrostatics pressure that allows water and low molecular weight solutes to pass through while stopping larger molecules. Membrane filtration is classified based on the membrane's properties (e.g., hydrophobicity, pore size) into the following categories: i) microfiltration (MF) (i.e., pore size between 0.1 to 10 micrometers, effective for removing suspended particles and large bacteria), ii) ultrafiltration (UF) (i.e., pore size between 0.01 to 0.1 micrometers, effective for stopping proteins, viruses and silica), iii) nanofiltration (NF) (i.e., pore size 0.001 to 0.01 micrometers, effective for multivalent ions, synthetic dyes, sugars and specific salts), and iv) reverse osmosis (RO) (i.e., 0.0001 to 0.001 micrometers, effective in removing almost all dissolved salts and minerals, organic molecules, microorganisms). Recently, biological membranes have been gaining attention, especially in urban wastewater treatment. In detail, MBR is an advanced treatment technology that combines biological processes (e.g., activated sludge) with membrane filtration [93]. However, in the recent work of Lopez-Herguedas and co-workers (2024) [94], it was shown that only 27% of the CECs initially detected in the effluent were efficiently removed using the MBR filtration and most of the CECs persisted at elevated concentrations in the effluent. Therefore, further advancements are needed in this area to make innovative membrane filtration a viable method for CECs removal.

1.5.1.2.3 CECs removal with AOP

Advanced oxidation processes (AOPs) employ various technologies to generate reactive radicals (e.g., $OH^\cdot$, $SO_4^{\cdot-}$) that are able to oxidize complex organic pollutants [95]. In detail, $OH^\cdot$ and $SO_4^{\cdot-}$ are among the most used radicals for water treatment, and their main reaction mechanisms are described in Equation 2 to Equation 9.



In detail, the most efficient AOPs are those that include an oxidizer (e.g., persulfate, H_2O_2) combined with an activator. Moreover, activation can occur due to several factors, such as temperature, UV radiation, and cavitation [96]. Among various AOPs some have proven to be more effective for CEC removal both alone and in combination with other treatment methods (e.g., Ozonation, Fenton process, UV- H_2O_2). In The following sections, some of these methods are discussed briefly.

1.5.1.2.4 Ozonation

Ozonation is an AOP that utilizes ozone (O_3) to oxidize organic contaminants in water. The process involves both direct reactions of ozone with pollutants and indirect reactions through the generation of hydroxyl radicals ($OH^\cdot$), which are highly reactive and non-selective oxidants. The mechanisms are shown in Equation 10 and Equation 11.



These reactions lead to the breakdown of complex organic molecules into simpler, less harmful compounds. Ozonation has been effectively applied in the degradation of various CECs, including pharmaceuticals and personal care products [97].

1.5.1.2.5 Fenton process

The Fenton process involves the reaction of hydrogen peroxide (H_2O_2) with ferrous iron (Fe^{2+}) to produce hydroxyl radicals, which then oxidize organic contaminants. This method is particularly

effective in degrading a wide range of CECs in wastewater [98]. Equation 12 and Equation 13 indicate the key mechanisms involved in a Fenton process that enables it to continuously generate hydroxyl radicals, thus facilitating the degradation of persistent organic compounds.



1.5.1.2.6 UV/H₂O₂ process

The UV/H₂O₂ process combines ultraviolet (UV) irradiation with hydrogen peroxide to generate hydroxyl radicals, which are capable of oxidizing a broad spectrum of organic contaminants. This AOP is effective in the degradation of various CECs present in water and wastewater. The hydroxyl radicals produced in this reaction are highly reactive and can non-selectively oxidize organic pollutants, leading to their mineralization. The UV/H₂O₂ process has been applied in the treatment of wastewater containing pharmaceuticals, pesticides, and other emerging contaminants [99].



1.5.1.2.7 Membrane technologies

Membrane technologies have proven to be one of the most effective methods in wastewater treatment for their efficiency in separation of contaminants from wastewater [100]. In detail, membranes such as microfiltration (MF), ultrafiltration (UF), nanofiltration (NF), and reverse osmosis (RO) are used for various contaminants removal, including suspended solids and dissolved organic compounds (e.g., PFAS, pharmaceuticals, and dyes [101]. In this context, NF and RO are widely used for CECs removal due to their high efficiency [102]. In detail, NFs are characterized by pore size between 0.5-2nm and operate under pressures between 4 to 30 bar depending on the type of the NF membrane used. These membranes are also suitable to remove divalent and larger monovalent ions, and pathogens, making them suitable for various treatments [103].

RO membranes have smaller pore size (i.e., 0.1 to 1nm) and usually operate under higher pressures (i.e., 3 to 80 bar) depending on the sacel and type of wastewater [104]. These membranes are highly effective in removing a wide range of contaminants (e.g., organic molecules, dissolved salts, viruses, microorganisms, and PFAS) [105]. Furthermore, membrane bio reactors (MBRs) that combine biological treatment with membrane filtration are commonly used for municipal and industrial effluents [106].

1.5.2 Cavitation technology

Cavitation is a phenomenon where vapor bubbles form in a liquid due to localized pressure reductions, subsequently collapsing and releasing energy. In detail, it involves rapid pressure variations that lead to bubble nucleation, growth, and collapse, generating intense localized forces, high temperatures, and hydroxyl radicals. The produced radicals can be used for the degradation of

pollutants, (Equation 2-Equation 5), leading to the application of this phenomena to several fields, such as water treatment, material processing, and biomedical fields [107,108].

This process can be induced through different mechanisms, including hydrodynamic, ultrasonic, optical, and particle cavitation [109]. The following section briefly describes and discusses the two most used processes (i.e., ultrasonic cavitation and hydrodynamic cavitation).

1.5.2.1 Ultrasonic cavitation

Ultrasonic cavitation is induced by high-frequency sound waves (e.g., from 16kHz to 2MHz) dispersing through a liquid medium, creating alternating compression and rarefaction cycles [110]. During the rarefaction phase, cavitation bubbles form, while the compression phase leads to their violent collapse, producing localized high temperatures and pressures (Figure 1.14). Ultrasonic cavitation has been widely explored for its potential in wastewater treatment, demonstrating efficiency in removing turbidity and breaking down contaminants, such as the work of Zhang and co-workers [111], in which they employed ultrasonic cavitation to remove o-cresol from wastewater. Although ultrasonic cavitation has shown good results at lab or pilot scales, its high energy consumption prevents large-scale application as it would render the entire treatment economically unsustainable. Moreover, the depth to which ultrasonic waves penetrate the medium is limited, restricting its efficiency with high volumes [112,113].

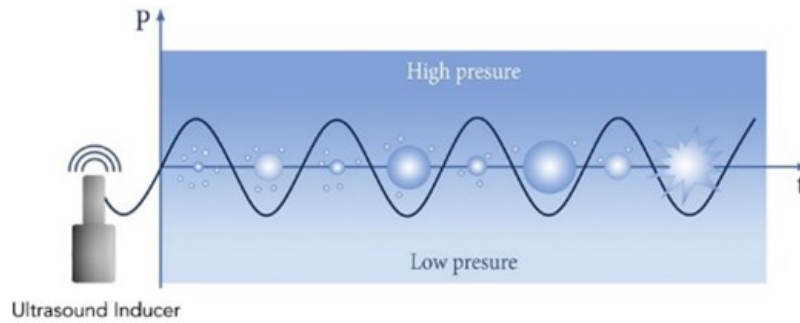


Figure 1.14: Ultrasonic cavitation and bubble formation with wave alterations [114]

1.5.2.2 Hydrodynamic cavitation

Hydrodynamic cavitation (HC) is a way of producing cavitation bubbles in a flowing liquid using pressure variations, usually achieved with Venturi tubes, orifice plates, and rotating cavitation generators [115]. Such processes are governed by Bernoulli's equation (i.e., Equation 15), where the pressure reduces at certain points, inducing cavitation and the formation of both micro and nano bubbles. Their collapse leads to very high localized temperatures (as much as 7200K) and pressures (up to 10^8 Pascal), enabling hydroxyl radical formation [116,117].

$$P + \frac{1}{2}\rho U^2 = C(\text{constant}) \quad \text{Equation 15}$$

Where U is the rate of flow of water at a certain point, P is the pressure, and ρ is the density of the liquid. Furthermore, the geometry of the Venturi tube has a marked influence on cavitation efficiency, and micro- and nano-bubble formation plays an important role in pollutant degradation [118]. The mechanism of micro- and nano-bubble generation in a Venturi tube is illustrated in Figure 1.15.

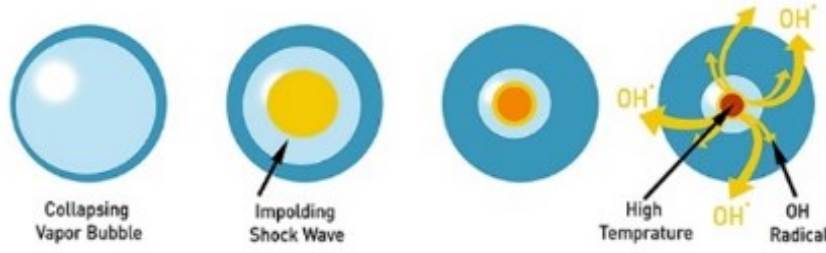


Figure 1.15: Hydrodynamic cavitation mechanism in bubble nucleation and hydroxyl radical generation [114]

The cavitation number (K_c), which indicates the ability of the liquid to withstand cavitation, is a very important parameter for optimization affecting HC performance [119], as shown in Equation 16.

$$K_c = \frac{P_{loc} - P_{vap}}{\frac{1}{2} \rho_1 U_{loc}^2} \quad \text{Equation 16}$$

Where P_{loc} is the atmospheric pressure, U_{loc} is the stream's velocity, and P_{vap} is the vapor pressure. Cavitation occurs when K_c is either equal to or less than the cavitation inception number (K_i), which is determined by laboratory data and indicates the cavitation intensity and yield (usually, $K_i < 3$) [108]. In fact, when reduced below a critical limit (typically $K_c < 3$), increased cavitation intensity occurs, thereby increasing the efficiency of $OH^\bullet$ radical formation [119]. Moreover, the temperature also significantly affects the cavitation yield by decreasing the viscosity (Equation 17, where μ_T is dynamic viscosity at temperature T , μ_0 is the reference viscosity, E_0 is the activation energy for viscous flow, R is the gas constant, and T is the absolute temperature in Kelvin) of the liquid that, in turn, increases the Reynolds number (Equation 18, where Re is the Reynolds number, ρ is the fluid density (kg/m^3), v flow velocity (m/s), D is the characteristic length (e.g., diameter of the Venturi or orifice) (m) and μ is the dynamic viscosity of the fluid (N.s/m^2)). The increased Reynolds number indicates a more turbulent flow (and, therefore, more cavitation yield). However, it should be noted above ca. 40 - 50°C, the vapor pressure increases to a point when it interferes with bubble formation [120], creating the condition called supercavitation [121].

$$\mu_T = \mu_0 \cdot e^{\frac{E_a}{RT}} \quad \text{Equation 17}$$

$$Re = \frac{\rho \cdot v \cdot D}{\mu} \quad \text{Equation 18}$$

Furthermore, the temperature can also affect the $OH^\bullet$ radical generation through the cleavage of water molecules inside the cavitation chamber (e.g., Venturi, orifice) via the extreme local conditions (i.e., high temperature and pressure). In detail, Yeneneh et al. (2024) [122] showed that the optimal temperature range for $OH^\bullet$ radical generation is from ca. 35°C to 40°C. Below this temperature range, the number of bubbles decreases, and fewer radicals are produced. However, if the water is too hot

(i.e., above 40°C), the vapor pressure inside the bubbles is much higher. Therefore, when the bubble collapses, this high vapor pressure acts as a cushion, absorbing some of the shock and energy of the collapse. Essentially, the vapor inside the bubble resists compression and prevents the walls from collapsing as rapidly or violently as they would, thus reducing the radical generation and cavitation effect simultaneously. However, it should be noted that in pilot-scale systems, controlling the temperature within such intervals can pose significant challenges since both initial water temperature and heat exchange efficiency are affected by several factors (e.g., lab temperature, pipe material, temperature of cooling water). In detail, even though a heat exchanger was built and integrated into the system, it was impossible to maintain the system in the optimal temperature range, but the temperature was maintained between 20 and 40°C.

Consequently, due to the combined effect of cavitation phenomena and OH[•] radical generation, hydrodynamic cavitation has shown the potential to remove organic pollutants such as pharmaceuticals, industrial dyes, and solvents [123]. Different studies demonstrate its efficiency in combination with other advanced oxidation processes (AOPs), such as hydrogen peroxide, ozonation, and the Fenton process. For example, Gagol et al. (2018) [124] achieved a 60% removal of p-nitrophenol using HC with H₂O₂, while Fedorov et al. (2022) [125] demonstrated a similar treatment with 98.37% degradation of dodecyl sulfate. Moreover, Araujo et al. (2020) [126] investigated the use of HC with H₂O₂ to degrade natural organic matter (NOM) represented by humic acid (HA).

The characteristics of the bubbles generated using cavitation systems are usually studied considering the following parameters:

- i. Average diameter, defined as the mean diameter of all bubbles detected by the laser particle size analysis;
- ii. Frequency, defined as the mean volumetric fraction of the nanobubbles having the average diameter;
- iii. Z-potential is defined as the overall charge of all bubbles present in the system.

1.5.3 Water and wastewater treatment interferences

Several substances can interfere with water and wastewater treatment processes by inhibiting or diminishing their usual functions and efficiencies [127] as they can act as scavengers for oxidants (e.g., ozone, hydroxyl radicals, and sulfate radicals), occupy adsorption material's active sites, cause membrane fouling, and lead to emulsification processes.

In particular, the presence of Natural Organic Matter (NOM) poses great challenges in the treatment of water and wastewater [128,129] since i) it can act as scavenger for active species leading to increased oxidants consumption [130], ii) it can act as a precursor of disinfection by-products (DBPs) leading to environmental and health risks [131], iii) it can accumulate within membranes' surface

resulting in decreased permeate flux, increased cleaning and replacement frequency, thus increasing operational costs [132]. iv) the variability in its composition can affect coagulant demand and sludge production, making it difficult to optimize coagulation/precipitation processes. For these reasons, to decrease the formation of unwanted by-products, lower operational costs, increase treatment efficiency, and improve the maintenance of treatment systems, removing NOM from the treated water before the main treatment is necessary.

In this work, a hydrodynamic cavitation system designed for specific nanobubble production via a Venturi tube was employed and tested for various organic compounds (i.e., NOM and industrial dyes: Reactive Red 120, Congo Red) with and without the presence of oxidants (i.e., H_2O_2 and $\text{Na}_2\text{S}_2\text{O}_8$ (Sodium persulfate)) to compare the treatment efficiency for various organic compounds and via a specific Venturi tube with nanobubble production.

2. Chapter 2: Materials and Methods

2.1 Chemicals and reagents

Humic acid (HA, Fluka Chemika, technical grade) was used as a proxy of Natural Organic Matter and dissolved in MilliQ water to prepare a 200mg_{TOC}/L solution. The solution was stirred at room temperature for 36 hours in the dark and centrifuged 3 times at 9000 rpm for 15 minutes using a centrifuge (Eppendorf 5910Ri) to eliminate any suspended particles. Congo Red (CR, dye content $\geq 35\%$, Sigma-Aldrich) and Reactive Red 120 (RR120, dye content ca. 50%, Sigma-Aldrich) were used as model dyes in this study. Solutions of the selected dyes (10mg/L for CR and 15mg/L for RR120) were prepared using MilliQ water and centrifuged 3 times at 9000 rpm for 15 minutes to eliminate any suspended particles.

Sodium persulfate ($\text{Na}_2\text{S}_2\text{O}_8$, $\geq 98\%$, Sigma-Aldrich) and hydrogen peroxide (H_2O_2 , 30% w/w, Sigma-Aldrich) were used as oxidizing agents. Sodium chloride (NaCl , 99%, Carlo Erba) was used to simulate saline conditions in wastewater during hydrodynamic cavitation experiments. Sulfuric acid (H_2SO_4 , 98% w/w, Sigma-Aldrich) was used for pH adjustment during hydrodynamic cavitation experiments. A 45 g/L solution of Sodium sulfite (Na_2SO_3 , $\geq 98\%$, Carlo Erba) was used as a radical quenching agent to avoid further reaction of the oxidants used in the cavitation experiments, with a Na_2SO_3 :oxidant molar ratio of 22.04 for PS and of 195 for H_2O_2 [133,134].

Ferric chloride (FeCl_3 , $\geq 98\%$), ferric sulfate (FeSO_4 , $\geq 98\%$), polyaluminum chloride (AlCl_3 , 98%), H_3PO_4 85 % (w/w) solution (ACS reagent), NaOH 30 % (w/w) solution (technical grade), and glucose ($\text{C}_6\text{H}_{12}\text{O}_6$, $\geq 98\%$) were purchased from Merck KGaA (Darmstadt, Germany) and used for leachate treatment.

HCl (35%), HNO_3 (69%), HF (48%), and B(OH)_3 were purchased from PanChem (AppliChem GmbH, Darmstadt, Germany) at high purity for trace metal analysis.

Ultrapure water with a resistivity of 18.2 M Ω cm was obtained with the MilliQ system from Merck KGaA (Darmstadt, Germany). All reagents were used as received without further purification.

The molecular structures of the substances used to test the performance of the developed hydrodynamic cavitation system (i.e., humic acid, Congo Red, and Reactive Red) are shown in Figure 2.1. It should be noted that for both the dyes and natural organic matter solution, simplified effluents were created in order to avoid additional interference.

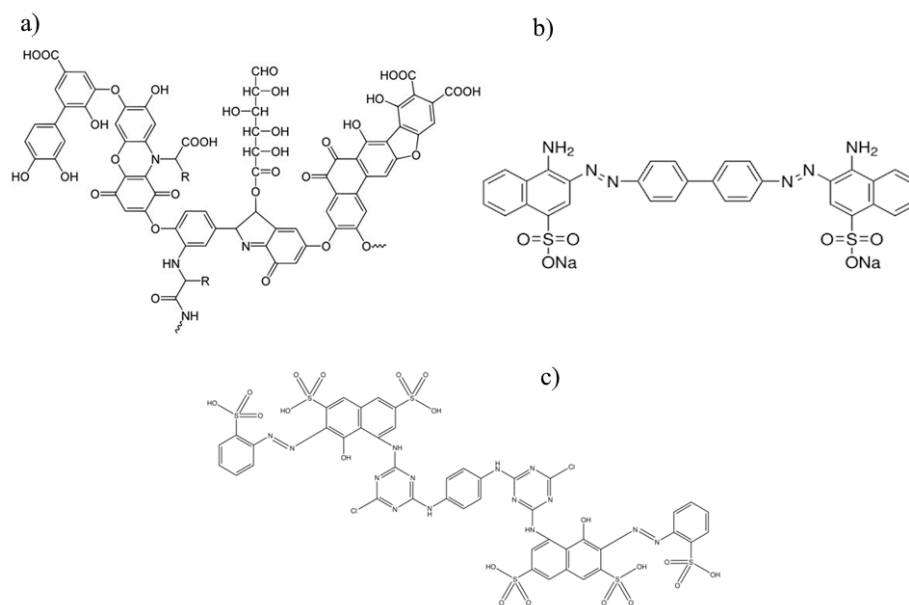


Figure 2.1: Molecular structures of Humic acid (a), Congo Red (b), and Reactive Red 120 (c) [135]

2.2 Instrumental analysis

2.2.1 Inductively Coupled Plasma Optic Emission Spectroscopy (ICP-OES) and inductively coupled plasma mass spectrometry (ICP-MS)

Metal concentrations in liquid wastewater samples were measured using inductively coupled plasma optical emission spectrometry (ICP-OES, Spectro Genesis - AMETEK, Berwyn, PA, USA) following the APAT-IRSA-CNR 3020 standard [136], while metal concentration in leaching tests samples was measure using Inductively coupled plasma mass spectrometry (ICP-MS, NexION 350D - Perkin Elmer, Waltham, MA, USA) following the U.S. EPA 6020B method [137].

2.2.2 Ultraviolet-Visible (UV-Vis) spectroscopy

The concentration of CR and RR120 in water samples was determined by UV-Vis spectroscopy (Lambda2S, PerkinElmer) at wavelengths characteristic of each dye (i.e., 580 nm for CR and 520 nm for RR120) [138]

2.2.3 Total Organic Carbon (TOC) analysis

TOC analysis was carried out using a Shimadzu TOC-5000 APC, using 680 °C catalytic combustion with NDIR detection to measure both total carbon (TC) and inorganic carbon (IC) [139].

2.2.4 LC-MS for PFAS analysis

The analysis of the samples was conducted on 1mL samples centrifuged at 9000 rpm. The samples were then diluted with a 10%MeOH-water mixture. To 300μL of this solution, 6μL of the internal

standard was added, and the samples were analyzed by LC-MS/MS (Compact qTOF (Bruker Daltonics, Bremen, Germany) coupled with a Bruker UHPLC system (Elute Plus)) following U.S. EPA 533 method [140].

2.2.5 Ionic chromatography

Ion chromatography (IC) was used to measure anions (nitrates, nitrites, chlorides, and sulfates) following the APAT-IRSA-CNR 4020 [141] standard, while ammonia analysis was performed according to the ASTM D6919-17 [142] standard.

2.2.6 pH

pH of water and wastewater samples was measured using the APAT-CNR-IRSA 2060 standard [143].

2.2.7 Chemical oxygen demand (COD)

Chemical Oxygen Demand (COD) was measured according to APAT CNR IRSA 5130 Man 29 2003. Specifically, potassium dichromate was added to the water sample, which was then heated in an oven at approximately 150°C for two hours to facilitate the oxidation of organic matter. After the heating period, the sample was allowed to cool. The remaining unreacted oxidizing agent was then measured using a colorimetric method. COD was calculated based on the amount of oxidizing agent consumed, which indicates the level of oxidizable organic substances in the sample [144]

2.2.8 Percentage of Volatile Substances and Moisture Content

The moisture content was measured according to the analytical method UNI EN 14346:2007 Method A. Specifically, the samples were collected, weighed (P_1), dried in an oven for twelve hours at 105°C, and then weighed again (P_2). In the case of oxidation sludges, the remaining sample was further heated to 550°C for three hours and weighed again (P_3) [145]. The percentage of volatile matter was calculated using Equation 19.

$$(P_2 - P_3)/(P_2 - P_1)/ 100 \quad \text{Equation 19}$$

3. Chapter 3: Trends and characteristics of employing cavitation technology for water and wastewater treatment with a focus on hydrodynamic and ultrasonic cavitation over the past two decades: A Scientometric analysis

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3.1 Introduction

The availability of water resources suitable for human use has become increasingly scarce due to [146] a combination of several factors, such as increased water demand for industrial (e.g., textiles, drugs, and plastics production) [2] and agricultural activities [147] leading to the emission of several classes of chemicals in the environment (e.g., petrochemicals, pharmaceuticals, plant protection products, and fertilizers) [147–151]. This issue is also further complicated by the predicted increase in urban population, which is estimated to reach up to 6.7 billion by 2050, with the consequent increase in both wastewater produced and freshwater water resources needed [152,153]. Since other freshwater sources, such as desalinization processes or rainfall collection, face important technical challenges [26,153,154], this will create a negative feedback loop that perpetuates the cycle of resource depletion [155,156]. Thus, it is fundamental to develop new technologies for managing, treating, and reusing water and wastewater [27].

Among the different approaches and technologies considered for this activity, advanced oxidation processes (AOP), such as vacuum ultra-violet photolysis, ultra-violet photocatalysis, photoelectrocatalysis, ozonation, and cavitation [157–162] are attracting much attention. However, not all AOPs are currently cost-effective at the industrial scale, as they require substantial amounts of chemicals and energy expenditure to operate efficiently [163].

On the contrary, conventional hydrodynamic cavitation (HC) systems do not require the use of additional chemicals and use a significantly lower amount of energy compared to other AOPs, thus showing great potential to be scaled up for industrial use [164].

Even though cavitation has been studied extensively, there is still a distinct lack of knowledge on its uses for water or wastewater treatment. In fact, a preliminary analysis of the Web of Science (WoS) database showed that between 2001 and early 2022, only 166 review papers on this topic were published. Among these works, many reviews were mainly focused on the treatment aspects of cavitation techniques, mostly focusing on the same repetitive material, going through the details of various devices and apparatuses capable of inducing hydrodynamic cavitation in wastewater treatment. In total, 23 reviews were concerned with ultrasonic (ultrasound applications) and 24 with hydrodynamic cavitation.

Moreover, this investigation also showed the lack of scientometric reviews on cavitation processes applied to water-related issues and other environmental applications and on how cavitation technologies can contribute to ameliorating environmental pollution problems.

Additionally, considering the huge number of publications (i.e., 2551 as of August 2022) regarding “cavitation AND (water OR wastewater)” that were added in the last 20 years to the WoS database,

it is a virtually impossible task for researchers and scholars to be acquainted with all these publications, let alone find the main trends and points of focus of this research topic.

To address this lack of knowledge, Chapter 3 reports the paper “Trends and characteristics of employing cavitation technology for water and wastewater treatment with a focus on hydrodynamic and ultrasonic cavitation over the past two decades: A Scientometric analysis”. In this work, a scientometric analysis from 2001 to 2022 was carried out on this topic by investigating the major aspects of the literature published in the WoS database, including publications number and citations, keyword choice, keyword co-occurrence, authors' networks, countries' contributions, major journals, and keywords evolution. To better visualize the obtained data, SNA (social network analysis) was also used [165,166]. Furthermore, this work includes a critical evaluation of the obtained literature trends from an environmental science and engineering perspective to provide a more comprehensive overview of this topic's past and present trends and to support future research.

3.2 Methodological approach

3.2.1 Data collection, screening, and visualization

The bibliometric analysis conducted in this study was carried out by applying the following process:

i) selecting and acquiring the most dependable database and software, ii) identifying keywords that are pertinent to the subject matter (i.e., keywords related to cavitation, water, and wastewater treatment) iii) narrowing the search results based on document type, publication date, and scientific categories, iv) exporting the data obtained to other literature-managing tools by using the Web of Science (WoS) exporter for further analysis, v) visual representing the data utilizing social network analysis (SNA), and vi) interpreting the visualized data.

The Web of Science database was selected as it is one of the most powerful and dependable repositories of scientific papers [167,168]. Additionally, WoS includes dedicated tools (e.g., InCite) to assess bibliographical data in various formats, which facilitates the process of removing irrelevant publications. Furthermore, the Web of Science platform is compatible with various literature analysis tools ([169,170]), such as “InCite” and “Visualization of Similarities” (VoS), that were selected for this work.

In detail, in August 2022, literature data from the Web of Science was compiled and refined. The primary search was carried out using the “advanced search tool,” opting for “cavitation OR ultrasonic irradiation” as keywords to group the various expressions used to describe cavitation since it is the main focus of the current work. Specifically, this search was carried out using the “Topic search” option, where the search is conducted in “title, abstract, author keywords, and keywords plus”.

To limit the selection of publications to the ones related to water and wastewater treatment, additional conditions were used by applying the “All fields” option with the search string “water treatment OR wastewater treatment OR hydrodynamic cavitation OR ultrasonic cavitation.” This second string was added to the first with the AND operator. In detail, the “All Fields” allows one to search among all the possible fields of entry that might possess the desired keyword. The keywords “hydrodynamic cavitation” and “ultrasonic cavitation” were added to ensure that all works related to these technologies were included since their most relevant applications are usually related to environmental applications [171,172].

The search was further refined by period selecting all works from 2001 to August 2022, then only “Articles,” “Review articles,” and “Early Access” were selected by using the “Document Type,” thus excluding “proceeding papers,” “meeting abstracts” and other less relevant categories from the results. Moreover, the final results were refined based only on scientific categories deemed relevant to environmental applications, as reported in Table S1, thus obtaining 2551 articles.

Furthermore, GraphPad Prism (Version 9) and Microsoft Excel (2022) were used to visualize the data and results obtained in this work.

3.2.2 Scientometric analysis

The scientometric analysis was carried out as a six-tiered process by applying the following steps: i) analyze the language and article distribution, which determines the dominant publication languages and the percentage of each article type (articles, review papers, etc.), ii) study the publication growth trend to explain the changes occurring in the citations and number of publications, iii) investigate the major scientific categories that classify the articles’ main topics to illustrate which are the main fields of study regarding cavitation (e.g., environmental sciences, environmental chemistry, etc.), iv) analyze the contribution of each country to elucidate the geographical distribution of the research effort on cavitation, v) determine which are the journals more prolific in publishing cavitation-related articles, vi) carry out keywords, and cluster analysis to scrutinize the main keywords related to cavitation environmental applications and study how the obtained clusters of keywords are grouped (i.e., cluster stance).

3.2.3 Keyword co-occurrence analysis and keyword evolution visualization

Keyword co-occurrence analysis, a network mapping method focused on identifying the knowledge components, knowledge structure, and research trends based on the publications’ keywords [173], was carried out by applying the approach reported by [174]. Furthermore, all occurrences of keywords that could be considered synonyms were changed to the most common term (e.g., wastewater, waste-water, and wastewater), then only the 100 most used keywords were selected.

Cluster analysis of the results of the keyword co-occurrence search was carried out following the approach reported by de Toledo and co-workers (2019) [175] by means of the Visualization of Similarities (VOS) software. In addition, the VOS viewer program was used to visualize the network, and the "scale" and "size variation" were applied. In detail, each node represents a keyword, and the size of the circle is directly proportional to the number of publications using each keyword, while the thickness of the lines connecting them increases with the number of co-occurrences.

Keyword evolution visualization is a process where the occurrence of the keywords present during the investigated period (i.e., from 2001 to August 2022) is quantified for each year. In detail, the VOS software was used to select the occurring keywords for each year, and the number of occurrences of the 30 overall most present keywords was plotted in a stacked bar diagram with the Excel program.

3.2.4 Social network analysis

Social network analysis (SNA), a technique based on graph theory that analyzes the social networks present in the dataset [176,177], was applied to visually represented data and the bibliometric network through "Visualization of Similarities" (VoS) software (version 1.6.18) following the approach reported by Wojcik et al. (2021) [178]. In detail, the nodes in the network include journals, publications, citations, co-occurrences, co-authorships, and countries, which can help characterize these intricate connections.

3.3 Results and discussion

Cavitation technologies have shown great potential in many water and wastewater treatment applications, but the lack of comprehensive information on past and present trends in this field of research is still a challenge. For this reason, in this work, a scientometric analysis was carried out on the works focused on this topic published in the last 20 years. In particular, publications number and citations, keyword choice, keyword co-occurrence, authors' networks, countries' contributions, major journals, and keyword evolution were investigated, and the results are reported below.

3.3.1 Language and document type analysis

The main language and document type of the publications regarding a specific topic can give essential information on the scientific community involved and, therefore, on the future trends of the research. In detail, as illustrated in Figure 3.1, the results show that the majority of the selected publications are composed of articles (91.67%), followed by review papers (7.08%) and early access (1.24%). The results also showed that, as expected, most of the articles (i.e., 2135) were in English, while the other two most used languages, Chinese and Russian, with 29 and 8 articles, respectively [179,180]. The presence of Chinese as the second most used language can be attributed both to a large number of

contributing researchers and also to the polices promoted in China that favor the publication of national scientific journals [181].

Furthermore, while similar policies are also present in the Russian academic system [182], albeit to a lesser degree, to better understand why Russian was the third most present language, the search results were further refined to elucidate the recent trends (i.e., publications in Russian from 2018 to August 2022). Among these publications, the main topics covered were related to “cavitation application in mines” and “oil contaminated sites” [183,184], related to the fact that the mining sector (e.g., coal, gold, iron, and nickel) is one of Russia’s key economic sectors, with an estimated value of 75 trillion dollars [185]. In particular, several of the most difficult problems related to operating mines, such as waste deposit to the purification of minerals in floatation tanks, can be facilitated using cavitation methods. Furthermore, Russia’s oil and gas sectors comprise 40% of its GDP [186]. Thus, it is logical for this country to invest in research related to cavitation and its environmental applications.

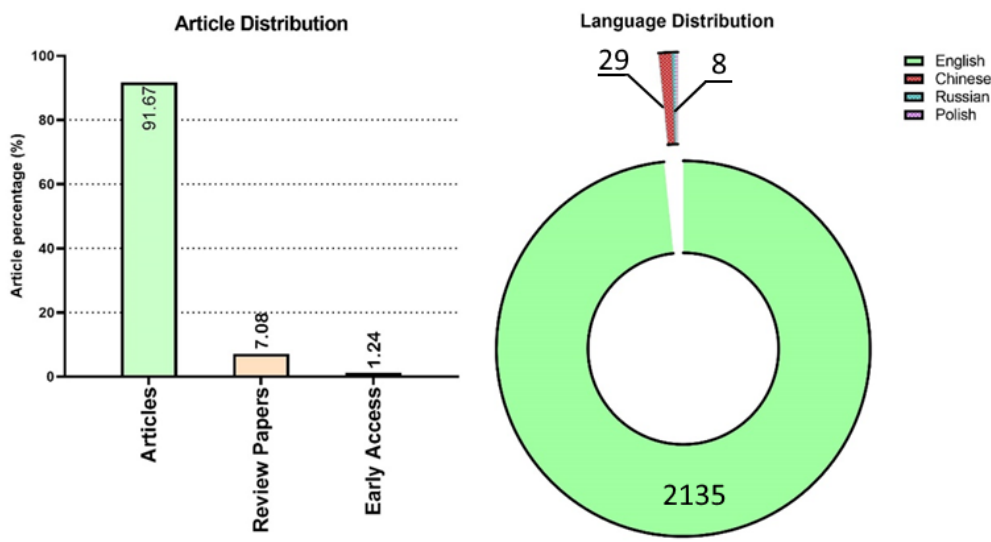


Figure 3.1: Global and language distribution of articles for cavitation and its application in water and wastewater treatment

3.3.2 Trend analysis of publications growth

The evolution of the number of articles and citations regarding the topic of cavitation is essential information to understand and clarify the number of resources dedicated to this field in the past and the prospects for future research.

As shown in Figure 3.2, the results showed that the number of publications related to cavitation in the field of water and wastewater treatment had been steadily rising during the investigated period, as shown by the increase in the number of publications per year (TP), culminating with the maximum reported for 2021 Together with the similar trend showed by the number of citations (TC), this showed that cavitation-related topics have been receiving a considerable and increasing amount of attention.

Conversely, the decrease in studies and citations found for 2022 can be related to several factors, such as that the year 2022 is still on-going, that the indexing of citations requires time to be automatically computed by online repositories and that the most recently published articles have yet to have the chance to be cited.

Moreover, as highlighted in Figure 3.2a, the rate of increase in the number of citations was not constant during the investigation period but showed a significant increase after 2017. To investigate this finding, the authors calculated the ratio between the number of articles and review papers (i.e. $\frac{\text{articles}}{\text{reviews}}$), showing that in the period 2012-2016, review papers had a smaller share with respect to the period between 2017 and 2021 (with a ratio of 17 and 12, respectively). This difference can be considered an important contributing factor to the increase in citation numbers found after 2017 since reviews are among the most cited publications to report on a topic's state of the art in the introduction section of many papers. To further investigate the reasons for the citation increase found after 2017, the “Analyze Results” WoS analytical tool was used to determine if there was any significant change in the scientific categories related to the publications on “cavitation.”

Furthermore, the maximum number of citations was reached in 2021; thus, an investigation of the main topics was carried out. The majority of the most cited articles in 2021 referred to the Chemical engineering category (95 articles), showing research focusing on innovative ways to implement various types of cavitations for wastewater treatment, such as the work by Kim et al. (2021) [187] that utilizes hydrodynamic cavitation for bacterial disinfection during microalgal biomass production. Furthermore, other uses of cavitation techniques, not solely focusing on water treatment but also on enhancing the effectiveness of energy recovery processes, were also present, such as using a cavitation reactor to enhance biodiesel fuel production [188].

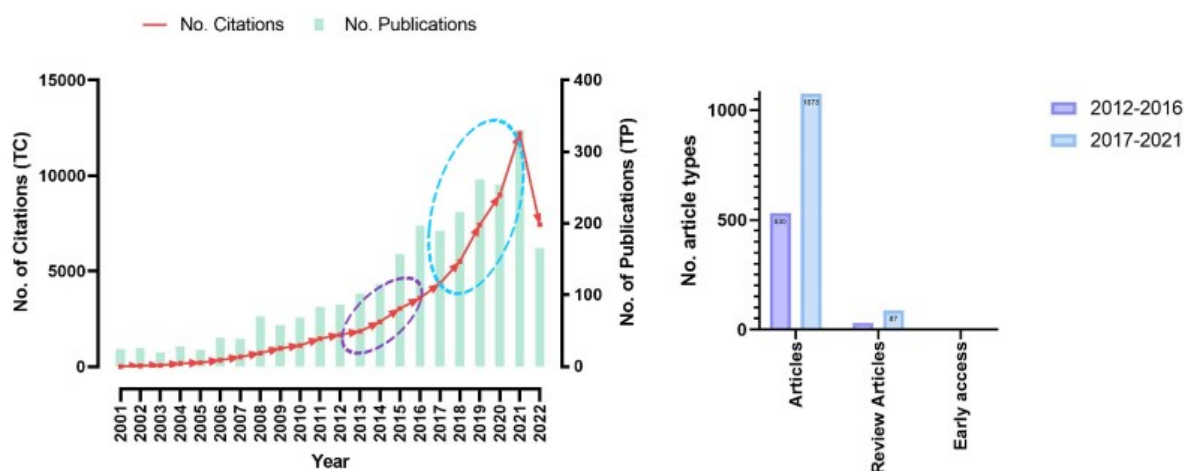


Figure 3.2: a) Annual number of publications and citations for cavitation. The purple and light blue circles highlight the 2012-2016 and 2017-2021 periods, respectively. b) Comparison of document type between 2012-2016 and 2017-2021.

3.3.3 Subject categories analysis

Cavitation can be considered a multidisciplinary topic since many of the several technologies used to induce cavitation phenomena, including hydrodynamic and ultrasonic cavitation, have various environmental, physical, chemical, and technical implications. Investigating which are the scientific categories where most of the research is focused and how this can change with time is fundamental to obtaining a clear picture of how scientific progress on the topic of cavitation is evolving.

In detail, the InCite tool of WoS was used to group the selected publications by their main scientific categories. The results showed that cavitation is a very multidisciplinary topic, with publications spanning up to 25 distinct topics, 11 of which are relevant to our study due to their connection to the environmental application of cavitation processes (Figure 3.3).

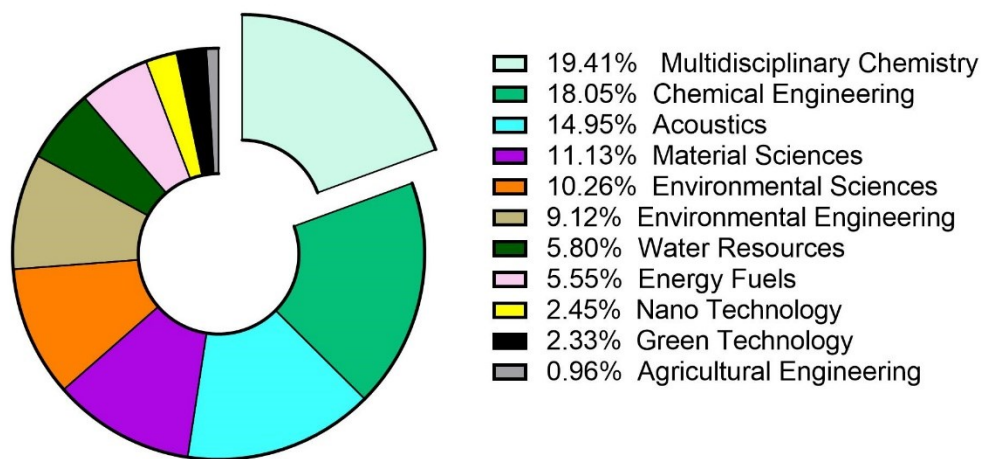


Figure 3.3: The nine most relevant scientific categories grouping the publications selected in this work

The most prominent categories found in this study were Multidisciplinary Chemistry and Chemical Engineering, with a share of 19.41% and 18.05% of the total publications, respectively. Moreover, a substantial overlap was found between “Environmental Sciences” and “Environmental Engineering”; thanks to their relevance to this article's aim, these two categories were extensively analyzed. To find the most recent trends in those two scientific categories, the publications belonging to these two categories from 2018 to August 2022 were selected. The results showed that the studies belonging to the Environmental Engineering category were mainly concerned with the construction and design aspects (e.g., modeling of the cavitation process and potential enhancement of existing cavitation devices), as is the case with the work of Dutta et al. (2021) [189], which strives to offer strategies to enhance venturi tubes for achieving better results in hydrodynamic cavitation.

Additionally, some articles aim to solely model the cavitation phenomena itself, as in the study by Sarvothan et al. (2019) [190] on the modeling of vortex-based hydrodynamic cavitation reactors. Furthermore, this category also included publications where other facets of cavitation were

considered, such as the work by Das et al. (2021) [191], where, in addition to modeling the cavitation process employed, a cost analysis was also carried out. By analyzing the topics studied in this scientific category, it could also be seen that hydrodynamic cavitation was much more studied than acoustic cavitation, and this could be ascribed to the fact that hydrodynamic cavitation is easier to both model and apply to scaled-up systems than acoustic cavitation [192–195].

The results of the in-depth analysis of the publications within the Environmental Sciences category showed that most were focused on the application of cavitation for the treatment of various types of wastewater, such as the work reported by [196] and [197] which focused on the treatment of textile and petroleum refinery effluent by applying hydrodynamic cavitation, respectively. In addition, several works also reported on novel processes for the treatment of less studied contaminants, such as the work by Honda et al. (2021) [198], which employs ultrasonic cavitation to inactivate algae and plankton, and the work by Hao et al. (2021) [199] who focused on ultrasonic-assisted cavitation in the process of microalgae oil extraction for hydrogen production.

Among the scientific categories shown in Figure 3.3., Nano Technology (i.e., nanoscience and nanotechnology) accounted for only 2.45% of the publications selected in this work. However, its importance showed a significant increase from its first appearance in 2008 (i.e., 3 articles per year) to 2019 (i.e., 12 articles per year and 221 citations), thus making its ranking among the scientific categories related to cavitation increase from 16th place to 9th place. This indicates a promising trend in the growth of this research. Therefore, an in-depth analysis of the works belonging to this scientific category was carried out.

In particular, the most recent articles in 2022 were related to ultrasonic cavitation. The article by Choudhary et al. (2022) [200] describes the use of ultrasonic cavitation for the pretreatment of a sucrose hydrothermal carbonization (HTC) precursor solution to control the size of the synthesized carbon nanospheres (CNSs). The combination of cavitation methods and nanotechnology also has a wide variety of other applications, such as in the article by Basov et al. (2022) [201], where silver nanoparticles (AgNPs) were obtained by cavitation-diffusion photochemical reduction, and their antibacterial activity on different surfaces was investigated during cycling freezing. The diversity and applicability of the many combinations of nanomaterials and cavitation are the main reasons we believe this category will be among the top 6 categories in the years to follow, particularly regarding environmental applications.

The Green Technology category (i.e., green and sustainable technologies) has also shown substantial growth during the investigated period. In detail, this category appeared in 2009 (1 article), but it did not receive significant attention until 2015, after which it experienced an exponential increase in the number of citations from 20 in 2015 to 524 in 2021. The articles in this group are quite multifaceted,

for example, on applying cavitation technologies and the energetic efficiency of cavitation processes. For example, Zieliński et al. (2019) [202] reported cavitation-based pretreatment for liquid wastes in a small-scale agricultural biogas plant to augment biogas production. This shows the potential of using cavitation in real-scale applications to reduce the environmental impact of energy production from organic waste, thus opening a new market and occupational opportunities in different fields. Moreover, Albanese et al. (2019) [203] focused on how hydrodynamic cavitation can be used to reduce the energy consumption of a process to produce biochar characterized by high porosity and surface area. A similar study was also carried out by Desikan and coworkers, who reported using hydrodynamic cavitation to decrease the energy needed for biomass pre-treatment for bio-ethanol production [204]. Furthermore, in the work of [205], low-level hydrodynamic cavitation was used to assist thermos-alkaline pre-treatment for sludge solubility. These considerations are fundamental for the development of more sustainable cavitation processes. Therefore, we think the importance of the Green Technology category will continue to increase and will play an indispensable role in numerous scholarly publications.

3.3.4 Country analysis

Since the trends of the research focused on a specific topic can be closely related to the policies and funding programs carried out by different countries and institutions [206], investigating the geographic distribution of the publications related to cavitation is fundamental to obtaining a clear understanding of this topic present and future developments.

In detail, Figure 3.4 shows the 18 most prolific countries based on the number of publications regarding cavitation applied to water and wastewater treatment in the last 20 years. In particular, China and India were the two most productive countries, with 683 (i.e., 26.7%) and 431 (i.e., 16.9%) publications, respectively. The third place belongs to the USA, with 204 articles, followed by Russia, Iran, Japan, South Korea, Italy, England, Malaysia, Poland, France, Germany, Turkey, Canada, Brazil, Spain, and Australia. Based on the results shown in Figure 3.4, the most prolific countries include nations from various parts of the globe, ranging from well-developed countries (e.g., Japan and the USA) to developing countries (e.g., Iran and India), while also including some European countries such as Germany, Italy, and Spain.

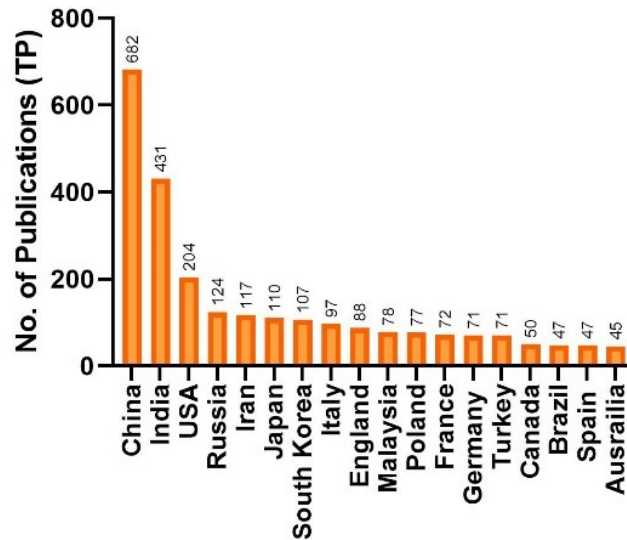


Figure 3.4: Top 18 productive countries from 2001 to 2022

Furthermore, an in-depth analysis was carried out on the specific topics researched in each country, and the following criteria were used to compare the results obtained: i) level of development (i.e., developed or developing country); ii) gross domestic product (GDP); iii) inclusion in the European Union; and iv) number of publications. In detail, based on these criteria, the following countries were chosen for further analysis and divided into the following groups: i) China, USA, and Russia; ii) India, Spain, and Iran; and iii) Germany and Italy.

In the first group, it can be seen that in recent years, China and the USA have moved on from basic applications of cavitation in water and wastewater treatment to more advanced uses. For example, Hung et al. (2021) [207] published a study where hydrodynamic cavitation, along with other techniques, was used to remove polycyclic aromatic hydrocarbons (PAHs) from dredged sediments, while De Alwis and co-workers (2021) [208] investigated the use of hydrodynamic cavitation for the exfoliation of graphene nanosheets. In addition, other advanced applications of cavitation studied in China include improving the functional properties of food proteins and milk pasteurization [209,210] by hydrodynamic and ultrasonic cavitation. Despite the development of those new applications, the results also showed that China is still heavily investing in the use of cavitation methods for wastewater treatment, such as treating tetracycline using HC [211]. The analysis of the publications in Russia for the last 3 years (i.e., ca.14 articles per year) showed a level of interest in the topic similar to what has been found for the USA (i.e., ca.17 articles per year), with research topics ranging from more basic research to the more innovative uses of cavitation. For example, Abramov and co-workers (2021) [212] investigated the simultaneous effect of hydrodynamic cavitation and plasma discharge on the bulk treatment of contaminated water, Matafonova and Batoev (2020) [213] exploited dual-frequency

ultrasounds (DFUS) for the elimination of pathogens and water disinfection, and Thangavelu et al. (2018) [214] studied the use of hydrodynamic cavitation in the delignification of corn cob via hydrodynamic cavitation and enzymatic pretreatment. Since the Russian economy is heavily based on the metallurgy and oil industry, several recent publications are focused on such topics, such as the work by Eskin et al. (2019) [215] on using ultrasonic cavitation in melt processing, which is a part of the metal casting industry.

The second group of countries (i.e., India, Iran, and Spain) was chosen because of their similar GDP levels (2.62, 1.28, and 1.43 trillion dollars, respectively) [216–218] and because they are all facing problems related to water scarcity and contamination. For this reason, the authors wanted to investigate if these have triggered differences or changes in their publications.

In detail, for these three countries, the results showed that many recent articles are concerned with industrial wastewater since environmental regulations are becoming more stringent, restricting the haphazard discharge of chemicals into water reservoirs or other water bodies. For example, in the recent article by Vilarroig et al. (2020) [219], an industrial-scale cavitation device was utilized for the pretreatment of animal breeding waste sludges. Moreover, the results also showed a significant difference in the researcher's focus in these countries on the cost of the studied cavitation processes. In fact, most of the articles where the cost of the investigated process was considered were from Spain and Iran, such as the work from Gallero-Valero and coworkers (2021) [220], who presented an overview of different wastewater treatment costs in the period 1950–2020. Similarly to what has been observed for Spain, a focus on the techno-economic assessment of the proposed cavitation process was found for Iran, as indicated in the publications by Gholami et al. (2021a,b) [221,222], who discussed the costs of using hydrodynamic and ultrasonic cavitation for enhancing biodiesel production. Since in Iran, environmental regulations are quite weak and very rarely applied [223], in the last three years, only very limited numbers of publications on cavitation refer to such standards. However, since one of the major issues for this country is the lack of operational incinerators, many articles focus on applying cavitation to manage industrial wastewater and landfill leachate [224].

The countries considered in the third group (i.e., Germany and Italy) are both parts of the European Union but have significantly different GDPs (1.89 and 3.85 trillion dollars for Italy and Germany, respectively) and different economic situations. These differences could also be seen in the publications produced in these countries. In fact, the research carried out in Italy in the last three years follows the same path as China and the USA, showing great interest in more innovative uses of cavitation, encompassing articles ranging from dye elimination from water [77,225], water disinfection [226], facilitating seed germination [227], and antibacterial treatments for longer food conservation [228]. In particular, further analysis of the studies published in Italy in the last four years

showed they were mainly focused on hydrodynamic cavitation, but the number of works focusing on ultrasonic cavitation has constantly been increasing, showing a promising prospect for the research focused on this topic.

On the contrary, in Germany, most research focused on industrial-scale applications of ultrasonic cavitation for wastewater treatment [229] or the physical processes regulating ultrasonic cavitation and the consequent bubble generation [230]. This shift in focus can be ascribed to the higher industrialization of Germany, which caused a higher demand for technologies focused on large-scale management of industrial wastes compared to Italy and other European countries. From the results, it can be seen that only a limited number of publications in Germany focused on more innovative uses of cavitation on environmental-agricultural topics [231], but this number is likely to increase in the following years thanks to recent shifts in Germany's policies on environmental management [232].

In addition to observing the most recent developments in the field of environment-related cavitation technologies, it is also crucial to understand the motives behind the most prolific countries' long-term publication trends. In particular, in Figure 3.5, we report the results found on the evolution of the annual number of publications on this topic for the eight countries previously discussed in this section.

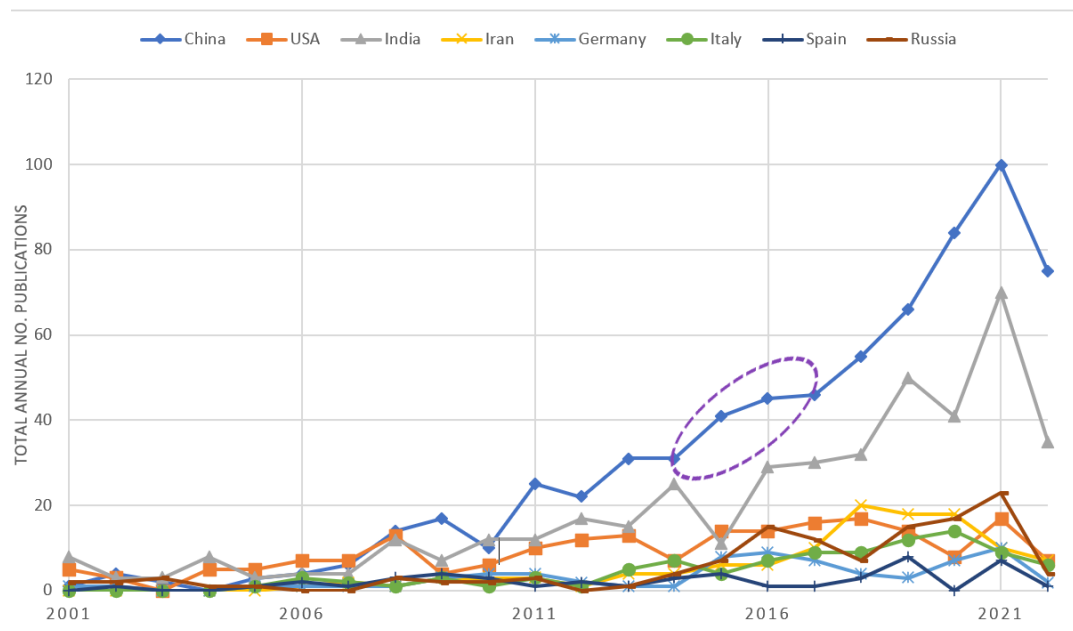


Figure 3.5: Annual trend of the total number of publications for the selected 8 countries.

The country with the highest number of publications in 2021 was China (blue line in Figure 3.5). However, before 2008, the USA (orange line in Figure 3.5) had a higher scientific production on cavitation processes for water treatment. This trend reversal did not depend on a drop in interest in the topic in the USA, which remained fairly constant during the investigation period but on a sudden

increase from the Chinese side. This increase is especially evident after the 2014-2017 period, suggesting the presence of a fundamental shift in China's environmental management policies, particularly regarding water and wastewater treatment. In fact, based on the environmental report by Tianjie and co-workers [233], in 2015, China declared for the first time a red alert regarding air pollution [234–236], and on January 1st of the same year, several more stringent environmental protection laws came into effect. In addition, many efforts directed to better conservation and management of environmental resources and to reduce carbon emissions [237–239] were carried out after the announcement in 2014 by the Chinese government that China would start decreasing carbon emissions by 2030 [240].

As expressed in Figure 3.5, the second largest increase in the number of publications for the investigated period was found in India (grey line in Figure 3.5), especially after 2016 -2018. This can be related to the increased interest in policies regarding the environment observed in India over the last few years. The latest report regarding India's environmental budget [241] shows a 20% increase in the allocation concerning the 2020-2021 period, as well as a clear change of policy on emissions with goals such as net zero emission by 2070 and increasing the share of renewable energy production in India by 50% by 2030.

The results reported in Figure 3.5 show that the number of works published in Iran increased noticeably after 2015 (yellow line in Figure 3.5) due to the more marked acknowledgment of the grave water shortage problems faced by this country [242] and the consequent need of more efficient and affordable water treatment technologies, such as those that can be provided by cavitation. Analyzing the most recent Iranian publications, it emerged that one of Iran's major uses of cavitation (mostly hydrodynamic cavitation) is for the floatation of minerals and their consequent purification since this country's economy is strongly based on the mineral sector [118]. As the industrialization of Iran, and therefore the need for more specialized wastewater treatment facilities, is steadily increasing, research is also expected to increase. However, due to the recent economic challenges and sanctions, the development of research related to cavitation and its uses in water treatment may face several challenges, as also shown by the decreasing number of publications in the last four years.

As for the other countries shown in Figure 3.5, the difference in the number of articles on cavitation related to water and wastewater treatment was less significant. Nevertheless, thanks to the many results showing the potential of cavitation for industrial-scale applications related and unrelated to the environment, other countries will soon follow the same trend and show more interest in taking advantage of cavitation and its various uses.

Other than investigating the general publication trends of the most prolific countries, it is also fundamental to investigate which are the trends in international collaborations to obtain a clear picture

of the direction that the research on cavitation related to water and wastewater treatment will take in the next few years. For this reason, a co-authorship network analysis was carried out among the top 19 countries with more publications on the topic focus of this work, and the results are presented in Figure 3.6.

In detail, co-authorship networks can be considered social networks made up of scholars who collaborate on a certain subject, thus constituting the nodes of the nodes of the network [243]. The level of connection between nodes (i.e., researchers) is defined as link strength, and it is proportional to the number of shared works. In Figure 3.6, this can be visualized by the thickness of the lines in the diagram.

The results showed that the strongest collaboration networks had been established between the USA and China, as they are also among the biggest contributors to this topic. Furthermore, close collaboration was also found between Iran and Turkey, as well as between Italy, Spain, and France, owing to the similarities between their socio-economic systems and their environmental regulations. The country with the most collaboration links in the network was India (31 links), which is higher even than the links established by China (28 links), thus showing that the sole number of publications (682 and 431 for China and India, respectively) should not be the only factor to be considered when assessing the contribution of a country to the international research landscape on a topic. Furthermore, the different policies these two countries applied greatly influenced this difference in the number and importance of international connections. This high number of international collaborations found in India can also be related to increased resources available for research focused on environmental issues, as explained above. These findings highlight the central role that India will continue to play in the field of research related not only to water treatment by cavitation processes but also to the larger landscape of environmental management.

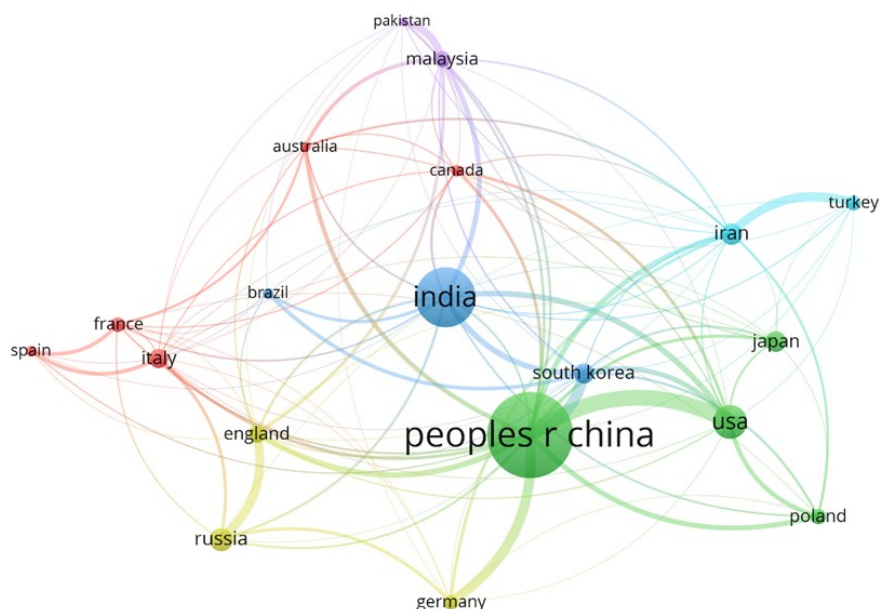


Figure 3.6: Diagram showing the network of collaborations between the 19 most prolific countries on cavitation based on co-authorship relations. The thickness of the lines connection each node denotes the number of collaborations, while the radius of the circle indicates the number of publications for each country.

3.3.5 Journal analysis

Other than the number of publications and citations and their respective trends, also the analysis of which journals have been chosen to publish the research carried out on cavitation applied to water and wastewater treatment can give fundamental information on the importance given to this topic and the future path of this research effort. Impact factor (IF) is one of the most dependable indexes for measuring the quality of scientific journals, and many researchers use it to select which journal they wish to publish their research [244]. Furthermore, a globally applied indicator is also a quartile ranking (QRs), supplied by the WoS citation report. Different databases, including Google Scholar, Scopus, and WoS, utilize slightly different methodologies to generate this impact score, but the results have been mostly consistent across these platforms [245–247].

The results show that the selected publications were published in about 200 journals, but 2 of these accounted for more than 530 publications. In particular, Table S 3-2 shows the 13 most used journals to publish on cavitation related to water and wastewater treatment based on both IF and QR rankings. Among these journals, the Journal of Ultrasonic Sonochemistry had the highest number of publications related to the topic of interest (i.e., 453 articles), with a focus on hydrodynamic cavitation and ultrasonic cavitation. Interestingly, the number of publications for the second most chosen journal (i.e., Desalination and Water Treatment) is about five times less, with 81 articles. This indicates a quite specific focus on ultrasonic cavitation and hydrodynamic cavitation with respect to other cavitation-induced processes, but the recent discoveries on optical cavitation and particle cavitation application and optimization [248,249] will make studies on this topic more common in the future. The results also showed that the journals traditionally focused on environmental chemistry (i.e.,

Chemosphere, J. of Hazardous Materials, J. of Water Research, and J. of Cleaner Production) accounted only for a minor part of the articles (i.e., 96), highlighting that the diffusion of this topic within the environmental chemistry community is still limited, but in the future, this trend will likely increase thanks to the recent developments and rising acknowledgment on water scarcity problems across the world [153,250]. Our bibliographic research also showed that 11 out of 12 selected journals belong to the 1st quartile of their respective scientific categories, highlighting that researchers generally chose high-ranking and eminent journals to publish on cavitation.

3.3.6 Keywords co-occurrence network analysis

Keywords are used to describe the most important aspects and topics of a publication, especially for indexing purposes across the main scientific repositories such as Scopus, WoS, PubMed, and Google Scholar [251]. For this reason, we applied keyword co-occurrence analysis [173,252] to show the most popular study subjects across the topic of cavitation related to water treatment. Among the 8709 keywords that were found, we selected only those that occurred more than 5 times, thus selecting 793 keywords. As explained in section 3.2.3, we selected only the 100 most used keywords, while occurrences of keywords that could be considered synonyms were changed to the most common term. The results are shown in Figure 3.7, where each node represents a keyword, and the thickness of the connecting lines increases with the number of co-occurrences [175].

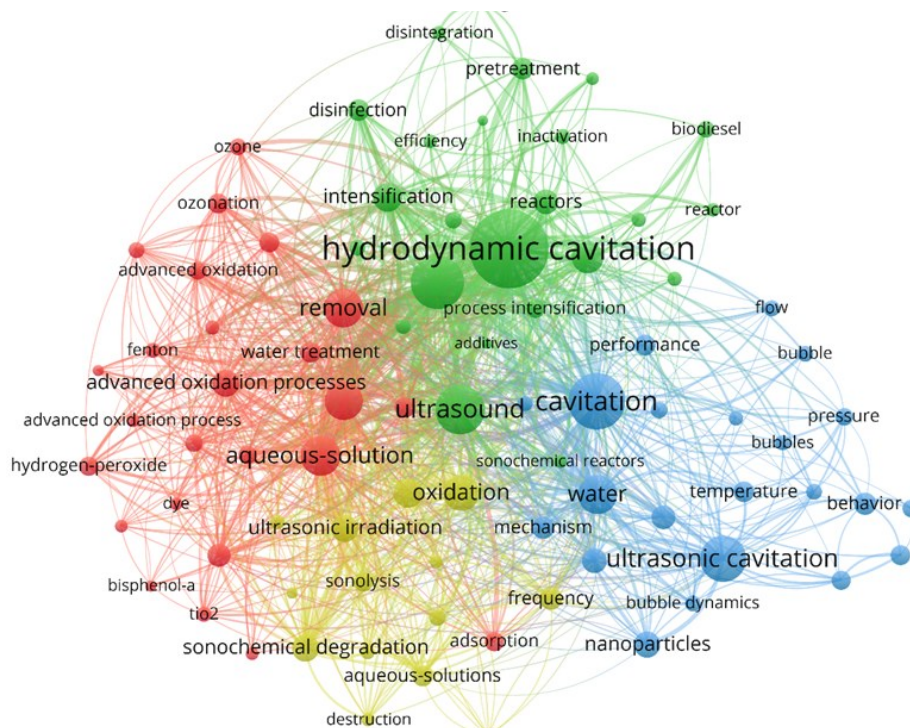


Figure 3.7: Co-occurrence network of the 100 most used keywords. The thickness of the lines connection each node denotes the number of co-occurrences, while the radius of the circle indicates the number of occurrences for each keyword.

Furthermore, each color represents one of the four clusters used to group the different keywords, as reported in Table 3-1.

Table 3-1: Main keywords for each of the four clusters identified.

Cluster (Color)	Top four keywords in each cluster (Occurrences)
1 (Green)	Hydrodynamic cavitation (784); Degradation (400); Ultrasound (356); Optimization (164)
2 (Blue)	Cavitation (447); Ultrasonic cavitation (310); Water (234); Nanoparticles (122)
3 (Red)	Aqueous solutions (247); Removal (236); Wastewater treatment (226); Advanced oxidation processes (126)
4 (Yellow)	Oxidation (203); Kinetics (139); Sonochemical degradation (125); Ultrasonic irradiation (124)

In detail, cluster 1 (green in Figure 3.7) contains three major keywords (i.e., “hydrodynamic cavitation,” “ultrasound,” and “degradation”) that show very strong links between each other, showing a close relationship between the type of process used (i.e., hydrodynamic cavitation and ultrasound-induced cavitation) and the aim of those studies (i.e., degradation of contaminants). Furthermore, these keywords are also connected to other words such as “process intensification” and “intensification,” “reactors,” “efficiency,” and “performance.” This also highlights the current focus on using cavitation-related technologies to increase the performance of conventional or innovative water treatment processes.

Moreover, cluster 2 (blue) is shown to contain keywords related to the study of the mechanisms regulating cavitation phenomena, such as “water,” “mechanism,” “temperature,” “bubbles,” “behavior,” “bubbles dynamics,” “pressure,” and “flow.” Figure 3.7 also shows that a significant part of this research is focused on ultrasonic cavitation due to the fact that the keyword “ultrasonic cavitation” occupies the second biggest node (i.e., it appears in the second largest number of publications) of the cluster and it is strongly connected with the majority of the other nodes in cluster 2. Figure 3.7 also highlights that the “nanoparticles” keyword has been receiving significant attention, especially in relation to “bubble dynamics,” since the dimension of the produced bubbles has been shown to be a fundamental factor in the efficacy of the treatment process [114].

Cluster 3 (red) contains most of the keywords related to water and wastewater treatment (e.g., “water treatment,” “wastewater treatment,” “removal,” and “advanced oxidation processes”). By observing how the keywords describing the contaminants whose removal is being studied relate to each other, indications of the main industrial sectors interested in this research topic can be gained. For example, the close relationship between “dye” and “TiO₂” keywords shows the central role of the textile and food industries in the topic since they are among the major users of such substances [253]. In addition, the many links between the red cluster and the “hydrodynamic cavitation” (blue cluster) confirm the

vast application of this process for wastewater treatment. Conversely, the keyword “adsorption” is almost unconnected from the other keywords in cluster 3 (red) that describe AOP methods, showing great new opportunities for researching this process as a part of integrated water treatment processes that use it in combination with other AOP methods, such as ozonation or cavitation methods. Some works have recently started to explore the combination of these techniques, such as Mohan and Gokul (2022) [254], who combined ozonation and adsorption for the treatment of landfill leachate.

Figure 3.7 also shows the scarcity of links connecting the keywords “water/wastewater treatment,” “advanced oxidation processes,” “hydrodynamic cavitation,” “Fenton,” and “ozonation” with the words “nano particles,” and “bubbles and “cavitation,” indicating that only very few studies have been investigating the combination of those different processes. This opens opportunities in the field of study related to wastewater treatment, thanks to the great adaptability of cavitation processes regarding the different technical challenges that will need to be overcome to guarantee better management of the word water supply.

Cluster 4 (yellow) brings together several keywords (e.g., “oxidation,” “kinetics,” “sonochemical degradation,” and “ultrasonic irradiation”) describing studies focused on studying aspects related to pollutants’ degradation kinetics with cavitation-based processes, and especially ultrasonic cavitation. For example, Wu and co-workers (2007) [255] reported on how the upstream pressures of the orifice plate, the cavitation number, and the solution temperature affect phenols’ degradation kinetics.

3.3.7 Keywords evolution analysis

From the analysis reported in section 3.3.6, it can be seen that in the last 20 years, the research focused on cavitation-based processes for water and wastewater treatment has evolved into a complex and interconnected network, but to better identify its future trends, a focus on how the use of the selected keywords has changed in time is necessary. In particular, Figure S 3-1 shows how the 30 most present keywords were used during the investigated period.

The results showed that several keywords considered relevant in 2001 (e.g., “microbial cell disruption using cavitation”) are no longer relevant, while others have gained more importance, such as “hydrodynamic cavitation” and “cavitation.”

In particular, the keyword “cavitation” has been gaining much interest since it is more general and can incorporate many research aspects, from investigating the cavitation phenomena to modeling aspects and environmental applications. Furthermore, thanks to innovations and discoveries in the last few years, especially after 2016, the use of “hydrodynamic cavitation” has significantly increased. This shows how, after investigating the more general aspects of cavitation, more diverse and specific processes emerged in this field and how this was reflected in the choice of keywords over the years. Moreover, “hydrodynamic cavitation” was the most used keyword in 2021, as well as a fundamental

node in the co-occurrence network discussed in section 3.3.6. Thus, it is expected that this topic will be the focus of an increasing number of publications in the following years.

Table 3-1 shows how the competition between ultrasonic and hydrodynamic cavitation evolved over the years, with 800 and 300 total occurrences, respectively. In detail, the use of “ultrasound,” “ultrasonic cavitation,” and “hydrodynamic cavitation” was relatively constant until 2012 and then had a marked increase in 2014 (Figure S 3-2), thanks to the discoveries on the multiple possible applications of these processes, such as for water treatment [256–258], microbial inactivation, and food processing [259–261]. However, the increase in “hydrodynamic cavitation” was much more significant, thanks to the discovery that HC could be more easily used as a pretreatment method integrated into other processes [196,197,256,262,263] than ultrasonic cavitation. The application of ultrasonic cavitation showed a more contained increase due to the several technological, energetic, and cost limitations discovered for its application [264], but thanks to the discoveries made on how to limit the effects of these problems [125] the focus on this topic will increase, especially in countries where there are already some advanced applications (e.g., Germany).

The results also show that while researchers are pursuing cavitation methods, their water and wastewater treatment applications are not on the same level as those focused on the more generic aspects of cavitation. In fact, the keywords “water treatment” and “wastewater treatment” have significantly fewer occurrences (i.e., between 250 and 300) than the three main keywords (i.e., “cavitation,” “ultrasonic cavitation,” and “hydrodynamic cavitation”). Nevertheless, their utilization has been increasing from 2018 and 2017, respectively (Figure S 3-2), showing that the interest in this topic will likely increase in the future.

The keyword analysis results also show the use of cavitation processes during the past two decades for water and wastewater treatment as a stand-alone method is limited [193,265], and such industrial-scale uses of these technologies are not pervasive. In fact, they are mostly combined with other keywords indicating other processes, such as membrane filtration and chemical oxidation [266,267]. Consequently, future research will use processes combining cavitation and other technologies.

The results reported in Figure S 3-2 also highlight the evolution of the keywords “nanotechnology” and “nanobubbles,” which appeared in 2008 and 2011, respectively. Both of these keywords were used to a limited extent until 2013 when their importance increased significantly thanks to the focus on nanoscale materials and processes caused by the several innovations in the field of nanoscience that characterized that period [268,269]. In particular, Figure S 3-2 shows that the use of “nanoparticles” increased much faster than the use of “nanobubbles,” indicating that the research has been focused for the most part on the use of cavitation processes for the synthesis of nanomaterials [164,200,208,270,271]. While the trend shown in Figure S 3-2 indicates that the use of nano-sized

bubbles in cavitation processes is still in its infancy, the occurrence of “nanobubbles” started to significantly increase in 2017, corresponding to the shift from studying surface nanobubbles (that cannot be utilized for water treatment and other environmental applications) to bulk nanobubbles [272–274]. Nevertheless, the topic of nanobubbles concerning water treatment has not been extensively investigated, but very promising results have been reported by several authors [27,114,118,275] indicate its potential use for water treatment, especially with hydrodynamic cavitation. In addition, several other applications of nano-sized cavitation have also been reported for other fields, such as the study by Xuan and Nastac (2017) [276] on materials manufactured by also using ultrasonic-induced nanobubbles.

The results reported in Table 3-1 and Figure S 3-2 show that only a limited focus was given to “energy” and “energy efficiency” in the cavitation field. However, with the rising importance of each process’s sustainability, researchers are expected to give more importance to these concepts in their studies.

In this section, the authors gave an overview of the trends regarding the most important keywords used in the field of cavitation-based water treatment processes, but the trends of the rest of the keywords reported in Table 3-1 can be seen in Figure S 3-3.

3.3.8 Concluding remarks and future directions

In the present work, a scientometric analysis of the bibliography (i.e., 2551 publications) related to the application of cavitation processes for environmental applications, especially water and wastewater treatment, was carried out, and a critical evaluation of the present and future trends of this field of research were provided. Considering the last 20 years, the number of publications and citations showed an incremental trend, proving the increasing significance of these applications of cavitation related to environmental and water management. The increasing number of citations was attributed to the multidisciplinary nature of the topic and to the many applications found in many fields, both directly and indirectly related to the environment.

The languages used in most articles, after English, were Chinese and Russian, demonstrating the effects these countries’ policies had on the development of the research on this topic, as well as the focus on studies related to their mayor economic sectors, such as oil extraction and mining.

The most prominent scientific category for the topic of water treatment-related cavitation processes was “Multidisciplinary Chemistry” with 19.4% of the overall publications, but “Environmental Sciences” and “Environmental Engineering” accounted for a combined 19.3 %, showing a positive trend toward the use of cavitation for environmental applications.

Country analysis revealed that three groups based on GDP similarity, state of development, and publication behavior could be identified among the most prolific countries. The publication behavior

indicated that developed countries have the tendency to pursue more innovative uses of cavitation, while developing countries are more interested in implementing less complex applications in their major industrial sectors, in addressing their water shortage problems, or they are not in the situation to pursue more innovative applications due to economic limitations. As representatives, China, the USA, Russia; Iran, Spain, India; Germany, and Italy were chosen to be investigated.

Journal analysis highlighted that most studies were published in the “Ultrasonic Sonochemistry” journal, followed by “Desalination and Water Treatment.” Furthermore, while the journals traditionally focused on environmental chemistry accounted only for a minor part of the articles, this trend will likely increase thanks to the recent developments and rising acknowledgment of water scarcity problems worldwide. Most of the selected journals belong to the 1st quartile of their respective scientific categories, showing that researchers generally chose high-ranking and eminent journals to publish on the topic of cavitation.

Keywords co-occurrences and cluster analysis showed that four clusters could be identified, referring to improving the performance of cavitation-related technologies, the mechanisms regulating cavitation phenomena, the uses of cavitation specific for water and wastewater treatment, and studying aspects related to pollutants’ degradation kinetics.

Keywords evolution analysis showed that the use of both “hydrodynamic cavitation” and “ultrasonic cavitation” has seen a significant increase, but ultrasonic cavitation was less studied due to its technological, energy, and cost limitations, but thanks to new advancements on this topic the focus on this process will increase, especially in countries where there are already some advanced applications (e.g., Germany).

The occurrence of keywords related to nanoscience and water treatment showed significantly in the last five years, showing the increased interest in applying cavitation-based processes in these fields. These trends will likely continue to grow in the future thanks to new discoveries on the topic of nanobubbles and on the coupling of cavitation with other AOP. While only a limited focus was given to topics related to “energy” and “energy efficiency” in the field of cavitation during the investigated period, with the rising importance of each process’s sustainability, the attention given to these concepts will increase in the future.

The publications analyzed in this work showed that there had been significant developments in bringing attention to the industrial uses of hydrodynamic cavitation for water and wastewater treatment, especially in coupling this process with other processes such as AOP, electro-Fenton, and photochemical reactions.

In the case of ultrasonic cavitation, the studies found for this work highlighted that industrial-scale applications are generally more energy demanding, thus presenting many challenges for future

researchers to obtain more sustainable processes involving this technology. Furthermore, the combination of hydrodynamic and ultrasonic cavitation as a single process will offer many opportunities for future research.

Based on the results of the reported scientometric analysis, we showed that the future directions for the topic of cavitation-related water and wastewater treatments would probably shift towards more environmentally friendly applications of hydrodynamic and ultrasonic cavitation, as well as towards more green and sustainable approaches, especially considering the potential of this technology to address the increasing demands of water pollution and shortage. Moreover, the research on cavitation will also increase regarding its uses for purposes other than water treatment, such as manufacturing nanomaterials, food production, and medicine.

3.4 Supporting Information

Table S 3-1: Relevant and irrelevant scientific categories used in this work to filter the obtained results from the bibliographical search

Relevant categories	Irrelevant categories
Chemistry Multidisciplinary, Engineering Chemical, Acoustics, Materials Science Multidisciplinary, Environmental Sciences, Engineering Environmental, Water Resources, Energy Fuel, Metallurgy Metallurgical Engineering, Engineering Mechanical, Chemistry Physical, Physics Applied, Biotechnology Applied Microbiology, Food Science Technology, Nanoscience Nanotechnology, Green Sustainable Science Technology, Engineering Manufacturing, Physics Multidisciplinary, Mining Mineral Processing, Engineering Multidisciplinary Chemistry Applied, Polymer Science, Multidisciplinary Sciences, Electrochemistry, Agricultural Engineering, Materials Science Coatings Films, Mineralogy, Chemistry Analytical, Engineering Industrial, Materials Science Composites, Thermodynamics, Pharmacology Pharmacy, Public Environmental Occupational Health, Materials Science Characterization Testing, Agriculture Dairy Animal Science, Marine Freshwater Biology, Ecology, Engineering Petroleum, Nuclear Science Technology, Geochemistry Geophysics, Microbiology, Chemistry Organic, Agriculture Multidisciplinary, Engineering Marine, Environmental Studies, Geosciences, Oceanography, Biology, Fisheries, Chemistry, Inorganic Nuclear, Engineering Ocean, Materials Science Textiles, Plant Sciences, Soil Science Biochemical Research Methods, Biodiversity Conservation, Forestry, Horticulture, Infectious Diseases	Surgery, Limnology, Materials Science Ceramics, Materials Science Paper Wood, Mathematics Applied, Medicine Research Experimental, Nutrition Dietetics, Construction Building Technology, Engineering Civil, Physics Atomic Molecular Chemical, Automation Control Systems, Biochemistry Molecular Biology, Optics, Instruments Instrumentation, Physics Fluids Plasmas, Radiology Nuclear Medicine Medical Imaging, Physics Condensed Matter, Mechanics, Materials Science Biomaterials, Spectroscopy, Toxicology, Computer Science Artificial Intelligence, Computer Science Information Systems , Dentistry Oral Surgery Medicine, Biophysics, Computer Science Interdisciplinary Applications, Physics Nuclear, Chemistry Medicinal, Clinical Neurology, Engineering Aerospace, Imaging Science Photographic Technology, Anatomy Morphology, Cell Biology, Engineering Geological, Immunology, Medical Informatics, Microscopy, Neurosciences, Physiology, Sport Sciences, Astronomy Astrophysics, Dermatology, Education Educational Research, Education Scientific Disciplines, Endocrinology Metabolism, Gastroenterology Hepatology, Geology, Mathematics, Mathematics Interdisciplinary Applications, Medicine General Internal, Obstetrics Gynecology, Pathology or Peripheral Vascular Disease, Remote Sensing, Rheumatology, Robotics, Social Sciences Interdisciplinary, Transplantation, Transportation Science Technology, Urology Nephrology

Table S 3-2: The 13 most used journals to publish on the topic of cavitation related to water and wastewater treatment based on both IF and QR rankings.

Journal	Number of publications
Ultrasonic Sonochemistry	453
Desalinization and Water Treatment	81
Chemical Engineering Journal	59
Separation and Purification Technology	46
Chemical Engineering and Processing Process Intensification	36
Industrial Engineering Chemistry Research	33
Journal of Cleaner Production	31
Advanced Materials Research	27
Journal of Environmental Chemical Engineering	27
Journal of Hazardous Materials	23
Bioresource Technology	22
Chemosphere	22
Wear	22

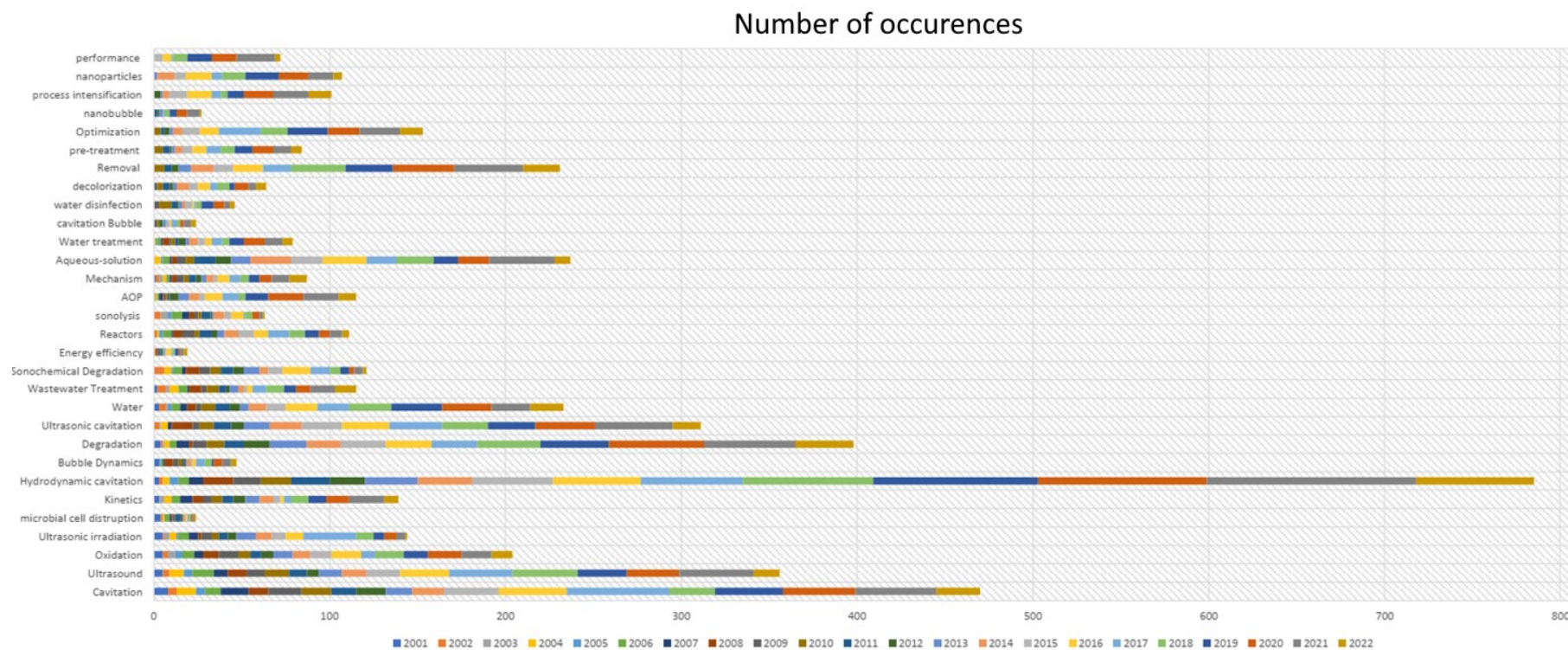


Figure S 3-1: Number of occurrences for each year of the 30 most used keywords during the period from 2001 to August 2022.

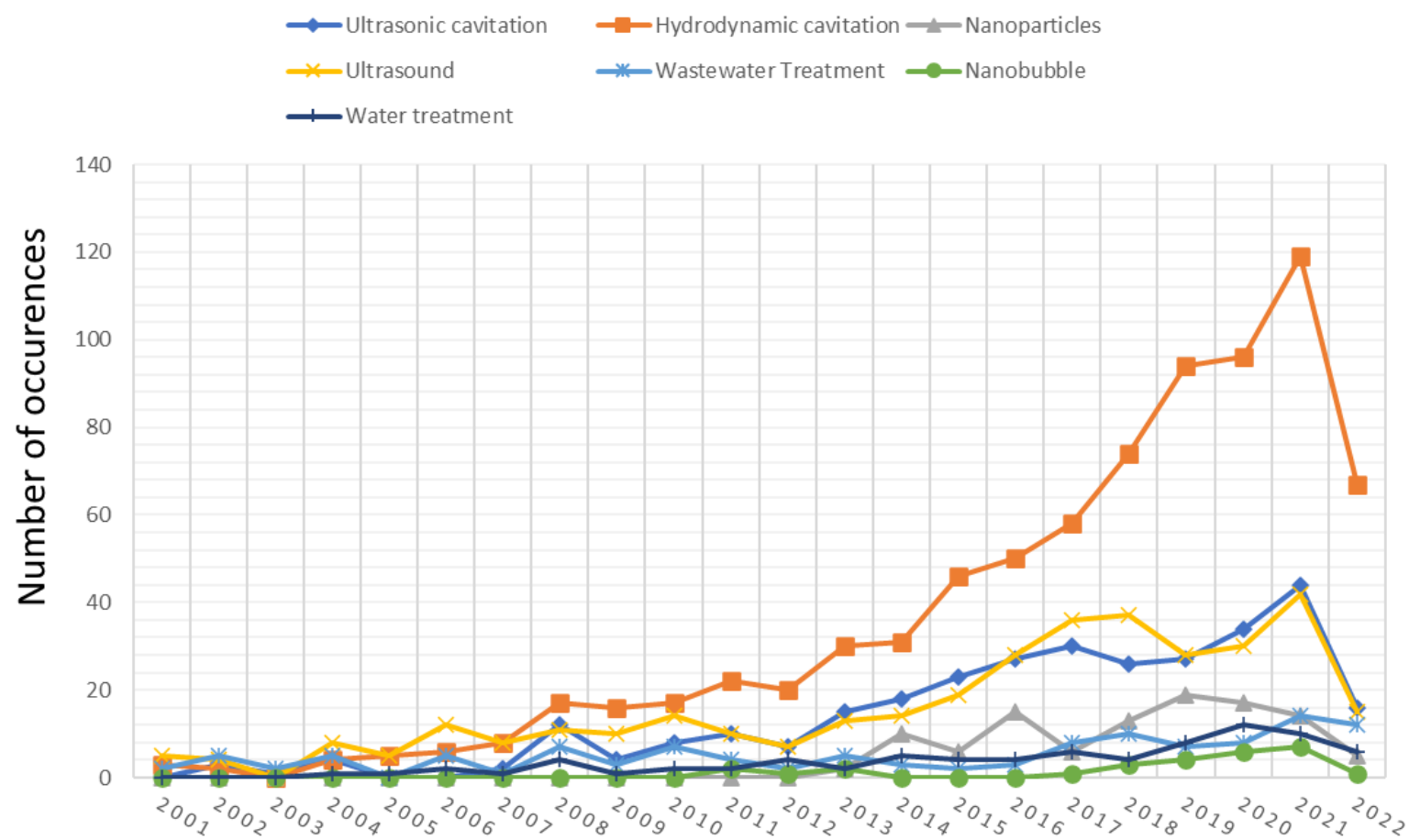


Figure S 3-2: Evolution trend of the most important keywords during the period from 2001 to August 2022.

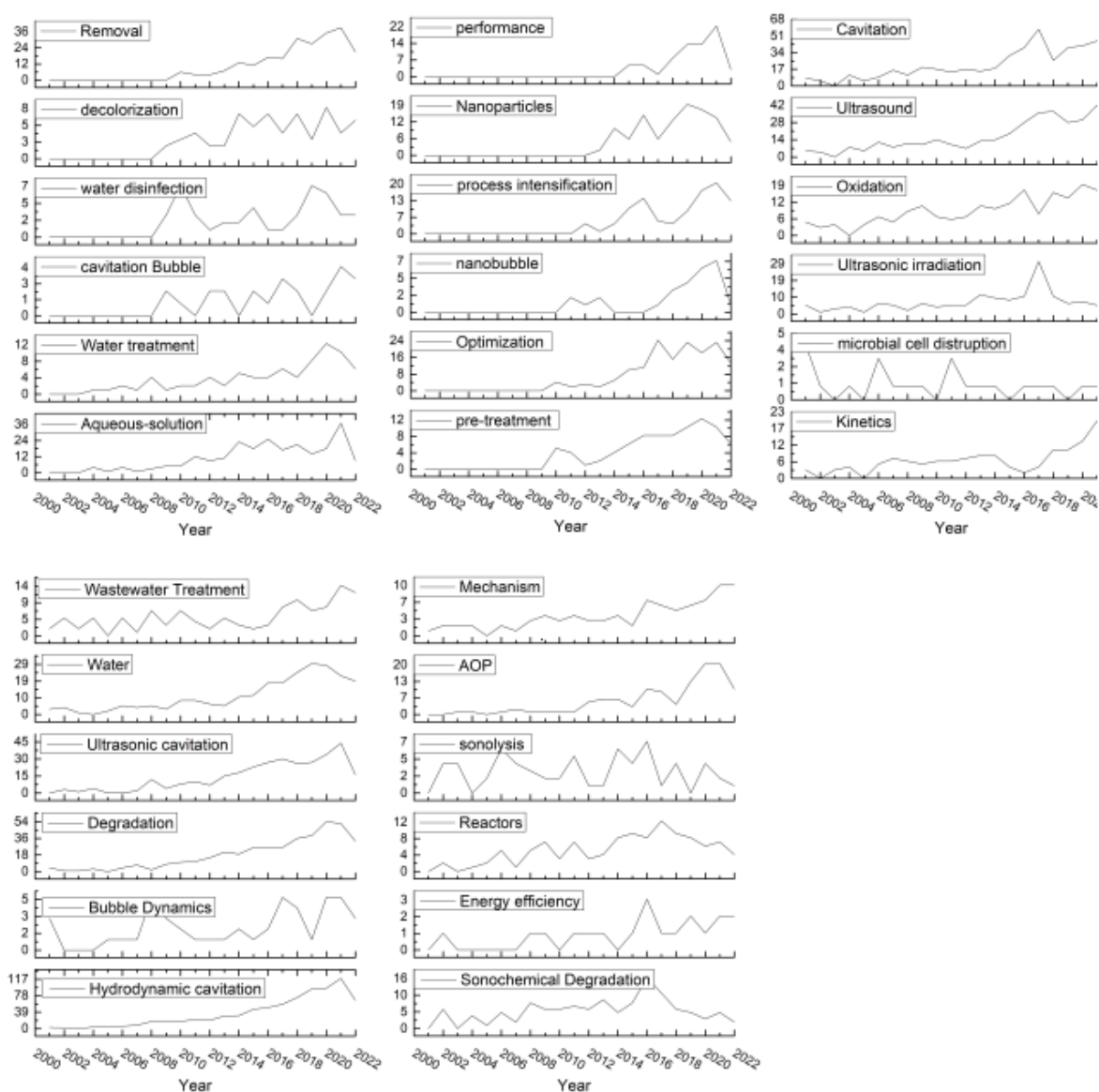


Figure S 3-3: Number of occurrences for the 30 most used keywords in the field of cavitation research related to water and wastewater treatment from 2001 to August 2022.

4. Chapter 4: Water-Related Life Cycle Assessment Applications Over the Past Decade: A Scientometric Analysis

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4.1 Introduction

Water's inherent versatility renders it indispensable in a myriad of environmental applications. These range from harnessing river resources for hydropower generation [277] to repurposing urban water for industrial purposes [278]. However, the escalating anthropogenic strain on water reserves has imposed substantial constraints on water resources, compelling immediate action to promote water reuse [279].

Consequently, there has been an emphasis on pioneering approaches within the realm of water and wastewater treatment [27]. These innovative strategies encompass a diverse array of methodologies, ranging from deploying hollow-fiber membranes to address durability concerns over the long term to utilizing advanced oxidation processes (AOPs) to degrade various contaminants, including pharmaceuticals, employing electro-Fenton, ozonation or ultraviolet photocatalysis, among other methods [161,162,280,281].

Simultaneously, especially in densely populated regions (e.g., Brazil and Malaysia), there is a rising interest in strategies to mitigate the strain on water resources, with an emphasis on rainwater, greywater, and stormwater harvesting [282]. The increasing water demand, coupled with diminishing availability due to economic growth, underscores the exacerbation of global water scarcity problems [153,283,284]. This has prompted the exploration of unconventional water sources, such as desalination, as a means to mitigate the impending water scarcity [285].

However, the fact that is almost neglected is the environmental impacts each treatment method inherently possesses. In fact, wastewater treatment plants (WWTPs) are one of the contributors to greenhouse gas (GHG) emissions [286,287] along with wastewater collection systems (WCSs) [288]. Moreover, Bai et al. (2022) [289] indicated that for a WWTP in Guelph, Canada, the amount of CO₂ emitted from the aeration tank/activated sludge alone mounted to 6743.8 eq.kg/day. For the same WWTP, the capture and combustion of methane produced by biological treatment and anaerobic digestion for energy regeneration can produce a significant amount of CO₂ equivalent, specifically to 12937.9 CO₂ eq.kg/day.

In this regard, it is vital to envisage a holistic view of the potential impacts of various applications of water in the environment; thus, a cradle-to-grave approach is imperative to analyze the consequences (e.g., climate change, acidification, eutrophication, ozone depletion, human health impacts, ecotoxicity) [290–293].

Consequently, the gap between the assessment of environmental impacts and water applications is filled with life cycle assessment (LCA). LCA assesses the potential environmental effects of a product or service by compiling and analyzing all data necessary to quantify the inputs and outputs of the product system that cross the system boundary [294–296].

Understanding and reducing a system's overall environmental impact over its whole life span is the primary objective of LCA, which promotes more environmentally friendly production and consumption patterns [297].

This holistic approach allows for a more accurate understanding of the environmental implications of different approaches and helps identify opportunities for improvement and optimization [298]. In addition to conducting an LCA, employing a Life Cycle Cost Analysis (LCC) approach allows stakeholders to make well-informed decisions while also considering the economic aspects of each option. This approach considers a project's financial aspects, thereby assessing its current and long-term feasibility [299] and elucidates economic impacts that are indispensable to a project. For example, Roebuck et al. (2011) [300] indicated that a residential rainwater harvesting (RWH) system in the United Kingdom and two commercial RWH systems in the United States have shown unsatisfactory returns on investment (ROI), as their payback periods extend beyond the expected lifespan of the RWH systems. Xu et al. (2017) [301] analyzed the capital costs of three different configurations to treat stormwater runoff and redevelop a 7672 m² recreational lot, showing that subsurface gravel wetlands had the highest cost per treated stormwater volume (\$12.64 USD/m³ in 2018), followed by bioretention systems (\$6.75 USD/m³ in 2018), and sales (\$4.64 USD/m³ in 2018). Even though LCA is getting more attention due to the imposition of environmental regulations and sustainable approaches on the companies [302,303], most publications are case studies.

A search on the Web of Science (WoS) database revealed that in the last decade, only 28 reviews were published on LCA and its implementation on any technology or process for the treatment of water and wastewater, as well as to any water use (or reuse) to produce food, goods, energy, or services, hereafter will be referred to as “water-related LCA” works.

Moreover, this analysis also showed the absence of scientometric reviews on water-related LCA. Since it is very time-consuming for scholars to study all the publications related to a specific field in order to comprehend the overall trends and hot topics of published literature [304–306], scientometric analysis and literature classification play a vital role in assessing and integrating published works, enhancing researchers' information, and analyzing the future directions of scientific disciplines.

For these reasons, in this work a scientometric analysis was performed on publications focused on water-related LCA from 2011 to 2023 by using the WoS database.

Chapter 4 reports on the paper “Water-Related Life Cycle Assessment Applications Over the Past Decade: A Scientometric Analysis”, where a scientometric review was carried out to critically assess the recent literature trends on water-related LCA from an environmental science standpoint, in order to offer a more extensive overview of the past and current directions in this subject area and to facilitate future research endeavors.

The analysis examined various aspects such as the number of publications and citations (focusing on the advancements in environmental impact categories), document type, topic analysis, country analysis, country co-authorship network, keyword co-occurrence, and keyword evolution.

In detail, we focused on exploring high-level trends in: i) publications and citations, ii) categories and scientific topics, iii) the evolution and co-occurrence of keywords to indicate the gaps in knowledge, iv) the role of economy and countries' agenda that shapes the water-related LCA studies, v) emerging trends and innovative applications of water, and vi) future directions based on the cumulative effects of the mentioned factors that will move forward the topic of water-related LCA in the future.

4.2 Methodological approach

4.2.1 Data collection, screening, and visualization

The bibliometric part of the current study was done employing the procedure below, reported according to the PRISMA 2000 framework for systematic reviews (Figure 4.1): i) Selecting the most comprehensive database, ii) recognizing the keywords that are relevant to our topics (i.e., keywords related to LCA, water treatment, water applications, water reuse, wastewater, and water management), iii) eliminating the redundant results selecting the specific document types, topics, and the selected publication period to minimize the presence of irrelevant publications, iv) exporting the data from WoS to visualization tools and software for Social Network Analysis (SNA), and RStudio Bibliometrix toolbox; v) interpreting the results [166,307,308].

The initial search was refined using the “advanced search option” and a comprehensive list of keywords incorporating all potential articles. For the first part of the query, title search (TI) was chosen as it was imperative that the title included LCA or its components (i.e. (TI=LCA Or TI=“Life Cycle Assessment” Or TI=“life cycle analysis” Or TI=“hybrid life cycle analysis” Or TI=“hybrid LCA” Or TI=simapro Or TI=“life cycle cost assessment” Or TI=“life cycle cost assessment analysis” Or TI=“life-cycle cost assessment” Or TI=LCCA Or TI=LCC)). The OR operator was used between the keywords to expand the search to all the possible articles that might have included an LCA study. The second part of the search was focused on selecting LCA studies related to water use and was connected to the first query using the AND operator. The title search was chosen also in this case to reduce the risk of irrelevant articles. When one chooses the topic search instead of the title, even the slightest inkling of the keyword in the abstract or keywords plus can cause the article to appear among the search results even if the article is not necessarily related to the main keyword, for this reason the second part of the search was opted for as follows: AND (TI=water Or TI=wastewater Or TI=“water treatment” Or TI=“wastewater treatment” Or TI=“urban water system” Or TI=wastewater treatment system Or TI=urban water cycle Or TI=water depletion Or TI=rainwater Or TI=stormwater Or

TI=surface water Or TI=runoff Or TI= “water treatment plant” Or TI=river Or TI=“water footprint” Or TI=“drinking water” Or TI=groundwater Or TI=“surface water” Or TI=“Potable water” Or TI=“water supply” Or TI= “Water quality” Or TI=“Water management” Or TI=“water pollution” Or TI=“water contamination” Or TI=“water scarcity” Or TI=“Rainwater harvesting” Or TI=greywater Or TI=blackwater Or TI=“Water monitoring” Or TI=“Stormwater management” Or TI=“Water filtration” Or TI=“water desalination” Or TI=“water storage” Or TI=“water chemistry” Or TI=“water engineering” Or TI=“water policy” Or TI=“water analysis” Or TI=“water cycle” Or TI=“water and energy” Or TI=“water cycle” Or TI=“water reuse” Or TI=Water reclamation). As before, all the keywords were connected with the OR operator, and the keywords were not limited only to water treatment aspects but to all the environmental systems that involve water use and water management [309].

The selected publications were narrowed down by focusing on works published between 2011 and 2023. This period was selected since the low number of publications on water-related LCA before 2011 did not allow for a comprehensive analysis on research trends. Furthermore, links between the increased number of publications after 2011 and development of LCA tools and impact categories were investigated and are shown in section 4.3.1. Articles published during 2024 (i.e., 43 publications at the moment of data collection in April 2024), were not considered since it would not be possible to derive meaningful conclusions before the year was concluded [310].

The search criteria were refined to include only “Articles”, “Reviews”, “Proceedings”, and “Early Access” through the “Document Type” filter, resulting in 634 articles. Finally, as recommended by the PRISMA 2000 framework, the articles that were less relevant were removed after retrieving and evaluating each report to avoid incorrect exclusions [311]. In detail, we excluded articles where LCA evaluations were present but not focused on water treatment, management, or use.

Number of records during each step of the selection process is shown in Figure 4.1a, while the methodology and data analysis carried out in this work are shown in Figure 4.1b.

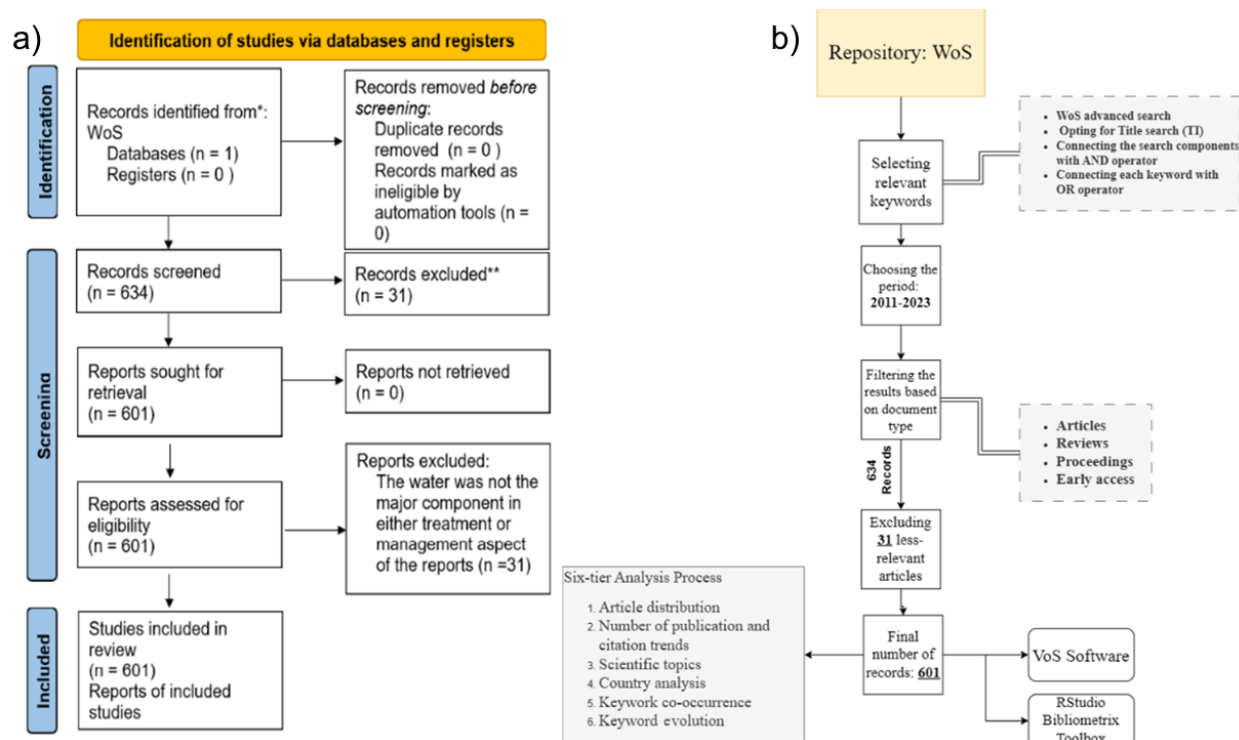


Figure 4.1: Records screened in this work reported following the PRISMA 2000 framework for systematic reviews [311] (a) and Methodological approach framework used in the present work (b).

*: Records identified from each database or register searched.

** : All exclusions were done by human and not automatic tools.

4.2.2 Scientometric analysis

The scientometric analysis comprised a systematic six-tier procedure, encompassing the following stages: i) examination of article distribution to discern the prevalent publication languages and the proportional representation of various article types (e.g., research articles, reviews, etc.), ii) number of publications analysis to elucidate shifts in citation and publication trends, iii) exploration of primary scientific categories (i.e. topics), classifying the central themes of articles, showing the primary study domains related to LCA on water usage (e.g., environmental sciences, environmental engineering, etc.), iv) assessment of each country's contribution to unveil the global distribution of research efforts on the selected topic, v) keyword and cluster analysis to find key terms associated with water-related LCA applications and discern the clustering patterns of obtained keyword and vi) keyword evolution to uncover the patterns of emergence and decline, quantifying the shifts within the specified timeframe [312].

4.2.3 Social network analysis and thematic mapping

To better visualize and investigate the main aspects of the literature published on water-related LCA in the WoS database and characterize the intricate connections within the dataset (e.g., co-authorships network, keyword choice, co-occurrence, and evolution) Social Network Analysis (SNA) was applied

by means of the “Visualization of Similarities” (VoS) software (version 1.6.20) [178,313]. Additionally, Bibliometrix and Biblioshiny (open-source toolboxes in Rstudio, version 2024) were used to conduct thematic mapping of: i) main topics, ii) country citations distribution, iii) and thematic evolution of keywords within selected topics (e.g., “Environmental Sciences”, “Environmental Engineering”) [314].

4.2.3.1 Keyword Dynamics: Co-occurrence and evolution mapping

Keyword co-occurrence analysis was applied in this study to recognize knowledge components, knowledge structure, and research trends based on the keywords present in publications in the selected time period using VoS software [173,174,315,316]. Only authors' keywords were selected, and keywords plus were omitted to eliminate any potential biases from indexing terms and to maintain the specificity of keywords directly related to the core subject, thus obtaining a list of 1623 keywords [317,318].

In order to avoid the presence of low-impact, repeated, or irrelevant keywords in the analysis, several steps were taken: i) finding synonymous keywords and substituting them with the most occurring one (Table S1), and ii) setting the minimum number of keyword occurrences to be included in the analysis to 5 (software default). This resulted in a list of 66 keywords (Table 4-2).

The final list of keywords was processed using the method reported by Tamala et al. (2022) [319] through the application of VoS software, where each node in the visualization represents a keyword, the circle size directly corresponding to the number of occurrences featuring each keyword, and the thickness of the connecting lines is proportional to the number of co-occurrences.

To evaluate how the use of keywords changed during the selected period (keyword evolution), we investigated how many times a keyword was used each year, but keywords occurring only once during the whole period were considered irrelevant and excluded from the analysis.

4.2.3.2 Co-authorship mapping

SNA was applied to investigate the collaborations between various countries based on co-authorship through VoS software [35]. In detail, the number of publications is represented by each circle diameter, while the thickness of the lines connecting the nodes indicates the number of collaborative works. Furthermore, countries were grouped based on how frequently they collaborated with each other [320,321].

4.2.3.3 Thematic maps

Thematic maps were obtained using the *Biblioshiny* package from the *Bibliometrix* toolbox in RStudio. In detail, a thematic map visualizes the relationship and the development of different themes or topics in a certain research topic (i.e., water-related LCA). The topics are categorized on the X-

axis (relevance or centrality), which indicates how well a theme is connected to the overall research field, and the Y-axis (development or density), which reflects the internal cohesion and development level of a theme. Centrality represents the importance of a theme to the broader context, with higher values indicating strong connections to other themes, while density measures the strength of the connections among the keywords used to describe a specific theme, with higher values signifying strong connections, and thus a well-developed and self-sufficient research trend. These axes divide the map into four quadrants: i) motor themes (top-right quadrant) with high centrality and density, representing well-developed, critical topics driving the research field; ii) basic themes (bottom-right quadrant) with high centrality but low density, indicating fundamental but underexplored areas; iii) niche themes (top-left quadrant) with low centrality but high density, representing advanced and specialized but isolated topics; and iv) emerging or declining themes (bottom-left quadrant) with low centrality and density, highlighting areas that are either new and underdeveloped or losing relevance. It should be noted that also when using the Biblioshiny toolbox synonymous keywords were changed with the most occurring one (Table S1).

4.2.3.4 *Sankey diagrams*

Two-field Sankey diagrams are used in bibliometric analysis in order to visualize the relationship between two subsets of research data (e.g., countries and their most chosen journals). The flows between the selected fields elucidate the connections between these data sets, exploring possible interconnections. In detail, this diagram was used to investigate which journals are used by the researchers of the most prolific countries to disseminate their results [322].

4.3 Results and discussion

LCA-based approaches are gaining much attention since they can be applied to evaluate many aspects of processes and activities, such as environmental impact and resource consumption [323,324]. However, while the application of these approaches to investigate topics related to water use and reuse is increasing, there still is a lack of comprehensive studies that analyze the past research trends and elucidate in which direction the use of LCA is progressing and which areas are not receiving enough attention that could be equally important. In this context, the present work conducted a bibliometric and scientometric study concentrating on the last 12 years (2011-2023). Specifically, the trends of citations and number of publications, authors' and countries' networks and contributions, publication and citation trends, countries' citation distribution, thematic mappings of the topics, keywords co-occurrences and interpretation, and keywords' evolution were presented in the following paragraphs.

4.3.1 Citation and publication trend analysis

The number of publications and citations can indicate interest in specific research topics. Moreover, investigating the reasons behind the changes in publication and citation patterns can provide useful insights for researchers to direct future research. In detail, Figure 4.2 illustrates the publication and citation trend regarding water-related LCA studies in the selected period, highlighting that the number of publications has increased significantly from 20 studies in 2011 to 94 publications in 2023, with a gradually increasing trend except for the year 2022, in which a noticeable drop in the number of publications was observed. It should be also noted that less than ten publications each year were present before 2011, preventing the possibility to extrapolate any high-level trends before that year. The significant increase in the number of publications from 2011 could be related to several major developments in the field of LCA, especially considering environmental impact categories.

In LCA analysis, impact categories are metrics used to quantify the impacts of the studied topic, and in the case of water-related work they include, for example, “water scarcity”, “water quality”, “climate change impact”, “eutrophication potential”, “human health impacts”, etc.

In detail, the first major advances occurred between 2003 and 2006, and later in 2010, leading to the publications’ increase shown in 2011 as the evolution of metrics related to the quantification of climate change impacts connected to water-related processes has heavily improved the environmental footprint assessment of these systems (Table 4-1).

One of the first changes was first implemented in 2003 thanks to the development of databases (e.g., Ecoinvent) that provided consistent emission data for various processes, including water management [325]. The next major development occurred in 2006 when the International Panel on Climate Change (IPCC) published a set of guidelines that included a methodology for GHG-related impacts calculation, which were adopted as standardized metrics.

In 2010, the adoption of the Greenhouse Gas Protocol [326] and the development of software such as Sphera (Gabi) and OpenLCA [327,328] lead to the better description of specific processes such as non-conventional water and wastewater treatment technologies (water desalination systems, greywater reuse, rainwater harvesting, etc.) considering, for example, upstream energy use and downstream energy impacts. These advancements were then documented in the International Reference Life Cycle Data System (ILCD) handbook published by JRC European Commission [329]. After 2011, several fundamental developments were also carried out, including the adoptions of new methods and policies. In 2013 ISO 14067 extended carbon footprints to incorporate downstream and upstream emissions in water treatment systems [330]. Upstream emissions refer to GHG generated during the production and transportation of essential inputs for a treatment process (e.g., chemicals,

energy, and materials), while downstream emissions encompass emissions generated after the treatment (e.g., treated water distribution to end-users and wastewater or sludge management).

In 2016, The United Nations Environment Programme (UNEP) and the Society of Environmental Toxicology and Chemistry (SETAC) collaborated to publish the first part of a comprehensive guideline that standardized several indicators related to climate change and water use impacts [331], while the second part was published in 2019 with new standards for indicators on eutrophication, acidification, mineral resources, and soil quality [332]. In this context, recent studies also extended these approaches to consider emissions from imported goods and services, not limiting inventories to country or region where the studied processes are carried out [297,333].

Moreover, in 2017 estimations related to the “water scarcity” impact category were further developed by the introduction of the “Available Water Remaining” (AWARE v1.0) model, developed by the Water Use in Life Cycle Assessment (WULCA) working group under the UNEP-SETAC Life Cycle Initiative. This model quantifies the impacts of water consumption in a specific region over the remaining available water resources [334]. In particular, this methodological development facilitated the implementation of comparative analysis across various geographical locations worldwide.

Regarding the developments of the “water quality” impact category, Pradinaud et al. (2019) [335] developed a framework that integrated water quality into LCA while emphasizing the role of freshwater as a resource, through indicators such as recovery periods, and consumption-pollution effects. This allowed for more comprehensive LCA studies that were able to integrate water quality into the impact assessment process along with water quantities.

Table 4-1: LCA impact category development timeline.

Year	Development	Description
2003	Ecoinvent database	Introduction of a comprehensive database providing consistent emission data for various processes, including water management.
2006	IPCC Guidelines for GHG Emissions	Publication of standardized methodologies for calculating global warming potential metrics.
2010	Integration of Advanced Water Treatment Systems (e.g. desalination) in LCA	Methodologies evolved to incorporate impacts from specific processes such as non-conventional water and wastewater treatment technologies, supported by tools like GaBi and OpenLCA.
2013	ISO 14067 Publication	Extension of carbon footprint methodologies to include upstream and downstream emissions in water treatment systems.
2016	UNEP-SETAC Guidance Document Vol. 1	Standardization of impact assessment indicators, including those related to climate change and water use
2017	AWARE Model Development	Introduction of the Available Water Remaining (AWARE) model to quantify the impacts of water consumption in specific regions.
2019	UNEP-SETAC Guidance Document Vol. 2	Extension of standardized impact assessment indicators to include eutrophication, acidification, mineral resources, and soil quality.
2019	Pradinaud et al. (2019) Framework for Water Quality in LCA	Development of a framework that integrates water quality considerations into LCA, emphasizing the role of freshwater as a resource.

As shown in Figure 4.2a, a sharp increase in the number of publications in 2023 with respect to the previous year can be observed.

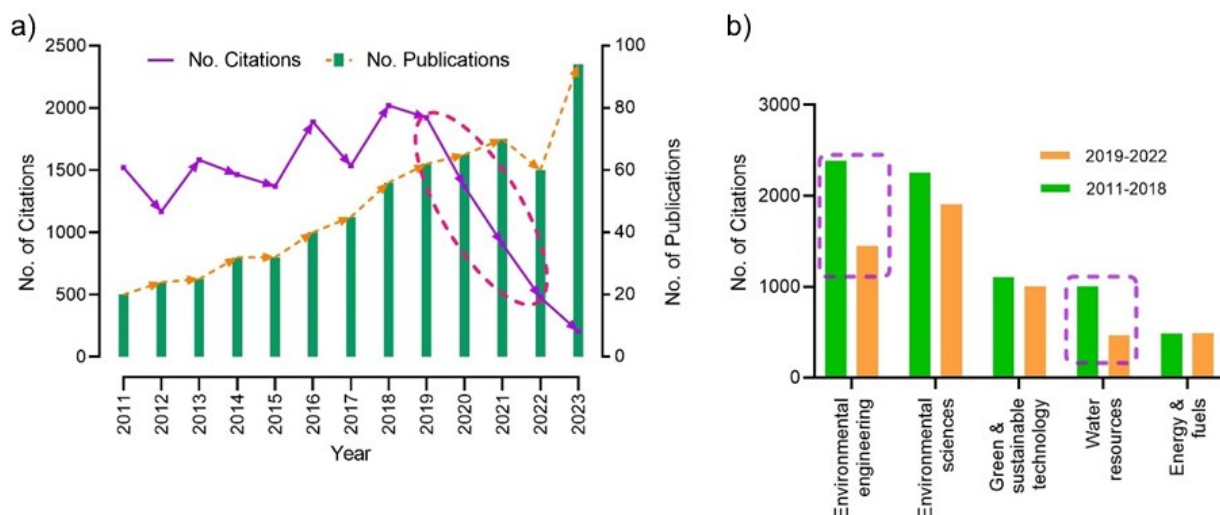


Figure 4.2: Annual number of publications and citations for water-related LCA (a) and detailed category analysis in two periods: 2011-2018 and 2019-2022 based on the number of citations (b). Red circle indicates drop in citation numbers from 2019, while purple rectangles indicate the categories among which citation numbers differed markedly between 2011-2018 and 2019-2022.

In order to better understand this inconsistency in the publication trend, a two-tier analysis was conducted. First, major advancements in the 2022-2023 period regarding water-related LCA methodologies were investigated to understand if they could be linked to the increased number of publications. Then, in the second tier, a “thematic map” analysis was carried out using the

Bibliometrix toolbox from RStudio comparing works from the years 2022 and 2023 to highlight major research topics and how they changed during this period.

The first-tier analysis indicated two major advancements that might be related to the increased number of publications in 2023. The first development concerned life cycle impact assessment (LCIA) methods through a major update of the characterization factors used to quantify damages on freshwater species due to water consumption, which increased the LCIA coverage to over 2000 river basins globally [336]. In detail, the new characterization factors were divided into four sets, distinguishing between i) marginal and non-marginal and ii) regional and global impacts of water consumption on freshwater biodiversity. These new tools were applied by several authors to quantitatively investigate the impact of water use on biodiversity, such as the study by Vasquez-Mejia and co-workers on finfish aquaculture [337] and the work of Zuorro and co-workers, (2023) [338] that evaluated the environmental impacts of producing 1kg of biomass for animal feed grown in inland fisheries effluents as a culture medium using the ReCiPe method [339,340].

The second major advancement leading to the increased number of publications in 2023 could have been the release of the updated version (v2.0) of the AWARE model. The new model offered improved data and extended geographical coverage for water scarcity impacts through updated characterization factors (CFs) representing “relative available water remaining per area” in a watershed.

The second-tier analysis results are reported in Figure 4.3 using a thematic map to illustrate the major topics occurring in 2022 and 2023.

The motor themes (i.e., high centrality, high density) topics shown to drive the research on water related LCA in 2022 and 2023 were quite different. In detail, motor themes in 2022 were “environmental impacts”, “water disinfection”, and “international development”, showing a focus on topics of high international relevance as they are very important issues when addressing water safety both on the regional and global scale. On the contrary, the motor themes in 2023 showed a strong focus on environmental sustainability and technological processes. In fact, numerous publications were on topics such as “anaerobic digestion” and “adsorption” suggesting a growing interest in renewable energy (i.e., biogas production), waste-to-energy approaches (i.e., using organic waste degradation to compensate for the needed energy during the treatment process), and material recovery.

Regarding the basic themes (i.e., high centrality, low density), one group of similar topics (i.e., red circle indicating LCA and wastewater treatment) could be found in both years, indicating a continuous interest in these subjects. However, in 2023 an additional set of basic topics (i.e., green circle) related to “circular economy”, “nutrient recovery”, and “municipal wastewater” was found. In detail, several

of these works focused on resource recovery and waste reduction, highlighting a push towards research more focused on sustainable approaches to the water-energy nexus management.

The results reported in Figure 4.3 showed also the presence of several topics, such as “wastewater reuse”, “energy”, and “advanced oxidation processes”, characterized by the use of several common keywords but that still lack connection to other major topics (i.e., niche themes). This shows the need for additional studies connecting these topics to other research areas, to fully realize the potential of the studied technologies to promote and better contextualize an effective and sustainable use of water resources.

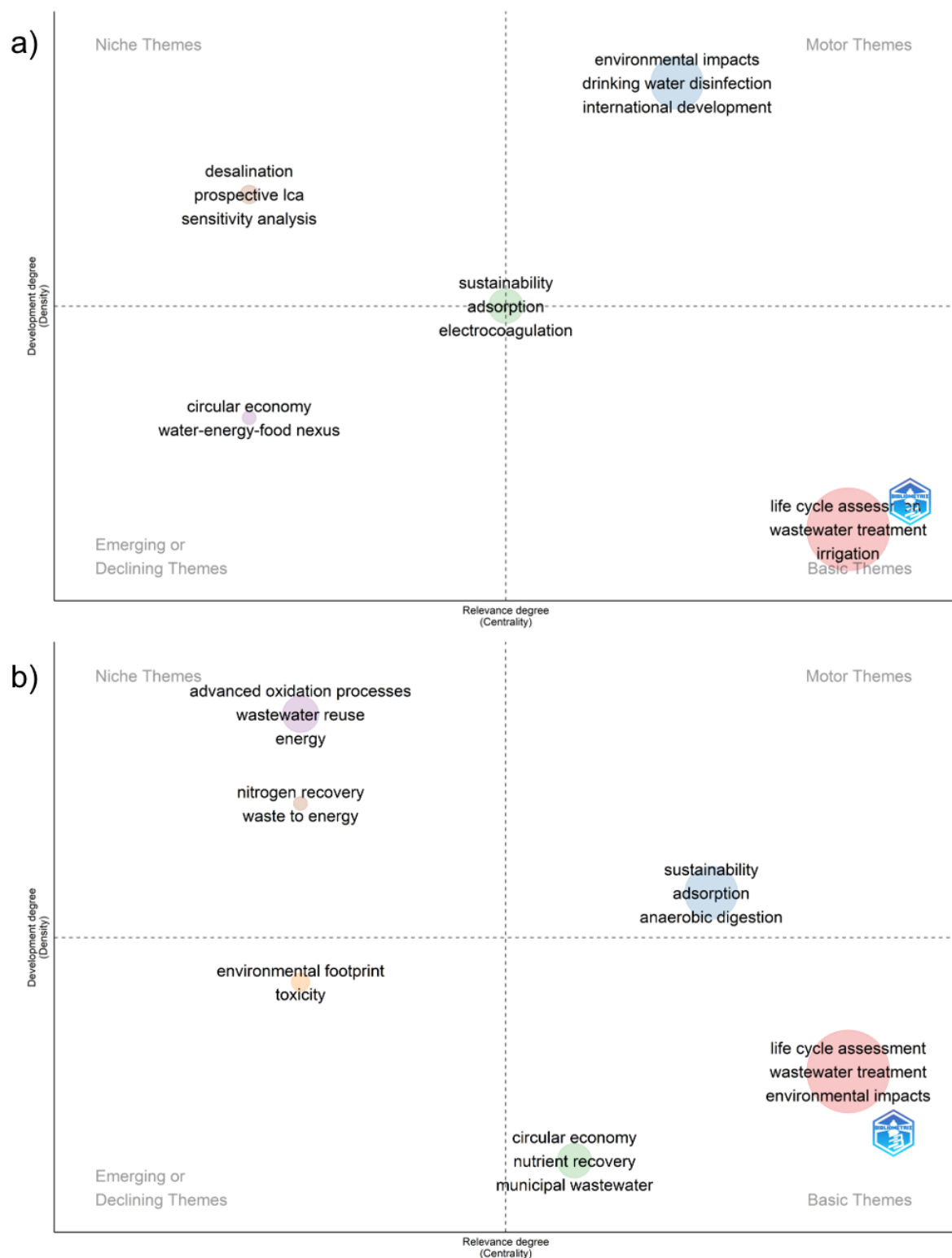


Figure 4.3: Thematic map comparison between 2022 (a) and 2023 (b).

In contrast to the number of publications, the number of citations did not show an overall increase during the whole analyzed period (Figure 4.2a), but only from 2011 to 2018 (+33%). In fact, a downward trend in the number of citations was observed from 2019 onwards, and a comprehensive analysis of available bibliographical and scientometric information was conducted to investigate the

reasons behind this decline by comparing the two periods (i.e., 2011 to 2018 and 2019 to 2022). Articles published in 2023 (for a total of 207 citations) were not considered in this analysis since they may not have had enough time to be properly indexed in the repositories and cited [341].

Publications present in the major repositories (e.g., WoS, Scopus, PubChem) are usually categorized by topic to allow for an easier search among works in the same fields. Therefore, important information can be obtained by investigating how publications are divided among topics. The five major topics from 2019 to 2022 and the period before 2011 to 2018 were compared (i.e., environmental sciences, environmental engineering, green and sustainable science, water resources, and energy and fuels), as shown in Figure 4.2b.

Among the mentioned topics, environmental engineering and water resources for the selected periods have the highest difference in the number of citations. This trend started in 2011 and continued to 2022, which has mostly caused a decline in the total number of citations. Consequently, the nature of the articles for these two topics in the studied periods was investigated to provide insights. In this case, articles for each period were sorted based on "highest citations", and the articles were assessed. It should be noted that "Environmental sciences, and green and sustainable technologies" also have different numbers of citations, but the major decline was caused by the difference in "Environmental engineering" and "Water resources."

Environmental engineering was analyzed first, and it was revealed that during the first period (i.e., 2011-2018), the heavily cited articles had a more general approach toward water applications in the environment. In these articles, the role of water in various environmental activities is studied. For example, the energy production process encompasses the extraction, transportation, and processing of fossil fuels, along with the biofuel production process that requires water for irrigation, cooling, washing, and distillation [342]. Therefore, the application themes were diverse, and other articles were able to cite these articles more easily based on their topic. Furthermore, holistic articles involving several aspects simultaneously, such as articles about water-energy nexus, gained more attention in various fields of study and were cited more pervasively.

Moreover, the articles concerned with food and agriculture that investigate the role of water applications in irrigation, water deprivation, crop production, etc., had a higher chance of getting cited since they had targeted various topics at the same time [343]. Furthermore, the detailed analysis for the period between 2019 and 2022 revealed that the highly cited articles have become more specific and more concerned with the topic of water and wastewater treatment. Therefore, the second most cited article, with 166 citations in 2019, studied the life cycle assessment of wastewater treatment in developing countries [344]. In fact, they have become more detailed, investigating aspects such as life cycle cost analysis (LCC) [345].

The transition from comprehensive subjects to more specific ones has resulted in a notable reduction in citation rates. This decline is attributed to the lower citation likelihood of articles regarding specific processes and/or applications with respect to works dealing with more general frameworks. In fact, over-specialization of water-related LCA studies may further diminish the citation chances due to small differences in the considered topics.

The results also showed that most of the publications analysed in this work were related to the application of LCA to specific processes, while methodological developments were the subject of a more limited amount of work, which were published in a few journals, including the International Journal of Life Cycle Assessment, Sustainability, etc. For example, Verones et al. (2017) [346] proposed a new methodology to address agriculture's impacts on biodiversity, a major water user worldwide, while Pradinaud et al. (2019) [335] introduced a new framework to include water quality in water use LCA.

4.3.2 Distribution of publications among articles, reviews, and conference proceedings

Analyzing the predominant languages and document types within publications related to a specific topic provides valuable insights into the involved scientific community, thus giving information on potential future research trends.

Figure 4.4 shows how publications in the selected period were divided among document types. Research articles have the highest share, with 519 publications (86%), followed by proceedings (54, 9%), reviews (28, 5%), and 7 corrections (not included in the analysis).

Since reviews are one of the most used information sources used by researchers to keep up-to-date with the latest developments in each field more easily, the topics discussed within each of these 28 documents were investigated to identify which areas currently lack comprehensive and updated reviews [347].

In 2023, eight reviews were published, and the majority of the works discussed conventional water and wastewater treatment systems, such as the work by Ribeiro et al. (2024) [348] on the LCA of several Fenton processes, while Martins Vaz et al. (2023) [349] focused on other water reuse technologies, namely rainwater harvesting, and management.

In the period from 2018 to 2022, fourteen reviews were published, mostly in 2018 and 2020, and several topics were discussed. Also, in this case, a relevant portion of these works (i.e., 12 out of 14) focused on water and wastewater treatment, while the other works dealt with agricultural irrigation, [350] and water reuse in urban areas, [351]. It also should be noted that among the reviews on water and wastewater treatment, most authors focused on LCAs conducted on conventional processes, while only X. Li and coworkers, (2018) [352] reported on information regarding more innovative technologies, such as bio-electron-Fenton systems.

These results showed how studies summarizing the most recent advancements in LCA regarding topics other than water treatment are still scarce. For example, there was no work reporting on the significant research that has been presented on i) the use of renewable energy resources, ii) innovative technologies, and iii) water reuse and management.

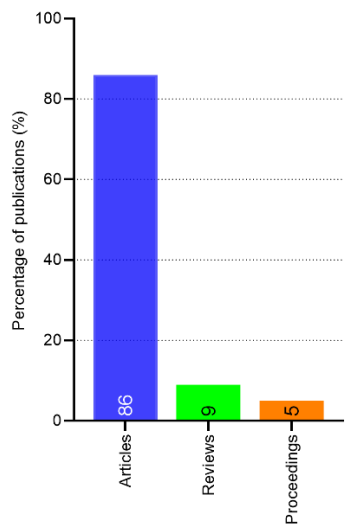


Figure 4.4: Document type distribution during the investigated period considering articles, reviews, and conference proceedings.

4.3.3 Topic analysis

LCA can be applied to many topics. However, several factors (e.g., availability of reliable data) can severely limit its use. For this reason, studying which topics are addressed within the published studies on water-related LCA can give fundamental information on current and future research topics. The results showed that the selected articles could be grouped within 60 (Table S2) topics, but most (i.e., 94 %) of these publications fell into 12 major topics (Figure 4.5). The four most prominent topics were “Environmental Sciences” (33%), “Environmental engineering” (22%), “Green Sustainable technology” (13%), and “Water resources” (9%).

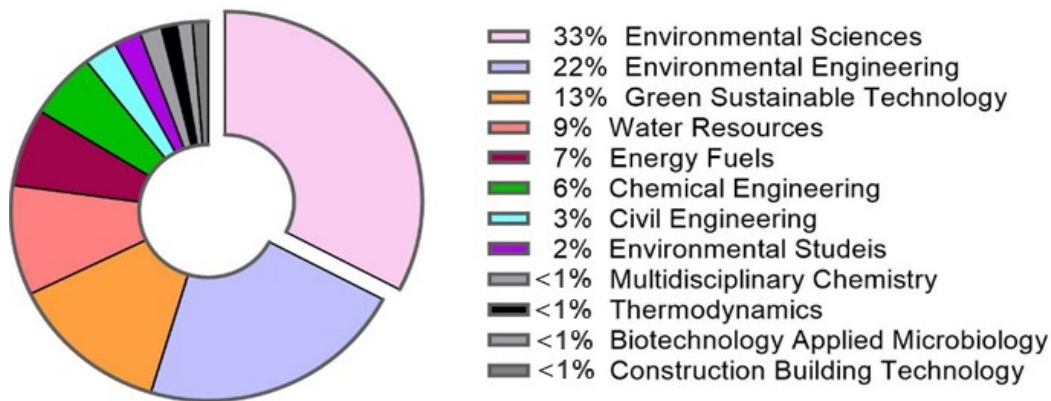


Figure 4.5: Top 12 most populated categories in the present study.

Since “Environmental sciences” and “Environmental engineering” are very broad topics and include most of the selected publications, analyzing the associations between the keywords used within each category and their use can provide valuable insights.

Since keyword co-occurrence analysis showed very similar networks for both topics it was not possible to carry out a meaningful comparison, however the thematic maps representing these two topics over the whole investigated provided more points of comparison (Figure 4.6).

Thematic analysis showed several different motor themes between environmental engineering (i.e., “water treatment”, “desalination”, and “potable water”) and environmental sciences (i.e., “carbon footprint” and “adsorption”), while “water treatment” was common in both. Therefore, a deeper analysis of the publications under the topic of “water treatment” was conducted to highlight if specific research trends were present in each subject category.

In detail, articles included in both categories were excluded from investigation, and the rest showed that articles on water treatment in environmental engineering tended to include LCA analysis on more complex systems and were more concerned with full-scale treatment systems and industrial issues. For example, in the work of Feijoo et al., (2023) [353], LCA was combined with scale-up modeling to assess the environmental impacts and operational performance of electrochemical advanced oxidation processes to remove carbamazepine from wastewater, and it was revealed that the auxiliary pumps were the main contributor to the environmental impacts. Another example was the work of Ahangarnokolaie et al., (2021) [354], in which an LCA study was conducted on both the sequential and simultaneous combination of electrocoagulation (EC) and ozonation (O_3) for textile wastewater treatment, concluding that the sequential combination was the most suitable option since it minimized energy and reagent use while maintaining efficiency.

On the contrary, the results highlighted that articles in the environmental sciences category were generally concerned with more conventional treatment methods, such as “adsorption”, as shown also by the presence of this keyword only in the environmental sciences motor themes.

For example, in the work of Sabando-Fraile et al., (2023) [355], an LCA study was conducted to measure the environmental impacts and resource consumption for the removal of Pb(II) from industrial wastewater using activated carbon from vine shoots. This work showed how response surface methodology can be used to optimize the process (e.g., optimizing the treatment conditions) and lower environmental impacts, thus making the technology more viable for scaling up and industrial use.

Furthermore, thematic analysis results also showed that articles published in the environmental sciences category seem to usually target conventional treatment technologies individually rather than in combination. For example, in the work of Goyal and Mondal (2022) [356] two conventional

removal techniques for arsenic and fluoride from groundwater (i.e., adsorption and electrocoagulation) were compared. On the contrary, works in the environmental engineering category also investigate technologies applied in combination and modified conventional approaches. For example, the work of Garcia-Orozco and co-workers, (2022) [357] reports an LCA study on a combined solar-photovoltaic electrocoagulation system used to treat chocolate factory wastewater while changing the material of the anodes (i.e., aluminum, copper, and zinc). In detail, the effects of process parameters such as pH, current density, and electrolysis time were investigated, revealing that aluminium was the most effective anode for organic removal (50% COD and 39% BOD reduction), but zinc (39% COD and 30% BOD reduction) had the lowest overall environmental impact.

Additionally, in the work by Samaraweera and co-workers, (2023) [358], LCA was conducted on the production phase of an engineered biochar (iron oxide-Douglas fir biochar composite (FDBC) developed to remove bromophenol blue (BPB) dye from water) to quantify the global warming potential (GWP) of this synthesis in terms of ozone depletion, particulate matter and water usage. The results showed that FDBC could be more easily recovered using a magnetic separation process thanks to the presence of iron oxide nanoparticles (Fe_3O_4), leading to a lower GWP than the unmodified version (i.e., 2.38 kg CO_2/g , and 4.69 L/g of water consumption)

It should be noted that some studies in the category of environmental sciences also included the comparison of combined technologies for water and wastewater treatment, however these studies usually are carried out with a lower level of complexity than those published in the category of environmental engineering. For example, in the work of Jachimowski and Nitkiewicz (2023) [359] two water disinfection technologies were compared based on their overall environmental impact through an LCA study. In detail, it was shown that combining UV with electrolytically generated sodium hypochlorite (UV/ NaClO) was more efficient but had a higher environmental impact than traditional use of gaseous chlorine due to the additional use of electricity and sodium chloride

As reported in Figure 4.6, both the environmental sciences and environmental engineering category showed two clusters of topics within the “niche themes” quadrant, however the nature of the topics was markedly different. In the category of environmental engineering, the “niche themes” quadrant included keywords associated with issues closely related to industrial and technical issues, such as “life cycle cost”, “industrial wastewater”, “PFAS”, and “advanced oxidation processes”. In fact, industrial wastewater and its treatment are one of the most pressing issues worldwide and the need to evaluate both the environmental and economic impact of the technologies used in this field is very important [360]. Moreover, LCC is a crucial topic regarding the development of effective and sustainable treatment processes since without a comprehensive cost-benefit evaluation, it would be

very difficult to opt for the most suitable technology even if the environmental impacts and treatment efficiencies are known.

As for the category of environmental sciences, the topics included in the niche quadrant tended to be more general, such as “bio energy”, “greenhouse gas emissions”, and “eutrophication”, “sustainability”, and “water reuse”. These topics showed that that articles published in the environmental sciences category are generally less concerned with the technical and economic aspects of innovative technologies but are more focused on more conventional processes (e.g., adsorption, coagulation, etc.) and their environmental impacts.

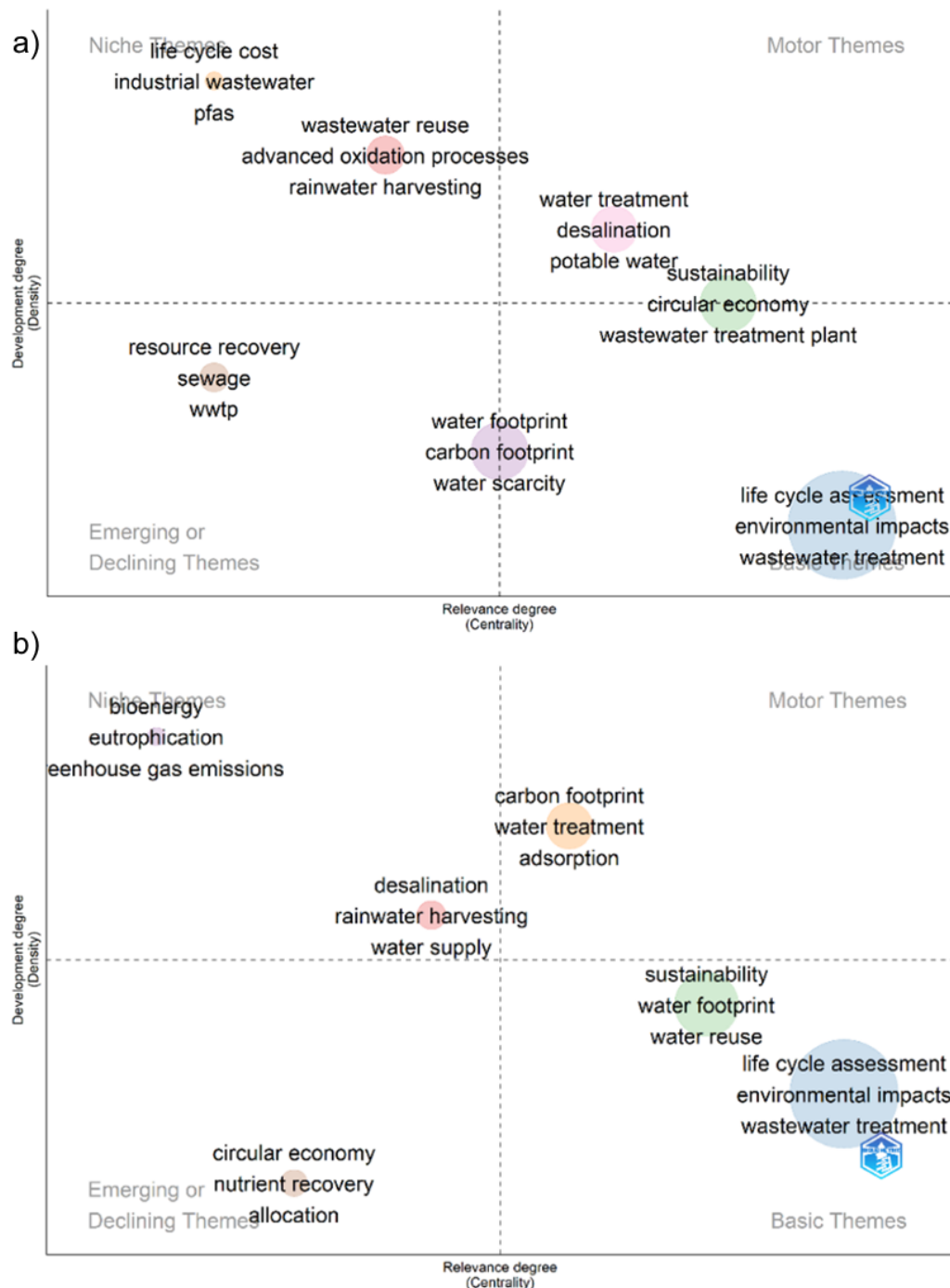


Figure 4.6: Keyword frequency over time for environmental engineering (a) and environmental sciences (b).

The other categories worth investigating were the “Green sustainable technology” and “Water resources”. The articles published in these categories showed some overlaps since these categories are directly related to water reuse. However, the main focus and nature of the articles within these topics differ significantly. Articles within the “Green sustainable technology” topic were more concerned with innovative technologies that enable sustainable water management (i.e., rainwater/stormwater use or greywater reuse). For example, the works of Ghimire et al. (2017) [361],

Leong et al. (2019) [282], and Tavakol-Davani et al. (2018) [362] discussed rainwater and greywater hybrid harvesting systems for both residential and commercial dwellings to understand the economic and environmental impacts of centralized main water systems (MWS) and decentralized rainwater harvesting systems (RWH) or greywater reuse (GWR). It should be noted that within this topic, only the work by Leong et al. (2019) [282] also considered a comprehensive life cycle cost assessment (LCC), and this lack of information may preclude comprehensive comparisons between the overall costs and impacts of the various processes and technologies investigated in the literature.

Works within the “Water resources” topic mainly reported on using LCA to evaluate various water management strategies and technologies to find the most sustainable and suitable options to investigate the environmental impacts of different treatment and supply methods used to address water scarcity issues. For example, Dewalkar and Shastri (2022) [363] proposed a comprehensive fuzzy multi-criteria decision-making framework integrating LCA and LCC for selecting the most suitable wastewater treatment systems in multistore residential buildings, while Uche et al. (2013) [364] investigated the LCA of household water heating systems in two Spanish urban areas, highlighting the significant energy consumption of such processes, and the works by Pradeleix et al. (2020) [365], and Walsh and Mellor (2020) [366], reported on the evaluation of different point-of-use treatment strategies for waste- and groundwater treatment.

It should be noted that a limited focus on economic aspects (i.e., LCC) of any water-related LCA studies can be linked to several issues: i) lack of information on the costs of each technology or management strategy can lead to flawed decision-making since stakeholders will not be able to opt for the best combination of environmental sustainability and economic feasibility, thus leading to problems in the long-term application of the selected options; ii) without clear estimations of their long run return of investment (ROI) innovative and promising technologies (e.g., greywater reuse and rainwater harvesting) might be resisted by decision-makers due to their initial costs; iii) the absence of LCC for any water-related project may lead to neglect cost-saving opportunities (e.g., resource recovery, energy efficiency, and operational costs optimization) since there would be no way to quantitatively estimate the overall cost of each step of the studied process; iv) LCA studies without economic insights may be less appealing to a larger audience, including investors, engineers and policymakers, and this will prevent an effective dissemination of new technologies and processes. In fact, water-related LCA can have the maximum impact only when the discussed technologies or processes can be applied at full-scale, and their implementation would be severely hampered by lack of information on the economic feasibility. Therefore, HC can effectively enhance AOP full-scale applications by improving pollutant degradation efficiency and reducing energy consumption [367].

For example, the work of Gawandes and co-workers (2024) [368] showed that integrating HC with Fenton process significantly increased the degradation rate from 62% to 90%.

On the topic of “Energy fuels”, most of the works focused on quantifying the environmental impacts of different energy sources for water-related applications and technologies, especially regarding greenhouse gas emissions and process efficiency. For example, Sadeghi and Ghandehariun (2023) [369] investigated a solar-powered water-splitting system for hydrogen manufacture, highlighting its efficiency and lower environmental impact compared to other more widely used processes. There have also been more complex approaches involving water applications, such as the work of Juneja et al. (2017) [370], in which they studied the production of renewable diesel from algae grown on wastewater. Their findings suggest significant mitigation in greenhouse gas production compared to conventional diesel.

These studies focus on evaluating the technical aspects of water management options and their environmental viability, showing a significant research trend toward studying energy sources alternative to fossil fuels. Moreover, these three topics are closely connected to several worldwide issues, such as energy and water demand [371–373], covering aspects related both to supply (i.e., “Energy fuels” and “Water Resources”) and use (i.e., “Green sustainable technology”) of water resources.

4.3.4 Country analysis

Analyzing which countries contributed most to the published literature on water-related LCA and the differences between their publication trends is essential to better comprehending how the direction taken by studies related to this topic is influenced by each country's laws, regulations, and economic status [114,374]. Therefore, a comprehensive country analysis was conducted in the present study to understand the potential future research trends on water-related LCA works.

Figure 4.7a shows the top 16 countries based on the number of publications that accounted for about 72% of the total published works, while Figure 4.8: shows the number of publications produced every year by the USA, China, Spain, Italy, France, England, Germany, and Canada.

In detail, the USA, China, Spain, and Italy were the four most prolific countries, accounting for 12%, 9%, 8%, and 6% of total publications, respectively. The rest of the works were almost evenly distributed among France, England, Germany, Canada, India, Sweden, Brazil, Australia, Iran, Switzerland, and Denmark.

Research carried out in the USA from 2017 to 2023 showed a growing interest in evaluating the environmental impacts of various water systems and technologies, particularly focusing on new water management strategies and resource recovery. For example, Taslakyan et al. (2023) [375] carried out an LCA and Techno-Economic Analysis (TEA) on a tertiary wastewater treatment system that showed

the good performance of biochar-integrated reactive filtration (RF) for phosphorus and micropollutants removal, while Santamaria et al. (2021) [376] highlighted both the considerable energy savings and decreased CO₂ emissions obtainable by using dynamic water flow glazing (WFG) systems (i.e., incorporating water flow within building facades for heating and cooling) and the high initial investments needed (i.e., 150-200 Euros per square meter of facade).

These works tended to optimize established processes and use cutting-edge technologies to improve wastewater treatment and develop new water management practices. However, they lack information about these systems' economic viability and widespread application.

In addition, these research trends are expected to continue due to the high attention given by policymakers, show also by the high budget allocation foreseen for the next few years (e.g., \$6 Billion budget allocation on clean drinking water and wastewater infrastructure announced on February 2024 [377]).

During the investigated period, publications by Chinese institutions showed significant changes, highlighting this country's growing environmental concerns and technological advances. In fact, most of the early studies were predominantly centred around conventional and established wastewater treatments [378–380], often highlighting the impacts and efficiency of various technologies. The results also showed that, while the main research interest remained focused on water and wastewater treatment, starting from 2017, many publications were reported on LCA for advanced technologies and their application, such as anaerobic ammonium oxidation for nitrogen removal such as the work of Gao et al. (2024) [381] and microbial electrochemical technologies for nitrogen recovery as described in the work of Zhai et al. (2023) [382], and microbial desalination cell in wastewater treatment and saline water desalination as investigated by Zhang et al. (2018) [383]. This clear focus on water and wastewater reclamation for China can be attributed to their increasing water needs due to growing population and urbanization Chen and coworkers, (2024) [384] while the shift toward more advanced technologies could be related to several factors, including China's increasing commitment to carbon neutrality and sustainable development. For example, the “dual control” approach aiming to limit carbon emissions by 2060 has spurred significant advancements in LCA methodologies and applications to better understand the environmental footprints of water-related technologies [385–387]. Furthermore, in response to the increase severity of the water scarcity issue, China has recently decided to introduce a new tax on drinking water consumption to fund further initiatives to address water shortage [385], therefore new studies focused on the economic aspects of water-related processes can be expected, in order to better guide policy development. The work by Liu et al., (2024) [388] also highlighted the recent investments by China on intelligent-based water management systems, in which they employed an AI-enabled water-saving control system with three

control modes: time division control, flow regulation, and critical point control according to actual flow to manage the leaking and subsequent replacement of the old pipework system in China. Therefore, new research focused on how machine learning- and artificial intelligence-based technologies can reduce the environmental impact of water use.

Figure 4.8: shows a significant decrease in the number of publications for both China and the USA between the years 2020-2021, and this could be attributed to several factors: i) the disruption in research global activities due to the Covid-19 pandemic [389], ii) interference in China-USA collaborations due to internal political initiatives (e.g., “China Initiative” by the USA government) that limited USA-China collaborative scientific works [390], iii) changes of available budget due to economic stagnation (e.g. 9% drop in China's research budget from 2019 to 2020-2021) [391] together could have caused the decline in the number of publications for the first two prolific countries.

Spain's publications show research interest quite similar to China's, remaining focused on water and wastewater treatment using both established and novel treatment technologies. The number of publications from this country remained mostly constant until 2023, when a marked increase was shown. In detail, publication from this country have been markedly focused on investigating many innovative water treatment and management strategies, such as greywater reuse, renewable energy-based technologies (e.g., photovoltaic-based treatment systems), and radical-driven AOPs. In particular, these techniques have been focused on addressing several of the most recent challenges (e.g., emerging contaminants, energy consumption, and resources’ recovery) of water and wastewater treatment systems. For example, Jiménez-Benítez et al. (2023) [392] reported on a LCA carried out for a full-scale anaerobic membrane bioreactor (AnMBR) applied for urban wastewater treatment. In detail, the AnMBR employs anaerobic micro-organisms to break down organic pollutants and produce biogas while a membrane filtration apparatus stops biomass and suspended solids from entering the effluent. The work by Guerra-Rodríguez et al. (2023) [393] reports on a LCA study carried out for a sulfate radical-driven AOP (SR-AOP) system used for water disinfection by means of sulfate radicals produced using a combination of UV-A LED, ozone, and peroxymonosulfate (PMS).

Moreover, Spain was showed a particular focus on the use of innovative water treatment technologies powered by renewable energy. For example, in the work of Nair and co-workers (2023) [394], a LCA analysis of three point-of-use water disinfection technologies (i.e., Solar Water Disinfection (SODIS), boiling, and chlorination) was carried out to reveal that SODIS had the lowest carbon emission while relying only on a renewable source of energy (i.e., sun) and was effective in eliminating the microbial community, making the water safer for use.

The results showed that the main topic research in Italy changed significantly during the investigated period. Similarly to Spain, works published during the investigated period were mainly focused on wastewater treatment [354,395,396], but significant attention was also given to drinking water, such as the work of Barjoveanu et al. (2019) [397] on the environmental performance of a drinking water treatment plant, and solar heating systems, such as the work of Maniakova et al. (2023) [398] that compared conventional and solar driven ozonation for urban wastewater treatment.

Furthermore, between 2015 and 2018 (Figure 4.8:), several works focusing on the interconnections and interdependencies between different sectors or resources (i.e., “nexuses”) were carried out, typically focusing on water, energy, and food systems. These studies aimed to understand how changes or interventions in one sector affect the others, promoting integrated management strategies to achieve sustainability goals [399]. For example, the work of Mancini et al. (2024) [400] investigated an integrated waste-wastewater-energy management strategy that included a thermophilic anaerobic digestion (AD), advanced wastewater treatment, and waste-to-energy system (WtE) to recover energy, biomethane and recyclable material. In detail, the thermal energy from the WtE system was used to satisfy the energy needed for AD and wastewater treatment, thereby minimizing greenhouse gas emission and landfill dependencies.

These types of publications show a trend, highlighting how the evaluation of environmental, social, and economic impact related to water will need more interconnected studies, no longer focusing on single aspects or technologies, in order to provide holistic assessments.

The results (Figure 4.7a) showed that England, France, and Germany published fewer articles on water-related LCA during the investigated period. While it is difficult to identify specific trends due to the few works published each year by these countries, the development of new policy objectives and priorities that concerned countries can offer some useful indications. For example, the several policy measures adopted by Germany (e.g., Federal Climate Protection Act [401] to achieve Greenhouse Gas (GHG) neutrality by 2045 may have shifted the focus of LCA research to applications related to energy production rather than water management, as shown by the Fraunhofer Institute focusing its research on economic assessments and public acceptability studies on renewable energy sources [402]. Another example of this trend can be seen in the case of England, where initiatives such as the “2008 Climate Act” (updated on January 2024) and the “Net Zero Government Initiative” to achieve net-zero GHG emissions by 2050 [403,404] have shifted the research focus towards projects with the most potential for lowering GHG emissions and promoting economic growth. In detail, according to the English Department for Business, Energy, and Industrial Strategy studies, to achieve this goal, an investment of roughly £70 billion per year (i.e., 1-2% of England's GDP) will be needed [405]. This emphasis on applied research and development for decarbonizing

homes, power, and transportation may have led to the less prioritization of water-related LCA studies unless they are directly linked to significant carbon reduction benefits or co-benefits within the broader net-zero strategy [403,406]. Furthermore, since the objectives determined by the recent zero-GHG agendas will require much information on the environmental impact also of other sectors than water treatment and use (e.g., construction, public and private transport, etc.), budget allocations could also shift to other research topics, depending on the needs and goals of different each organization and countries [407].

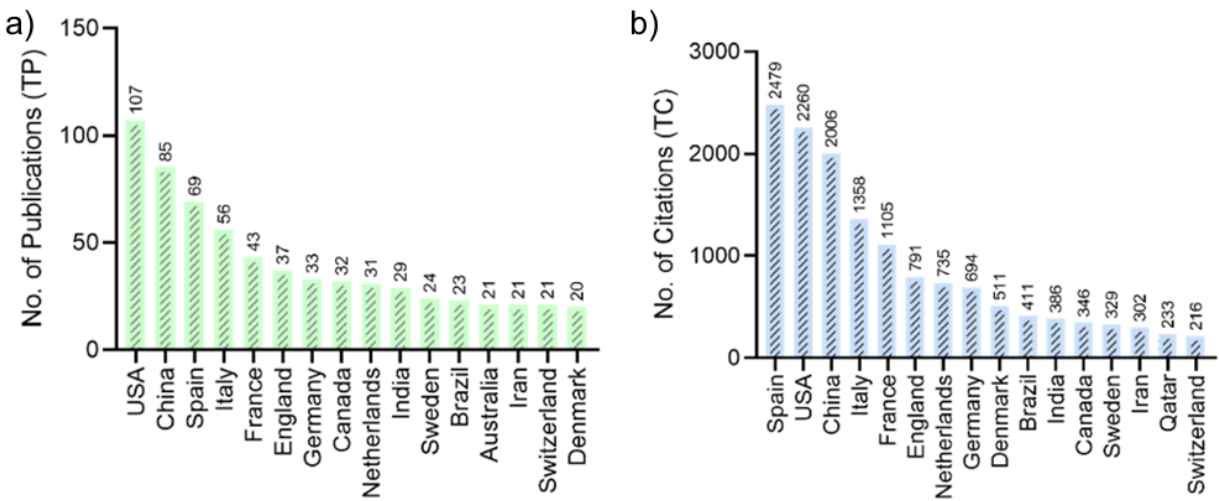


Figure 4.7: Total number of publications for top 16 publishing countries from 2011 to 2023 (a) and total number of citations (b) for top 16 publishing countries from 2011 to 2023 on the topic of LCA on water uses in the environment.

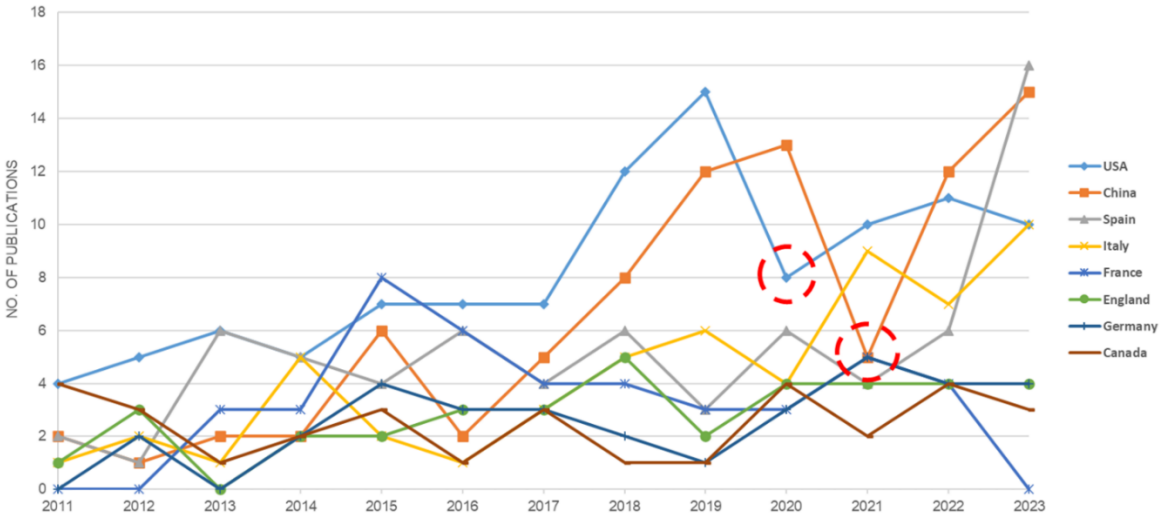


Figure 4.8: Top 8 countries' annual publication trends. The red circles indicate the years when publications from the USA and China markedly declined.

Aside from publication trends for each country, it is also fundamental to understand which countries are investigating topics that have more impact on the subject of water-related LCA. This can be

achieved by investigating the number of total citations for each country. As shown in Figure 4.7b, Spain had the highest total number of citations (i.e., 2497) even though it was in third place according to the number of publications (Figure 4.7a). The reasons that Spain's publications have been more impactful compared to other countries (e.g., Italy 1358 and France 1105) could be attributed to several factors: i) highly collaborative research: as shown in Figure 4.10 (and further discussed in section 4.3.5), Spain showed strong connections to other countries. In particular, collaborative research provides better citation opportunities for publications since it targets the more pressing issues shared among countries and can better address multi-faceted topics such as those related to water treatment, use, and management [408]; ii) publication in high-impact journal: as shown in Figure 4.9, Spain has been the greatest contributor to two of the most used journals in the field of water related LCA (i.e., “Journal of Cleaner production” and “Science of the Total Environment”, with 19 and 10 publications in the studied period (out of 85 and 39, respectively). Furthermore, these two journals are also highly multidisciplinary, making publications available to a wider range of researchers and thereby increasing their impact; iii) relevance of studied issues at the global scale: as previously reported, Spanish researchers focus a lot of attention on topics related to water scarcity such as water desalination (e.g., Navarro (2018) [409]) and drought management (e.g., Morote et al., (2019) [410]), that are of high interest also in other parts of the world affected by this issue.

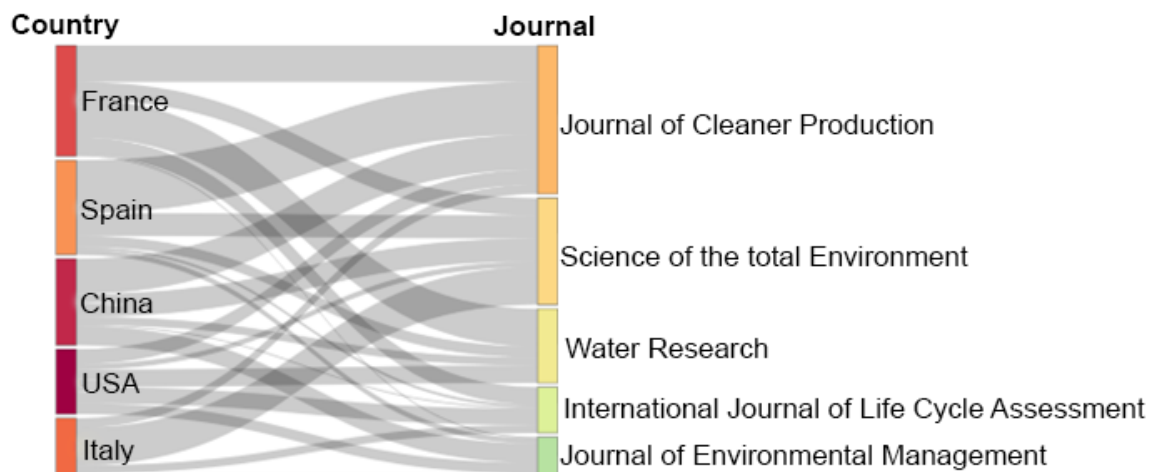


Figure 4.9: Two-field Sankey diagram showing the contributions of the five most prolific countries with regards to works published in the five most prominent journals regarding water-related LCA.

4.3.5 Co-authorship network

In addition to investigating the publication trends of each country, fundamental information on international research trends can also be obtained by studying the co-authorship network since it can elucidate the presence of global research trends and how well specific knowledge is disseminated [321]. Accordingly, a co-authorship SNA was carried out among the top 16 publishing countries, as

shown in Figure 4.10. The line thickness in the diagram represents the degree of collaboration (i.e., the number of shared works), and the diameter of the node shows the overall number of publications. Based on the analysis, the USA and China showed the strongest collaboration with a link strength of 13 (Figure 4.10). However, while these two countries were the most prolific in the selected period, the Netherlands showed the highest overall number of collaborations, with a total link strength of 48. This high degree of collaboration can be linked to policy developments by the Dutch Government, such as the Netherlands International Water Ambition (NIWA), comprised of four principal ministries of the Netherlands National Government, that aimed to increase water security and water safety by incentivizing more partnerships between public and private international organizations [411]. In particular, works published by Dutch institutions generally focus on conventional wastewater, water, and drinking water applications, such as the work of Baustert et al. (2022) [412] on water desalination in the steel industry and the work of Harris et al. (2021) [413] on wastewater treatment design. After Netherlands, Spain, and the USA have the highest number of collaborations, with total link strengths of 45 and 44, respectively.

Results of co-authorship analysis also confirmed how most countries in the blue and green clusters (i.e., China, the USA from the blue cluster, and Spain from the green cluster) tended to work on similar multidisciplinary topics, such as addressing the increasing water-energy problems, as also discussed in section 4.3.4. Furthermore, Figure 4.10 shows also how Italy, France, Germany Netherlands (i.e., red cluster) generally focused more on regional or country-wise general water issues rather than incorporating advanced technologies and water reuse.

We believe that the current international interest in the development and use of more efficient and sustainable water management approaches [414,415] will encourage the development of new international collaborations, especially regarding countries that are still less prominent on the international stage.

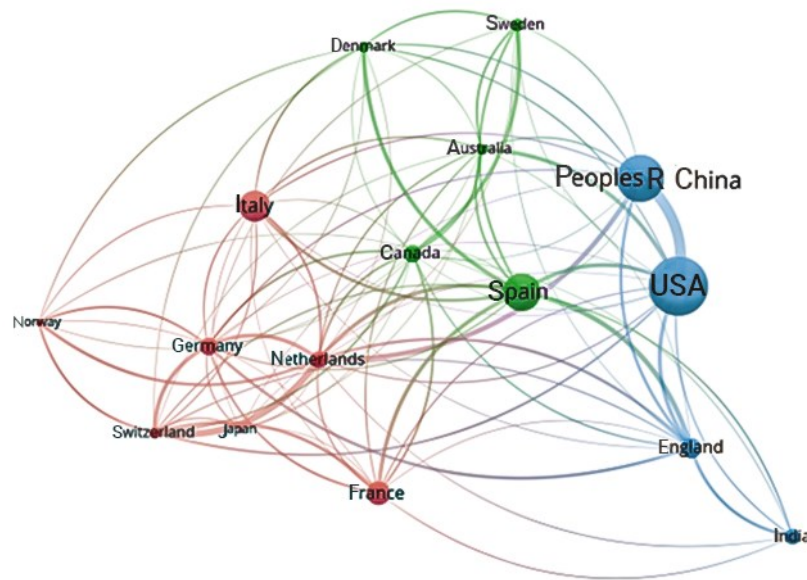


Figure 4.10: Co-authorship network of the 16 most publishing countries. Different colors indicate the results of cluster analysis based on how frequently they collaborated with each other.

4.3.6 Keyword co-occurrence analysis

To fully understand the focus of current research, recognize knowledge gaps, and predict future trends, it is fundamental to investigate how research effort has been divided among the most prominent topics. Understanding the broader implications and trends of water-related LCA application is a challenging task, but keywords can act as the guidelines for better understanding the various aspects of the published literature since they are commonly used for indexing purposes in all scientific databases (e.g., Scopus, and WoS) [416,417]. In fact, keyword co-occurrence analysis can reveal critical gaps between the interaction among different research topics, as well as show how they interact and evolve highlighting dominant topics and themes in water-related LCA.

Keyword co-occurrence analysis was conducted following the approach reported in paragraph 4.2.3.1, and a list of 66 keywords occurring at least 5 times was selected (Table 4-2) and grouped through cluster analysis by applying the VoS software (Figure 4.11).

Table 4-2: Keywords (ordered by occurrence) for all clusters

Cluster (color, topic)	Keywords (occurrences)
1 Red, (13 keywords on “Sustainable Water Management and Technology”)	Wastewater reuse (12); Rainwater harvesting (11); Hydrogen production (8); Reverse osmosis (7); Greywater (6); Life cycle cost (6); Solar energy (6); Advanced oxidation processes (5); Industrial wastewater (5); Multi-objective optimization (5); Super critical water (5); Uncertainty analysis (5); Urban water cycle (5)
2 Green, (12 keywords on “Environmental Impact and Sustainability of Water Management Systems”)	Carbon footprint (15); Water treatment (12); Wastewater treatment plant (11); Global warming potential (10); Life cycle inventory (8); Water use (7); Cumulative energy demand (7); Bottled water (5); Sensitivity analysis (5); Urban water system (5); Water quality (5); Water resources (5)
3 Dark Blue, (11 keywords on “Sustainable Wastewater Treatment and Resource Recovery in the context of Circular Economy”)	Wastewater treatment (62); Sustainability (42); Circular economy (14); Nutrient recovery (9); Eutrophication (8); Hybrid life cycle assessment (7); Techno-economic analysis (7); Bio energy (6); Greenhouse gas emission (6); Bio refinery (5); Recycling (5)
4 Dark Yellow, (9 keywords on “Water-related Nexus”)	Energy (14); Desalination (10); Anaerobic digestion (9); Sustainable development (8); Water supply (8); Water (7); Energy consumption (6); Nexus (6); Renewable energy (5)
5 Purple, (9 keywords on “Environmental and economic Assessment of Water Management Systems”)	Life cycle assessment (415); Wastewater (23); Drinking water (15); Constructed wetland (12); Adsorption (9); Biogas (9); Toxicity (7); Environmental footprint (6); Cost-benefit analysis (5)
6 Light blue, (6 keywords on “Wastewater Treatment and Resource Recovery”)	Environmental impacts (97); Micro-algae (9); Climate change (7); Sewage (6); WWTP (6); Resource recovery (5)
7 Orange, (6 keywords on “Sustainable Water Management in the Context of Water Scarcity”)	Water footprint (25); Water scarcity (14); Water reuse (12); Water consumption (11); Industrial ecology (5); Irrigation (5)

From the results reported in Figure 4.11, SNA found that the selected keywords could be grouped into 7 clusters, each connected to a specific topic: i) cluster 1 (red, on “Sustainable Water Management and Technology”); ii) cluster 2 Green, (12 keywords on “Environmental Impact and Sustainability of Water Management Systems”); iii) cluster 3 Dark Blue, (11 keywords on “Sustainable Wastewater Treatment and Resource Recovery in the context of Circular Economy”); iv) cluster 4 Dark Yellow, (9 keywords on “Water-related Nexus”); v) cluster 5 Purple, (9 keywords on “Environmental and economic Assessment of Water Management Systems”); vi) cluster 6 light blue, (6 keywords on “Wastewater Treatment and Resource Recovery”); vii) cluster 7 Orange, (6 keywords on “Sustainable Water Management in the Context of Water Scarcity”).

In detail, cluster 1 (red) contained the highest number of keywords (i.e., 13), and the one showing the highest number of connections was “Wastewater reuse” (i.e., link strength of 28), and it was linked to four other clusters (i.e., Cluster 5 (purple), Cluster 6 (light blue), Cluster 7 (orange), and Cluster 4

(dark yellow)) as shown in Figure 4.11. These results show how studies related to “Wastewater reuse” were mainly connected to “Water scarcity” and “Water footprint” (Cluster 7, orange), “Sustainability” (Cluster 3, dark blue), and “Environmental impacts” (Cluster 6, light blue), while no co-occurrences with keywords belonging to Cluster 2 (green, “Environmental Impact and Sustainability of Water Management Systems”) were shown. This shows that so far, very few studies have been conducted on applying LCA for wastewater reuse considering also both the energy demand and carbon footprint of such systems. Another field of study that has been scarcely investigated was also LCA related to wastewater reuse in urban water systems. These topics will call for further studies considering urban areas' current and projected population increase. However, the connections between “Wastewater reuse“, “Desalination”, and “Water scarcity” (Cluster 4, dark yellow) showed how researchers have started to investigate the water footprint of wastewater reuse systems in the context of desalination processes. For example, the recent article by Glover et al. (2022) [418] reported on the LCA of a wastewater treatment process for indirect wastewater reuse employing a novel membrane bio-reactor membrane distillation (MBR-MD), while Tarpani et al. (2021) [419] compared seawater desalination by reverse osmosis (RO) and indirect potable wastewater reuse.

Cluster 4 (dark yellow) contains keywords that can be closely associated with the concept of “Water-related Nexus“, the connections with the other clusters can give important information on how this concept has been integrated within the field of water-related LCA. This concept is of fundamental interest since it can provide a holistic view of interconnections and interdependencies between different systems and resources related to water management [420,421]. In particular, the keyword “Nexus” was most frequently connected to keywords related to energy use and wastewater (i.e., cluster 4 and cluster 5). However, only a few connections were found with keywords related to “Environmental and economic Assessment of Water Management Systems” (i.e., cluster 5, purple) despite the increasing interconnection of water supply, use, and treatment systems with other sectors (e.g., food production, drinking water, and energy production), thus highlighting the need for additional research.

In cluster 3 (dark blue), the most occurring keyword was “Wastewater treatment”, and it was strongly connected to “Sustainability” (i.e., link strength of 4), confirming the presence of studies on sustainable wastewater management and treatment already shown in the topic analysis (see section 4.3.3). Cluster 3 was also well connected to other clusters, showing the interdisciplinary nature of this topic. For example, “Wastewater treatment” was connected to “Cost-benefit analysis” (cluster 5, purple), showing interest in investigating both the total costs and benefits of a project for better decision-making, as highlighted by the work of López-Serrano et al. (2023) [422] in which cost-

benefit analysis was conducted on a decentralized wastewater treatment plant to support the development of new sustainable water management strategies.

While the results shown for cluster 3 highlight a wide application of “Cost-benefit analysis” in the field of water-related LCA, the applications of LCC (“Life cycle cost” in cluster 1 (red)) seem to be still limited to a relatively small number of topics such as “Rainwater harvesting“, “Greywater”, and “Water reuse”. This could be attributed to this approach's high information and data requirements, especially in the case of full-scale applications. Nonetheless, these studies are fundamental for stakeholders and decision-makers since they allow more effective management of monetary and time resources. Moreover, as an increasing amount of information related to water management technologies and processes is becoming available [423], further studies focusing on LCC are expected to be carried out.

Cluster 7 (orange) contained keywords related to “Sustainable Water Management in the Context of Water Scarcity”), showing several connections with cluster 1 (red). In detail, “Water scarcity” and “Water reuse” were connected to “Wastewater reuse” and “Rainwater harvesting“, indicating that several studies aim to address water scarcity problems through the reuse of rain- and wastewater. On the contrary, the lack of connection between cluster 7 (orange) and “Greywater” and “Urban water” in cluster 1 (red) showed a comparative lack of research on investigating the use of these resources to address water scarcity, in particular within urban environments.

It is also important to note how keywords related to economic aspects of water treatment, use, and management, such as LCC (i.e., cluster 1, red), cost-benefit analysis (i.e., cluster 5, purple), and techno-economic analysis (i.e., cluster 4, dark blue) are all isolated nodes with limited connections to other clusters, indicating that studying the economic impacts of water-related processes and technologies have not yet become an integral part of LCA analysis in this field. This lack of connections is especially apparent with regards to keywords related to more innovative technologies (e.g., AOP, unconventional membrane technologies, innovative desalination techniques, anaerobic digestion using micro-algae, etc.), indicating the need for further collaborative research in order to bring these technologies towards marketable applications.

Furthermore, while the central position of keywords such as “sustainability”, “wastewater treatment”, and “environmental impacts” shows a specific commitment to solve immediate environmental problems, the marginal position of keywords such as “greywater”, “rainwater harvesting”, “solar energy”, and “circular economy” indicate a lag in integrating more innovative water management strategies and renewable energy into the current practices. This will, in turn, make it more challenging to address established sustainability goals, like the United Nation’s Sustainability Development Goals (SDGs).

Keyword co-occurrence analysis was also used to gain more detailed information on the development and direction of LCA studies related to water and wastewater treatment processes and technologies, especially regarding conventional and more innovative solutions. In detail, the main keywords were: “reverse osmosis” (7 occurrences), “advanced oxidation processes” (AOP) (5), “super-critical water” (5), “nutrient recovery” (9), “recycling” (5), “desalination” (10), “anaerobic digestion” (9), “constructed wetlands” (12), “adsorption” (9), “micro-algae” (9), and “resource recovery” (5).

Keywords describing conventional technologies showed a higher number of connections to multiple clusters than those related to innovative technologies, showing their wider application. In detail, the higher number of links was found for desalination, anaerobic digestion, and reverse osmosis, (25, 15, and 12 links to other clusters, respectively). On the contrary, keywords related to innovative technologies mostly lacked connections to other clusters (e.g., nexus=6, membrane bioreactor=6, economic analysis=7, etc.) showing that several aspects of these processes are still unexplored, thus limiting their full-scale application due to lack of information on cost, efficiency, and scalability. It should be noted that, while the number of connections across clusters for keywords related to innovative technologies was generally low, some innovative keywords indicating these new technologies showed high link strengths to other clusters (e.g., rainwater harvesting=21, wastewater reuse= 27, etc.), thus highlighting that some innovative technologies are receiving more attention. While connections between keywords related to technologies and economic aspects were mostly limited, some research on the economic shortcomings of the most used processes was shown. For example, several works highlighted the high energy demands of RO systems. For example, Garfi et al., (2016) [424] reported that a domestic RO system generated 10-24% more environmental impact compared with the generated impact of other tap water alternatives, due to high electricity consumption. Similarly Goga et al., (2019) [425] identified electricity consumption as responsible for most of the environmental impact of seawater desalination and reclamation of mine-affected water (4.17 and 2.44 kg CO₂ eq for each kL of generated potable water, respectively) in South Africa, showing also how the use of solar and wind power could bring significant environmental benefits. The reason that some processes have fewer publications than others (i.e., AOP and membrane filtrations) could be related to the following reasons: i) lack of data on cost and scalability, ii) limited integration of LCC in the conducted LCA analysis, and iii) insufficient full-scale application studies. Therefore, the pilot- or full-scale study of these applications fundamental in increasing the number of LCA publications on these topics since LCA publications need reliable data.

The results highlighted also that most works linked to innovative technologies were related more to their environmental impacts than their economic feasibility. For example, Zhang et al., (2018) [426] carried out a LCA comparing the impacts of using microbial desalination cells (MDCs) incorporating

anion exchange membranes (AEM) and cation exchange membranes (CEM) to simultaneously treat wastewater and desalinate seawater vs the use of a conventional WWTP. The study showed that manufacturing and operation of MCDs were the major contributors to GWP (ca. 23% and 59%, respectively). This was due to the specific material used for the ion-exchange membranes and electricity use, showing how improving materials and energy could make these systems a viable substitute for other less efficient conventional water treatment systems

Regarding algorithm-based and smart water treatment strategies, it should be noted that while much focus has been given to their development, as shown for example by the works of Baarimah et al., (2024) and Ganthavee and Trzcinski (2024) [427,428], only very limited research has been carried out on their environmental impacts. For example, Bonoli et al., (2019) [429] calculated the environmental impact of several small urban greywater reuse systems that employ smart monitoring technology. This shows that there is still a notable gap between research on AI-based technologies optimization for water and wastewater treatment systems and their environmental impact analysis.

4.3.7 Keyword evolution analysis

In order to better understand how research on water-related LCA will evolve, fundamental information can be obtained by investigating how the major keywords have changed over time (e.g., when a keyword appeared if it was constantly used). For this reason, a comprehensive keyword evolution analysis was conducted, as shown in Figure 4.12.

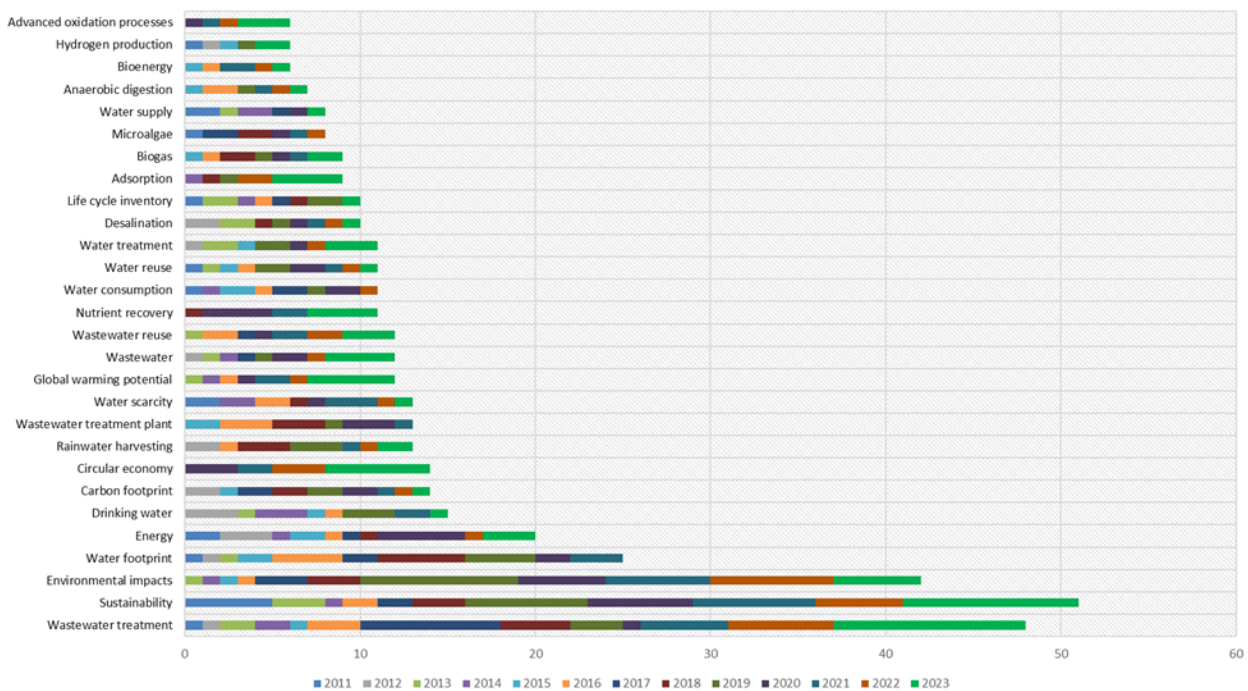


Figure 4.12: Keyword evolution for major keywords based on number occurrences. The keyword "life cycle assessment" was omitted from the figure to better visualize other keywords.

“Sustainability” was the most used keyword, and its use showed a significant increase in 2019 – 2020 when several policies related to addressing climate change (e.g., EU Green Deal and United Nations Climate Action Plan) [430,431] started to be implemented. Moreover, the keyword “Circular economy” also showed a similar trend, as it started to be largely used in 2020. This highlights the importance for researchers to stay up to date with the most recent policy proposals, as they strongly influence the national and international budgets allocated for research.

“Wastewater treatment” was the second most used keyword and it has been used during the whole investigated period, showing a significant increase in 2017 when China's Water Pollution and Control Law [432] came into effect. Similarly, other organizations are now focusing on this topic as water demand is steadily increasing. Therefore, more studies will be needed to investigate the performance and costs of different water management and treatment solutions.

“Bioenergy”, “Biogas”, and “Anaerobic digestion” emerged as keywords in 2015, reflecting the need for alternative energy sources other than fossil fuels in response to the increasing energy demand, and this could be ascribed to the United Nations’ “Sustainable Development Goals” adopted in 2015 [433]. In particular, goals 7 (“Clean and affordable energy”) and 13 (“Climate action”) directly call for energy-efficient solutions and climate resilience; therefore, technologies such as “Anaerobic digestion” that could convert waste into valuable energy resources such as “Bioenergy” in the form of “Biogas” aligned with this agenda. This was shown, for example, in the work of Vassalle et al. (2023) [434], in which the environmental impacts of an up-flow anaerobic sludge blanket (UASB) reactor coupled with a high-rate algal pond (HRAP) for wastewater treatment and bioenergy recovery were assessed.

The results (Figure 4.12) also showed that from 2017 to 2020, several keywords related to specific processes or treatments (e.g., AOPs, nutrient recovery, unconventional membrane technologies, innovative desalination techniques, anaerobic digestion using micro-algae, etc.) started to be used, indicating a growing interest in LCA studies addressed to practical applications and processes rather than generic frameworks. This shift reflects the urgent need to develop full-scale technologies and processes to address global challenges such as energy, water, and resource demand. However, the limited connections of these keywords with each other and other major keywords in other clusters, especially the economic themes, highlights the lack of information regarding the socio-economic impacts of these processes, leading to marked difficulties into successfully globally adopting these advanced solutions. Given this context, further research on pilot- and full-scale application of these technologies will be fundamental to obtaining the needed information to estimate, in addition to their environmental impacts, also their socio-economic viability.

4.4 Future direction and perspective of water-related LCA

The field of water-related LCA has shown a remarkable evolution in the last 12 years, as shown by the emergence of new frameworks, tools, and research topics. In particular, the analysis carried out in this work highlighted several trends with regards to research agendas and focuses. However, several knowledge gaps and research opportunities could also be observed:

- Integration of smart technologies and analytical tools into LCA frameworks and models:

While marked improvements have been shown for LCA frameworks, databases (e.g., Ecoinvent, Circularity Package, LCA Commons) and software (e.g., SimaPro, OpenLCA, and Gabi) in the past few years, the capability of machine learning- and AI-based technologies to handle, analyze, and correlate large volumes of data can offer the possibility of exploring interconnections between the various factors related to the environmental, social, and economic impacts of water-related technologies. Furthermore, further research should also focus on the integration of LCA with machine learning- and AI-based technology to improve optimization processes for water treatment, use, and reuse, thus leading to the development of water management strategies characterized by both high performances and low environment impact.

- Less-explored innovative technologies:

While many LCA studies have been focused on conventional water and wastewater treatment systems, the environmental, economic, and social impacts of more innovative water management options are still mostly underexplored. In detail, this could be due to both a lack of interest and a lack of the necessary information, therefore additional research focused on addressing this lack of information is urgently needed. Furthermore, present LCA frameworks in some cases have been shown to be inadequate to fully characterize the environmental impacts of some innovative water treatment and management technologies, so further effort should be made to adapt current LCA tools to better account for their specific characteristics.

This information will be vital to encourage the full-scale adoption of these emerging technologies, which will help to address global issues like increasing energy demand, water deficiency, and uncontrolled GHGs emissions.

- Social impact assessment:

Water-related LCA has been extensively applied to develop more sustainable water treatment and management systems, promoting more innovative approaches and technologies. However, the social impacts of the investigated topics have been usually neglected, even though they can be of major importance when evaluating the impact of

the studied systems on both economy and society. To address this gap, further focus and research both on improving socio-economic indicators and better characterized water-related processes are needed. In fact, the evaluation of long-term social benefits (e.g., job creation) can offer important information to stakeholders and decisionmakers when evaluating the adoption of new water treatment systems, especially those that may still be characterized by high initial investments.

- Life cycle cost integration:

While much effort has been made to investigate the environmental impacts of water-related processes and technologies, the economic implications of these applications remain much less studied. In particular, the use of LCC offers a viable tool to bridge the gap between environmental impact assessment and economic considerations, leading to the identification of more sustainable and pragmatic solutions.

In detail, further research aimed to obtain a holistic evaluation of water management options considering both environmental impacts (e.g., GWP, resource use, and energy use) and economic aspects (e.g., capital investment, operating costs, and ROI) will offer decisionmakers and stakeholders a full picture of the trade-offs between ecological and economic factors.

Moreover, further standardization of frameworks for integrating LCA and LCC is essential to promote reproducibility and reliability of studies, especially considering the harmonization of datasets on environmental and economic information.

- Temporal dynamics in water-related LCA:

Although extensive efforts have been made to estimate the environmental and economic impacts of water-related processes, current Life Cycle Assessment (LCA) studies usually do not properly account for temporal variations of several factors, such as resource availability, energy demand, and environmental stressors. However, these temporal dynamics are essential, as they can greatly change the effectiveness and impacts of the studied systems. In fact, LCA studies are usually carried out using integrated data obtained over long periods of time, not properly representing more extreme conditions (e.g., extremely dry/wet seasons, drought periods), although they are predicted to be more frequent in the future [435]. As an example, water scarcity in hot regions can be further exacerbated during especially dry seasons and lead to greater environmental impacts of water-consuming procedures such as irrigation or industrial cooling.

For these reasons, further LCA studies accounting also for these temporal variations are needed as they will assist policymakers to formulate more effective and adaptive water management approaches.

4.5 Conclusions

The work carried out in this study provided a comprehensive scientometric analysis of water-related LCA studies over the last decade, investigating the evolution of this research field, illustrating the most recent advances, and highlighting the most significant knowledge gaps.

The results showed how the developments in this field (including, for example, the increased attention to environmental impact assessment for water-related technologies) can be linked to international agendas (e.g., zero-GHG objectives and circular economic approach) and national needs (e.g., water scarcity and lack of resources). In particular, the analysis revealed that, while countries have shown different interests and research needs, shifting towards more innovative treatment technologies could create many opportunities, considering environmental, economic, and social impacts. Furthermore, the role of stakeholders, policymakers, funding agencies, in encouraging collaborative studies and initiatives was also explored, highlighting the need for more collaborative studies capable of better integrating available resources in order to address common issues, such as water scarcity and energy demand.

Marked differences in how various fields tended to address the topic of water-related LCA were shown, especially regarding “Environmental Science” and “Environmental Engineering”. In detail, the first tended to focus more on general aspects of individual technologies, while the latter usually focused more on combination of multiple processes and on more industrial issues, highlighting how more interdisciplinary and cross-sector studies could offer more complete insights into water-related technologies and processes, greatly enhancing the effectiveness of policy-making process.

Keyword co-occurrence and evolution mapping revealed which topics have been more investigated until now, as well as those that are now receiving more attention, such as greywater reuse, rainwater harvesting, and advanced AOPs. In particular, while conventional treatment methods are still widely applied and studied, many stakeholders and decision-makers are understanding their limitations and encouraging further studies on more innovative and sustainable alternatives. In this context, the incorporation of machine learning- and AI-based technologies into LCA and LCC frameworks will provide new opportunities to improve and optimize processes for water treatment, use, and reuse, thus leading to the development of more convenient and efficient water management strategies characterized also by high social benefits and low environmental impacts.

5. Chapter 5: Development and testing of a nanobubble-based hydrodynamic cavitation system

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5.1 Introduction

In this chapter, the results related to the manuscript “Development and testing of a nanobubble-based hydrodynamic cavitation system” are reported.

In recent years, hydrodynamic cavitation (HC) has emerged as a promising method for contaminant degradation and removal, offering a scalable, sustainable, and energy-efficient alternative to conventional treatment technologies while having fewer requirements than energy-intensive ultrasonic cavitation. In this thesis, the potential of enhancing HC efficiency using a nanobubble-based system through a specifically designed Venturi tube was explored. In detail, various Venturi tube prototypes were constructed and tested to identify the design that most effectively promoted the presence of nanobubbles, and the best alternative was used to build and test a pilot-scale hydrodynamic cavitation system for the degradation of organic substances.

The Venturi tube design was developed by integrating results from existing research and optimizing parameters through Design of Experiment (DOE) methodologies to enhance nanobubble generation. Preliminary test results from the prototypes were then used to develop a neural network model, enabling further exploration of alternative Venturi designs that were initially dismissed due to manufacturing constraints but showed potential for optimized bubble characteristics.

The efficiency of the hydrodynamic cavitation system was evaluated for the degradation of organic compounds, including humic acid (HA), Reactive Red 120 (RR120), and Congo Red (CR). These tests were conducted both in the presence and absence of oxidants, specifically H_2O_2 and Sodium persulfate, to assess their influence on degradation efficiency.

5.2 Experimental

5.2.1 Venturi tube design for Nanobubble generation

In order to investigate the influence of nanobubble-based hydrodynamic cavitation, we aimed to optimize a Venturi tube for generating smaller-diameter nanobubbles with higher Z-potential and higher frequency. Pertinent previous works were reviewed to compile findings on various geometrical parameters affecting Venturi tube performance. In detail, the following studies provided key insights:

- **Pourkarimi et al. (2017b)** [436] found that smaller internal diameters (2.2mm and 1.5mm) result in higher pressure, producing smaller bubbles.
- **Xiong and Felicia (2015)** [437] investigated 26 Venturi designs, assessing internal diameter (D_{in}), throat diameter (D_t), throat length (L_t), inlet angle (α°), and outlet angle (β°), establishing optimal ranges for bubble size distribution.
- **Deng et al. (2020)** [438] focused on feed diameter and throat diameter ($D_{in}=11\text{ mm}$, $D_t=1.5\text{ mm}$), offering further insights into bubble size and distribution.

Through these findings, it was determined that optimizing throat diameter (D_t), throat length (L_1), divergence length (L_2), and divergence/convergence angle (α) could enhance bubble size distribution, Z-potential, and diameter. Consequently, in order to systematically design the Venturi tube, the Design of Experiment (DoE) with Response Surface Methodology (RSM) was employed. This approach elucidated the connections between input variables (i.e., L_1 , L_2 , D_t , α) and outputs (i.e., bubbles' diameter, frequency, and Z-potential) to maximize coverage of the design space and minimize the number of experiments [439]. The DoE process was conducted using Design Expert software (Version 13), following the following steps: i) input variables were identified and introduced to the RSM model, ii) levels and ranges for these factors were determined based on the literature review and thesis objective (i.e., maximizing bubble durability and treatment efficiency), and iii) a full factorial design was chosen to ensure comprehensive exploration of parameter combinations. As a result, the RSM model suggested 24 runs with specific combinations of each factor, providing a structured experimental plan. The complete list of designs is shown in Table 5-1, and the Venturi tube design is shown in Figure 5.1.

Table 5-1: DOE suggested designs for the Venturi tube using full factorial design and RSM modeling

Design	D_t (mm)	L_1 (mm)	L_2 (mm)	α (°)
1	1	9.85	72.85	2.14
2	1.5	7	75.7	2.05
3	1.5	7	70	2.22
4	0.5	7	70	2.05
5	2	9.85	72.85	2.14
6	1	9.85	72.85	1.96
7	2	4.15	67.15	1.96
8	1.5	7	70	1.88
9	1	4.15	67.15	1.96
10	2	4.15	72.85	2.14
11	2	4.15	72.85	1.96
12	1	4.15	72.85	1.96
13	2	9.85	67.15	2.14
14	2	4.15	67.15	2.14
15	1	4.15	72.85	2.14
16	2.5	7	70	2.05
17	2	9.85	67.15	1.96
18	1.5	7	64.3	2.05
19	2	9.85	72.85	1.96
20	1	4.15	67.15	2.14
21	1.5	4.15	70	2.05
22	1	9.85	67.15	2.14
23	1	9.85	67.15	1.96
24	1.5	12.7	70	2.05

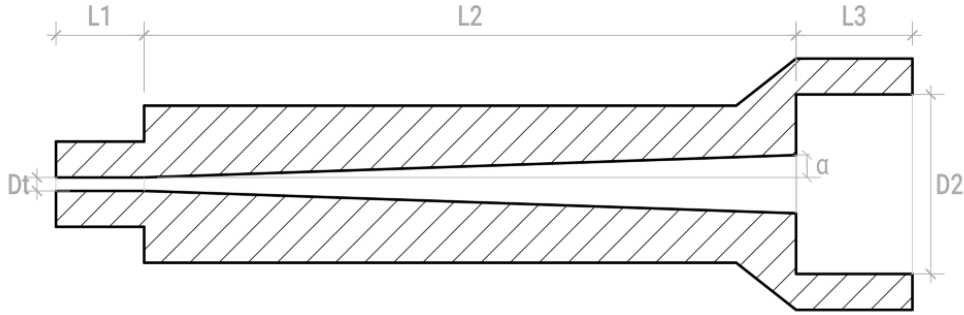


Figure 5.1: Venturi tube general design specifics, including the throat diameter (D_t), throat length (L_1), divergence length (L_2), divergence angle (α), and the connecting points to the other pieces, including (D_2) and L_3 .

Due to time and budget constraints, testing all 24 designs was impossible. Consequently, two designs were eliminated: Design 16 ($D=2.5\text{mm}$) was excluded based on literature findings indicating suboptimal performance and Design 4 ($D=0.5\text{mm}$) was excluded due to manufacturing challenges, including the risk of breaking the drill during production [119,438]. Finally, 10 prototypes (Table 5-2) were selected out of the remaining designs, focusing on configurations most likely to optimize bubble diameter, size distribution, and Z-potential based on literature information. The selected prototypes included four designs with $D=1\text{mm}$ (Design 20, 12, 22, and 6), four configurations with $D=1.5\text{mm}$ (Design 21, 18, 3, and 24), and two designs with $D=2\text{mm}$ (Design 7, 5).

Furthermore, Polytetrafluoroethylene (PTFE) was chosen for constructing the Venturi tube prototypes due to its chemical stability, cost-effectiveness, malleability, and sufficient hardness for maintaining structural integrity.

The prototypes were tested using a cavitation system equipped with a water jet pump (i.e., $P=\text{ca.}4\text{bar}$, Head=55m, Power=1.3kW, Flow rate(Q)=6000L/h) connected in-line with a laser particle size analyzer (LPSA, Malvern Mastersizer 2000) (Figure 5.2) to measure nanobubbles diameter and frequency [440]. In detail, each run lasted at least 45 minutes to ensure the system reached equilibrium conditions. Z-potential of the nanobubbles was measured by analyzing a 2 mL sample taken directly from the cavitation tank using Dynamic Light Scattering (i.e., HORIBA - SZ100) [441,442], and this measurement was taken three times for each prototype to ensure reproducibility and accuracy of the bubbles' characteristics.

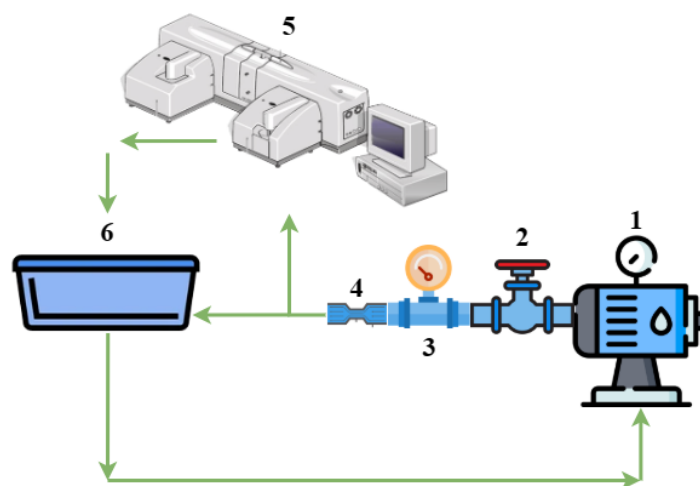


Figure 5.2: Schematic setup of laser particle size analyzer and bubble system for nanobubble size measurement: 1. Pump, 2. Control valve, 3. Manometer for pressure control, 4. Venturi tube, 5. Laser particle sizer (LPSA), 6. Water container

Table 5-2: Selected runs from DoE suggested experiments

Run	D_t (mm)	L_1 (mm)	L_2 (mm)	α (°)
3	1.5	7.00	70.00	2.22
5	2.0	9.85	72.85	2.14
6	1.0	9.85	72.85	1.96
7	2.0	4.15	67.15	1.96
12	1.0	4.15	72.85	1.96
18	1.5	7.00	64.30	2.05
20	1.0	4.15	67.15	2.14
21	1.5	4.15	70.00	2.05
22	1.0	9.85	67.15	2.14
24	1.5	12.7	70.00	2.05

5.2.2 Pilot plant and testing conditions

The following section describes the design and operating conditions of the pilot-scale HC system, as well as its testing conditions (i.e., the initial concentration of each substance, amount of oxidants, sampling). The architecture and parameters of the investigated neural network are also reported.

5.2.2.1 Cavitation system

As shown in Figure 5.3, the hydrodynamic cavitation system is composed of i) a not-self-priming pump (i.e., Sterwins 1.3kW, 5.5bar, 55m, 6000L/h), ii) a tank with a volume of 20L (38L×26.5W×28.5H) iii) the designed Venturi tube, iv) an heat exchanger to avoid increasing the temperature over 40°C, v) temperature and pressure indicators, and the necessary valves and discharge points. All pipes used were realized in PVC, with an internal diameter of 1 inch, while valves were made of stainless steel.

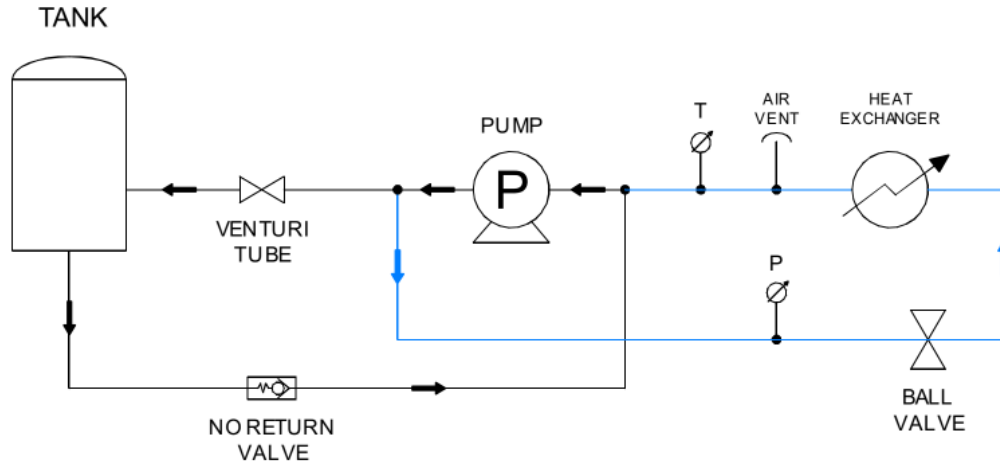


Figure 5.3: Schematics of hydrodynamic cavitation device

In detail, a bypass between the pump and the venturi tube was included to increase pressure and avoid pressure spikes safely. The heat exchanger consisted of a plastic pipe spiral submerged inside a tank filled with cooling water (100L, 15-20°C, 27.52L/h), and after cooling down, it flowed back into the pump. The flow directed to the Venturi tube was adjusted via the control valve to build the pressure for cavitation up to ca.4bar, as suggested by Zheng et al. (2022) [108]. The flow from the cavitation tank was redirected to the pump, thus creating a closed system.

The system was loaded for each test from the bottom of the heat exchanger (i.e., point No.2), as shown in Figure 5.4, to minimize the amount of air inside the system by pushing it out of the air-release valve (i.e., the valve before point No. 6). Finally, the pump and the rest of the system were filled from a valve situated above the pump.

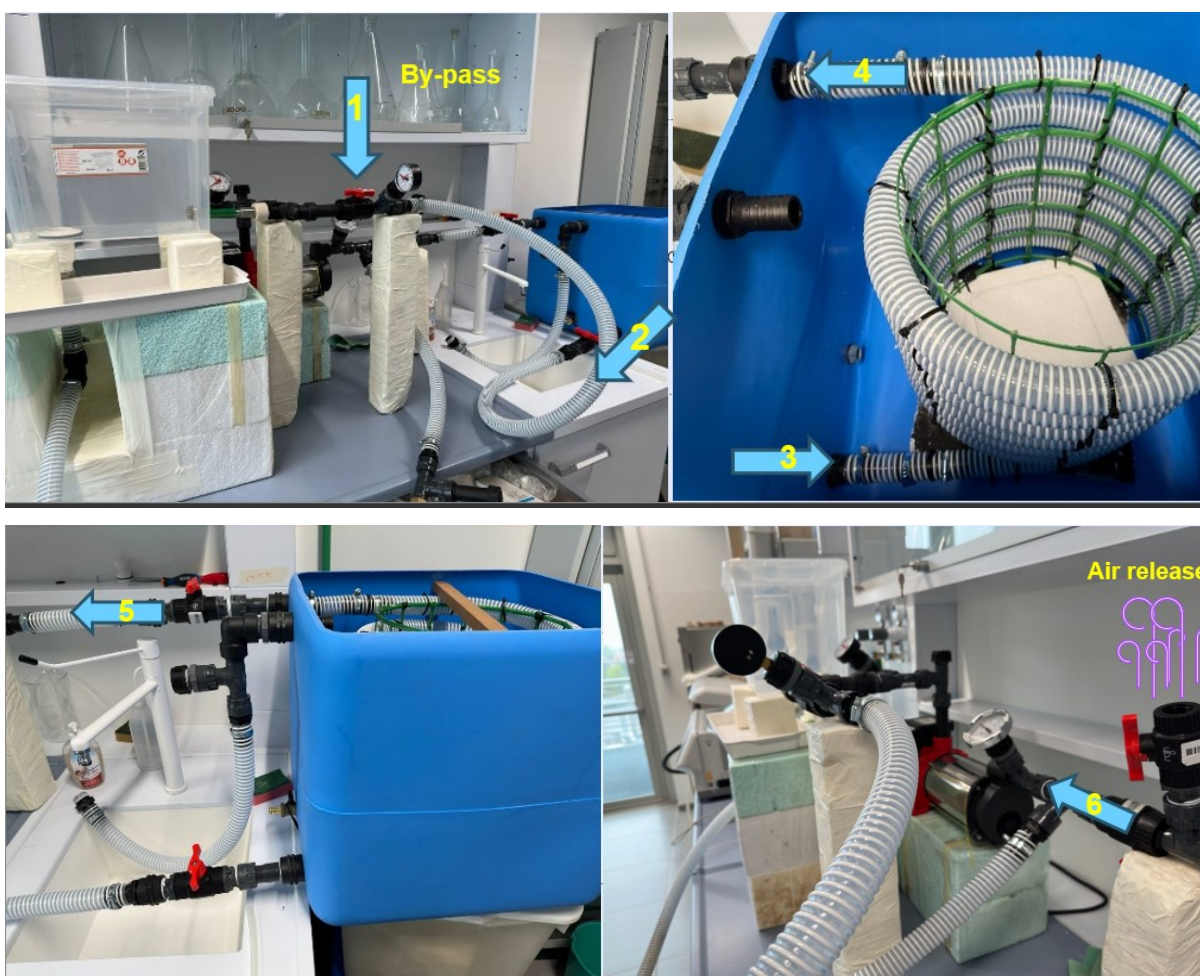


Figure 5.4: Pilot scale hydrodynamic cavitation system: 1) by-pass control valve, 2) by-pass flow into the heat exchanger, 3) point of system filling, 4) exit flow from the heat exchanger 5) flow to air release, and 6) flow back into the pump.

In order to test the efficiency of the HC system, the following substances were selected (Table 5-3):

- Humic acid (HA) as a proxy of NOM;
- Congo Red (CR) and Reactive Red 120 (RR120) as representative industrial dyes.

It should be noted that the concentration of NOM used in the experiments was taken based on the concentration range of organic matter in freshwater (i.e., 2-10 ppm), since the aim of the experiments with NOM was to understand the interference effects on drinking water production. While the concentration of dyes was simulated based on textile effluent. Similarly, the concentration of salt was also based on surface water for NOM tests and industrial effluent for dye tests. These measures were taken to keep the system less complex and to mitigate the risk of cross contamination from one test to the other. However, it is strongly advised that further tests can be conducted with real textile wastewater to better understand the effect of HC in a more complex environmental matrix.

Table 5-3: Initial concentration of selected substances

Substance	Initial Concentration	Reference
HA	ca.4 mg/l	[443,444]
CR	ca.10 mg/l	[445,446]
RR120	ca.15 mg/l	[258,447]

During each test, 15mL samples of the treated solution were taken every 5, 10, 15, 20, 40, 60, 80, 100, 120 minutes. When oxidants were used, 0.5mL of a 16.53mM NaSO₃ solution was added to each sample to stop any further reaction of any remaining radicals.

5.2.2.2 *Use of oxidants*

The literature indicates that sulfate and hydroxyl radicals can be used in the removal of a number of organic substances, such as NOM [448], pharmaceuticals [449], pesticides [450], and dyes [451]. However, very few works investigated the use of conventional hydrodynamic cavitation together with such oxidants, especially at relatively large scales (e.g., 5-15L). Regarding NOM, only Araujo et al. (2020) [126] reported on an 8L HC system using H₂O₂ for NOM degradation.

Additionally, all other works focused on cavitation in the presence of sulfate and hydroxyl radicals report results based on the use of ultrasonic cavitation at much smaller scales, such as the work of Wang et al. (2015) [448] which treated 200mL of water using ultrasonic cavitation and potassium persulfate or the work of Ji et al., (2020) [444] who degraded 100mL of a humic acid solution using an ultrasonic cavitation system in the presence of UV radiation and potassium persulfate.

Based on these studies, hydrogen peroxide (H₂O₂) and sodium persulfate (Na₂S₂O₈) were selected as oxidants to optimize the performance of the tested cavitation system. Sodium persulfate was chosen from among various peroxydisulfates (i.e., ammonium, potassium, and sodium persulfate), and both oxidants were used at a 230:1 molar ratio with each target compound.

Additionally, the effect of salt in hydrodynamic cavitation and persulfate was also assessed, for the first time in this work for NOM, to better account for the real conditions since water bodies contain salt that could interfere with the removal process. The amount of salt chosen for the NOM removal was taken from [452] and was set accordingly to 750mg/l. The effect of salt on NOM removal was investigated in the presence of persulfate and hydrogen peroxide. It should also be noted that using PS can alter the salinity of the water, while H₂O₂ does not have a similar effect.

5.2.2.3 *Batch tests without cavitation*

To estimate how much of the observed degradation could be attributed specifically to HC, a series of batch tests was carried out by preparing a separate 50 mL sample of each tested condition and heating it for 120 min within a temperature range comparable to average temperature of the pilot-scale tests (i.e., from 21 to 35°C with a slope of ca.0.35°C/min for 40min). Agitation of the solution was

maintained using a magnetic stirrer, and concentrations were measured only at the start and end of each test.

5.2.3 Neural network modeling of Venturi tube specifics and nanobubble characteristics

Various studies have investigated the relationship between Venturi geometry and nanobubbles' generation, such as the works of [436,453,454]. However, the response surface methodology (RSM) failed to explain the relationship between the Venturi tube geometry and the bubbles' characteristics (i.e., frequency, diameter, and Z-potential). Moreover, in a Venturi tube, even small changes in the geometry (i.e., divergence angle (α), throat diameter (D_t)) can significantly change the generated bubble's characteristics [455]. For this reason, the limited number of configurations suggested by the DoE approach may not cover the design space with sufficient precision.

For this reason, a neural network model was developed using the results (i.e., bubble diameter, frequency, and Z-potential) obtained for the prototype suggested by the DoE approach to i) explore other possible designs that would have needed more complex metalworking technologies (e.g., laser-drilling) ii) investigate the effects of smaller changes in the configuration with respect to the ones suggested by the DoE.

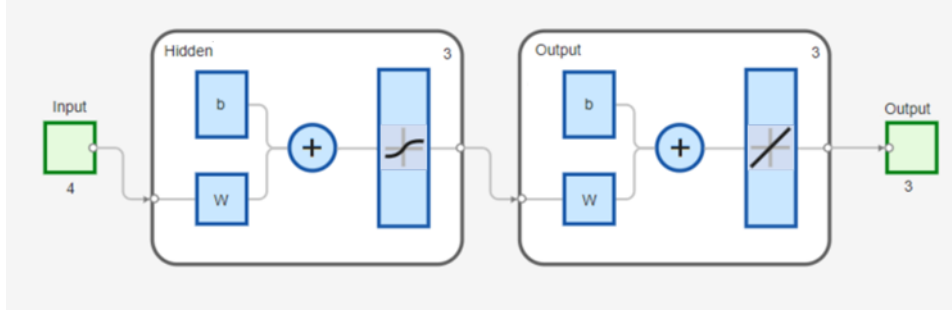
In detail, a neural network model was developed with 4 inputs (i.e., diameter (D_t), first length L_1 , second length L_2 , and divergence angle (α), and 3 outputs (i.e., diameter of the bubbles, frequency of the bubbles, and Z-potential).

Due to the small number of experiments, in order to prevent over-fitting, the size of hidden layers of the neural network was set at 3, the number of epochs was set at 10, and the weight regularization factor was set to 0.2 [456]. Similarly to what has been already reported for small datasets, no data was used for validation, but only for training (60%) and testing (40%) [457].

In order to select the optimal configuration, we tested several types of Neural Network models (i.e., radial basis function (RBFNN), general regression neural network (GRNN), and support vector machines (SVM)) alongside various learning algorithms (e.g., Levenberg-Marquardt (trainlm), Gradient Descent, and Bayesian Regularization) and learning parameters (e.g., weight, biases, number of epochs, learning rate, etc.). The final neural network selected was a feed-forward neural network composed of “trainlm” as the training function, “tansig” as the activation function in the hidden layers, and “purelin” in the output layer. The model had 3 hidden layers, a learning rate (performance regularization) of 0.2, and 10 as the number of epochs. The weights and biases for both the hidden layers and output layers are shown in Table 5-4.

Table 5-4: Weight and biases for hidden layers and output layers of the neural network

Weights connecting inputs to the hidden layer (W_1)	Biases for the hidden layer (b_1)	Weights connecting the hidden layer to the output layer (W_2)	Biases for the output layer (b_2)
$\begin{bmatrix} 0.16437 & -0.1646 & -0.3551 & -0.0708 \\ 0.7593 & 0.7047 & -0.1623 & 0.1295 \\ -0.4335 & 0.2571 & 0.4243 & -0.0675 \end{bmatrix}$	$\begin{bmatrix} 0.0646 \\ -0.0172 \\ -0.3035 \end{bmatrix}$	$\begin{bmatrix} 0.1570 & 0.4811 & -0.4871 \\ -0.2670 & -0.2104 & 0.7332 \\ -0.0663 & -0.8643 & 0.0698 \end{bmatrix}$	$\begin{bmatrix} -0.0403 \\ -0.0478 \\ 0.0496 \end{bmatrix}$

**Figure 5.5:** Neural network architecture: inputs, hidden layers, transfer functions, and outputs**Table 5-5:** Neural network architecture of hidden layers, transfer functions, learning rate, and epochs

Parameter	Value/name
Hidden Layer Sizes	3
Activation Functions	tansig
Training Function	trainlm
Performance regularization	0.2
Number of Epochs	10

In order to find the most optimized Venturi tube design, all possible combinations of design characteristics considered by the DOE (i.e., D_t : 0.5-2.5, L_1 : 4.15-12.7, L_2 : 64.3-75.7, and α : 1.88-2.22) were used as inputs for the trained neural network to estimate the resulting nanobubbles' frequency, diameter, and Z-potential. In detail, the DoE approach was used to select 20 equidistant points within each parameter's range.

Additionally, in order to find the most optimized combination of parameters, a scoring system was developed using the characteristics of the nanobubbles produced by the final prototype (i.e., $D=151.7$ nm, Frequency=89.65%, and Z-potential=-15.4 mv) as the benchmark, as shown in Equation 20 - Equation 23.

$$D_{score} = \frac{(D_{benchmark} - D_i)}{D_{benchmark}} \cdot W_D \quad \text{Equation 20}$$

$$F_{score} = \frac{(F_i - F_{benchmark})}{F_{benchmark}} \cdot W_F \quad \text{Equation 21}$$

$$Z_{score} = \frac{(abs(Z_{benchmark}) - abs(Z_i))}{abs(Z_{benchmark})} \cdot W_Z \quad \text{Equation 22}$$

$$Tot_{score} = D_{score} + F_{score} + Z_{score} \quad \text{Equation 23}$$

Where D is nanobubble diameter, F is nanobubble frequency, Z is nanobubble Z-potential, and $W_{D/F/Z}$ are weight representing the importance of each parameter. In detail, higher scores are given for lower diameter, higher frequency, and higher absolute value of Z-potential than the benchmark. The weights of the scoring system ($W_D=0.4$, $W_F=0.2$, $W_Z=0.4$) were selected based on the fact that during water treatment, the diameter and Z-potential have been shown to have a more important role in bubbles' durability and radical generation than frequency [458,459].

5.3 Results and discussion

5.3.1 Venturi tube design and testing

Results of preliminary testing with Venturi tube prototypes (i.e., nanobubbles' frequency, average diameter, and Z-potential), along with the characteristics of each configuration (i.e., D_t , L_1 , L_2 , and α), are shown in Table 5-6 and Figure 5.6.

Table 5-6: Results for prototype Venturi tubes

Prototype	D_t (mm)	L_1 (mm)	L_2 (mm)	α (°)	Bubble diameter (nm)	Frequency (%)	Z-potential (mV)
20	1.0	4.15	67.15	2.14	101 ± 4	60.6 ± 7.0	-18.1 ± 0.7
12	1.0	4.15	72.85	1.96	448 ± 15	67.1 ± 15.1	-14.7 ± 1.3
22	1.0	9.85	67.15	2.14	357 ± 11	71.7 ± 14.4	-16.8 ± 0.8
6	1.0	9.85	72.85	1.96	152 ± 3	89.6 ± 6.9	-15.4 ± 1.1
21	1.5	4.15	70.00	2.05	166 ± 5	59.9 ± 21.8	-18.9 ± 0.7
18	1.5	7.00	64.3	2.05	609 ± 38	32.6 ± 11.8	-21.3 ± 1.5
24	1.5	12.7	70.00	2.05	333 ± 11	70.8 ± 2.2	-22.9 ± 1.2
3	1.5	7.00	70.00	2.05	475 ± 52	37.2 ± 20.6	-15.4 ± 0.6
7	2.0	4.15	67.15	1.96	693 ± 54	39.4 ± 23.2	-18.2 ± 0.4
5	2.0	9.85	72.85	2.14	473 ± 15	64.0 ± 18.9	-23.0 ± 0.4

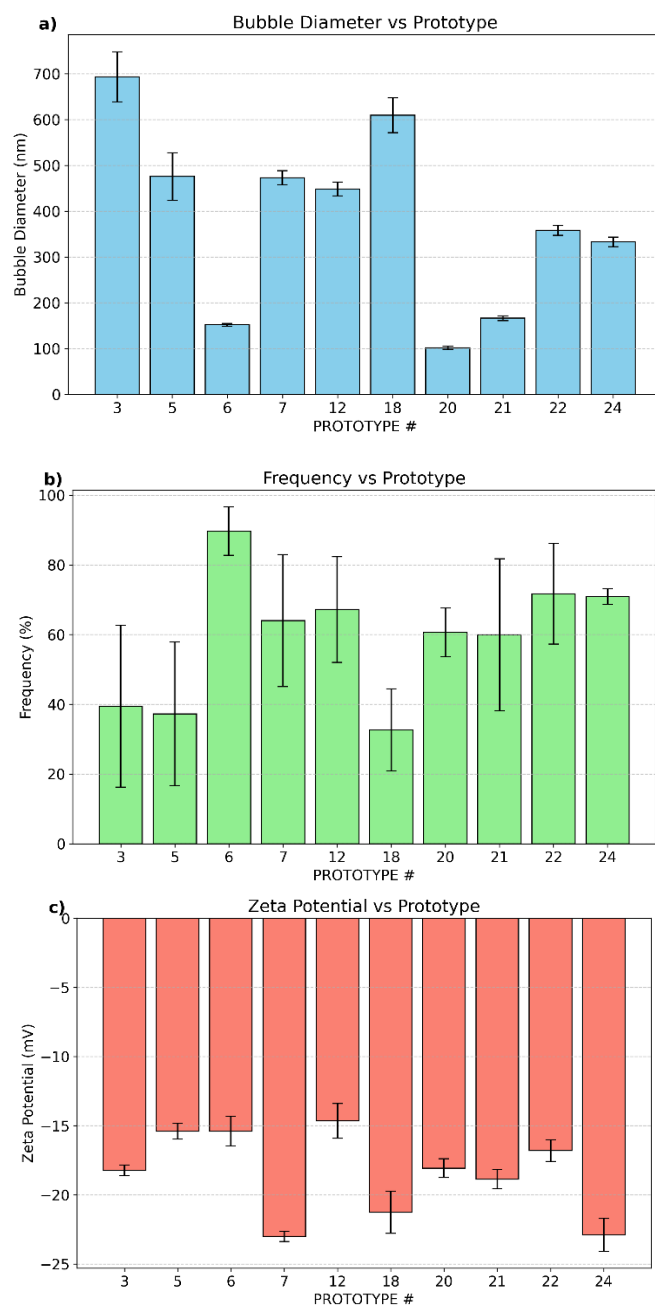


Figure 5.6: Average bubbles' diameter (a), frequency (b), and Z-potential (c) for each prototype. Error bars represent the standard deviation of the results.

Considering frequency, average diameter, and Z-potential, the results showed that Prototype 4 produced the best results: 89.7% of the produced bubbles had an average diameter of 151 nm and a Z-potential of -15.4mV. This prototype (i.e., i) $D_t=1\text{mm}$, ii) $L_1=9.85\text{mm}$, iii) $L_2=72.85\text{mm}$, and iv) $\alpha=1.96^\circ$) was then built from steel to carry out further tests without the potential release of chemicals (e.g., PFAS) into water.

It can be observed that reduction of throat diameter (D_t) leads to smaller bubbles formed with higher frequency. The rules of fluid mechanics can explain the reason. In detail, when the throat diameter (D_t) of a Venturi tube is reduced, the fluid is forced through a tighter space, increasing its velocity. This increase in velocity, as dictated by the continuity principle (Equation 24), indicates that the cross-sectional area (A) and the velocity (v) of the fluid remain constant along a streamline and Bernoulli's equation (Equation 25), which indicates that an increase in velocity is accompanied by a decrease in static pressure. This drop in pressure, along with the high velocity, creates a region of intense shear forces. These elevated shear forces are crucial for rapidly breaking the gas into smaller bubbles (i.e., smaller bubbles). However, if D_t reduction is combined with an increase in divergence angle (α), turbulence may become too strong, causing high possibilities of bubble coalescence or destruction of the formed bubbles. Thus, high divergence angles partially offset the high shear that encourages bubble formation [460].

$$A_1 \cdot v_1 = A_2 \cdot v_2 \quad \text{Equation 24}$$

$$P + \frac{1}{2} \rho v^2 + \rho gh = \text{constant} \quad \text{Equation 25}$$

Similarly, a longer L_1 can provide the necessary residence time for the flow to develop a stable shear profile, promoting the bubbles' surface charge and leading to a more negative Z-potential. In detail, the charge of the bubbles (nanobubbles) is negative as they acquire their charge from the attached $\text{OH}^\cdot$ radicals in the cavitation chamber (e.g., Venturi, orifice plate) onto their surface [461]. On the other hand, a shorter L_2 limits the time available for bubbles to coalesce, reinforcing both the high frequency of bubble formation and maintaining smaller bubble sizes. However, if L_2 is too short, there might not be enough time for $\text{OH}^\cdot$ radicals to attach to the bubble surface, thus decreasing the Z-potential.

5.3.2 Neural network modeling of Venturi tube specifics and nanobubble characteristics

In order to assess the performance of Venturi tube configurations suggested by the DoE that were not built due to technical difficulties, a feed-forward neural network was developed, as described in section 5.2.3. In detail, the results of the regression and fitting are shown in Figure 5.7 and Figure 5.8. It was shown that the regression coefficients for the training and test set were 0.80 and 0.69, respectively, and the overall regression coefficient of the model was 0.74.

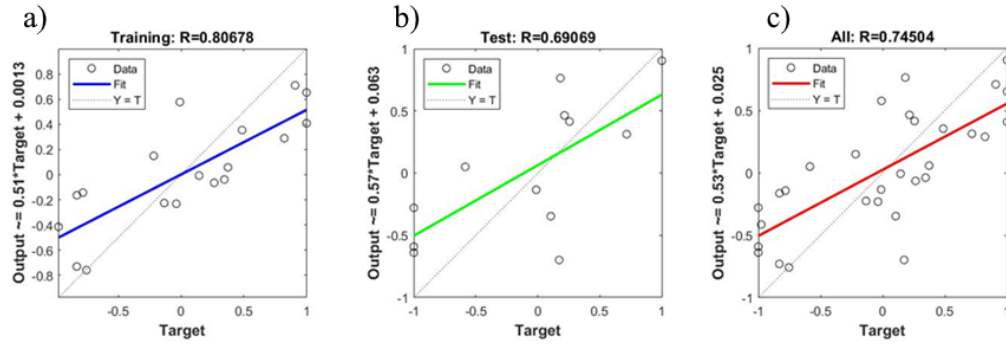


Figure 5.7: Model regression values (R^2) for training (a), test (b), and overall performance (c)

As shown in Figure 5.8, fitting charts plot the predicted vs. actual (target) to visually inspect how well the model has learned the underlying data patterns. Such plots help identify whether the network is overfitting (i.e., tracking training data closely but performing poorly on unseen data) or underfitting (i.e., failing to capture important patterns in both training and testing) by examining how closely the predictions align with the actual values across multiple samples [462]. The results of the fitting charts (Figure 5.8) highlighted that the model has shown good results for the nanobubbles' frequency and the Z-potential. However, the diameter of the bubbles was well represented only in the training test but was under- and over-estimated in the test set, indicating a possible overfitting of the model and thus the need for more prototypes to better understand the complex relationship between Venturi tube geometry and bubbles' diameter.

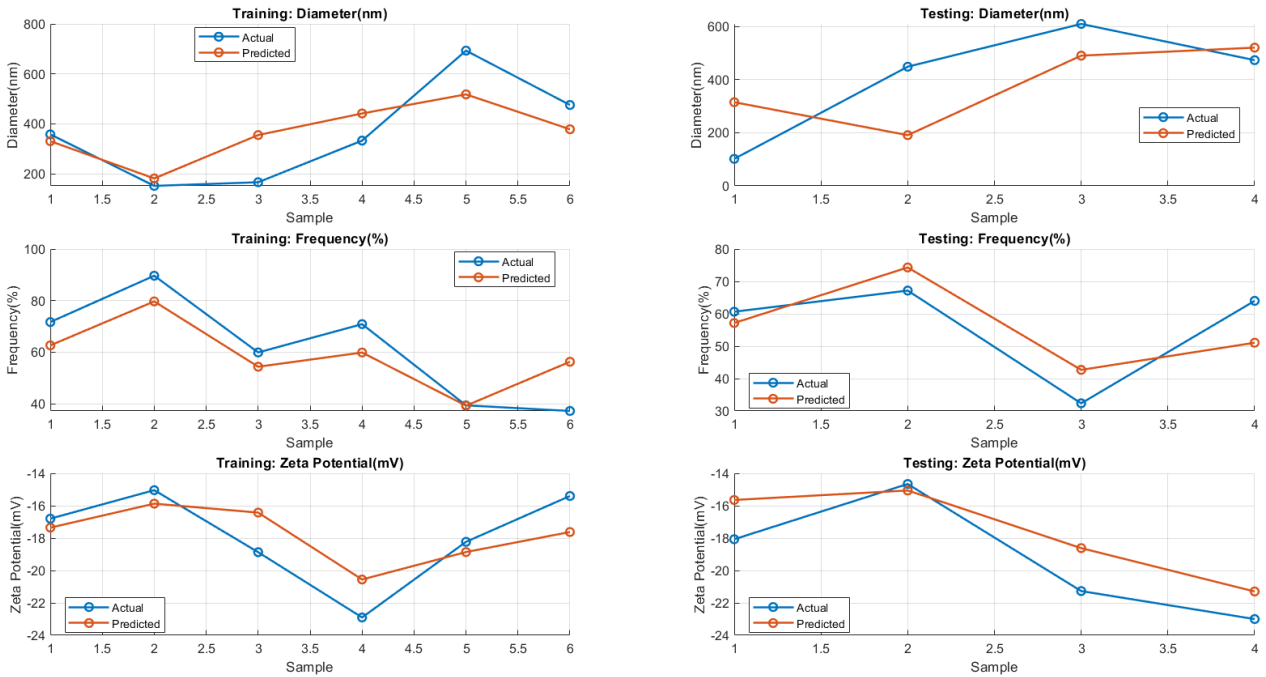


Figure 5.8: Train set data fitting (left column) and test set data fitting (right column) for diameter, frequency, and Z-potential.

Another common statistical value was also calculated to further evaluate the neural network's performance (i.e., Root Mean Squared Error (RMSE)) for each output (i.e., bubble diameter, frequency, and Z-potential) regarding both the training and test set (Table 5-7). The acceptable range for RMSE values differs based on the studied topic. However, for small datasets, RMSE values around 10-20% of each input's range are considered acceptable [463]. In detail, since the ranges for each model input were: i) diameter (i.e., from ca. 100 to 700nm), ii) frequency (i.e., from ca. 30 to 90%), and iii) Z-potential (i.e., from ca.15 to 23mV), acceptable RMSE values would be below ca. 120, 12, and 1.6, respectively. RMSE results also showed that the overall prediction accuracy for diameter was not satisfactory (i.e., RMSE of 151) but was more acceptable for both the frequency and Z-potential (i.e., RMSE of 9.9 and 1.8, respectively). This also indicated the need for data on additional prototypes to improve the neural network performance.

Table 5-7: Overall error values of the developed neural network

Output	RMSE	
	Training	Test
Diameter	122	180
Frequency	10.8	9.1
Z-potential	1.7	1.9

Moreover, the correlation between the predicted and actual values of the bubbles' diameter, frequency, and Z-potential was also estimated (Table 5-8). The results indicated both positive and negative correlations, highlighting that specific tradeoffs between nanobubble characteristics must be taken into account when choosing the optimal Venturi tube configuration based on the specific application of the cavitation system. For example, de Medeiros and Baltar (2018) [464] reported that a cavitation system used in mineral floatation processes gives better results when bubbles have smaller diameters than when they have higher Z-potential. In our case, nanobubbles' diameter showed a negative correlation with both frequency and Z-potential, showing that bubbles with lower diameters will generally be less stable and frequent in the studied range (i.e., from ca.100 to 600nm) [465].

Table 5-8: Correlation coefficients (R) between actual and predicted values

	Bubble diameter (m)	Frequency (%)	Z-potential (mV)
Bubble diameter (m)	1	-0.6	-0.7
Frequency (%)	-0.6	1	0.5
Z-potential (mV)	-0.7	0.5	1

As reported in Table 5-9, the optimization carried out with the neural network highlighted a Venturi tube configuration of: i) $D_t=0.5\text{mm}$, ii) $L_1=10.68\text{mm}$, iii) $L_2=75.7\text{mm}$, and iv) $\alpha=1.88$ as the one giving the best results (i.e., bubble diameter of 90.43 nm, bubble frequency of 89% and Z-potential of -14.81mV). In accordance with correlation coefficient results, this shows that a slight increase of

Z-potential must be taken into account to obtain a 40% reduction of the nanobubbles diameter (i.e., more efficient production of OH[•] radicals). It should also be noted that this Venturi tube with this configuration could be realized using laser drilling technology due to its small throat diameter, highlighting the importance of design accuracy in treatment efficiency.

Table 5-9: Comparison between nanobubble characteristics obtained from the optimized Venturi tube design based on DoE and neural network results.

Parameters	DoE suggested design (D _t =1mm, L ₁ =9.85mm, L ₂ =72.85mm, α =1.96°)	Neural network suggested design (D _t =0.5mm, L ₁ =10.68, L ₂ =75.7mm, α =1.88°)
Bubbles diameter	151	90
Bubbles Frequency	89%	89%
Bubbles Z-potential	-15.4	-14.8

5.3.3 Degradation of humic acid, Reactive Red 120, and Congo Red with HC

5.3.3.1 HC in neutral and acidic conditions

The overall degradation of the selected chemicals after 120 minutes is shown in Figure 5.9, showing that no degradation occurred in neutral conditions (pH=7.4) without the use of additional oxidants, as hydroxyl radicals do not persist in the solution long enough to interact with the substance to be degraded [466]. When HC was used in acidic conditions (pH=2), similar degradations were reached for HA and RR120 (ca.30%), while CR degraded ca.22%. In detail, CR has been shown to be less susceptible to hydrolysis than RR120 and HA due to its highly resonance-stabilized chemical structure thanks to the presence of two azo (–N=N–) bonds linking its biphenyl core with the two sulfonated naphthalene rings [467]. The limited degradation of the studied substances in the absence of additional oxidants could also be linked to the presence of several polar functional groups (e.g., -OH, -COOH, -SOOH), leading these molecules to preferably remain in the liquid phase rather than in the gas phase when cavitation bubbles are formed, thus escaping from the bubbles' collapse [468]. Other studies have reported on using conventional HC systems (i.e., microbubble-based) in the absence of additional oxidants to remove the target compounds. For example, Dehghani and co-workers [138] reported CR degradation of ca.12% using a 4L HC system (P=4bar and initial CR concentration of 20 mg/l), Saharan et al. (2011) [258] degraded ca. 20% of RR120 using a 15L HC system (P=5bar and initial RR120 concentration of 35mg/l), and Araujo and co-workers (2020) [126] used a 9L HC system (P=7.3bar and initial HA concentration of 100mg/L) to degrade about 16% of HA. While our results cannot be directly compared with these studies due to differences in system configuration and testing conditions, it can be seen that our nanobubble-enhanced cavitation system obtained generally better results. In detail, this could be attributed to nanobubbles' smaller diameters,

larger surface area-to-volume ratio, and higher Z-potential. This leads to a longer residence time in the system, which increases the probability of the selected molecules reacting with $\text{OH}^\cdot$ radicals resulting from bubbles' collapse, thus increasing the degradation.

Moreover, while degradation pathways were not investigated in our study, several authors have reported on how the target chemicals are degraded in the presence of $\text{OH}^\cdot$ and $\text{SO}_4^\cdot$ radicals. In detail, Kim and co-workers (2024) [469] used LC-MS to analyze the degradation of CR using an HC system (only in the presence of $\text{OH}^\cdot$ radicals), showing the degradation to include a sequential break of the two azo bonds to produce aromatic amines. Similarly, Zhang et al. (2007) [470] reported that also in the case of RR120, the $\text{OH}^\cdot$ radicals produced by cavitation can cleave the azo bonds. However, they also showed how they can react with the aromatic rings present in RR120 to form hydroxylated intermediates or to abstract hydrogen atoms, leading to further ring-opening reactions. As a result, aromatic rings in RR120 are oxidatively cleaved into smaller fragments such as catechols, phenols, and quinones, which can further oxidize to short-chain carboxylic acids. Furthermore, several authors reported on the oxidation of several functional groups present in HA (e.g, alcohols, carboxylic acids, ketones, ethers, amines) by $\text{OH}^\cdot$ radicals, leading to the formation of several intermediates [471,472]. In addition, aromatic rings where $-\text{OH}$ or $-\text{O}-$ groups are present were shown to undergo one-electron oxidation to form radical cations [126].

5.3.3.2 *HC in the presence of oxidants*

The results reported in Figure 5.9 show that the addition of both H_2O_2 and PS brought a marked increase in the degradation of all selected chemicals, ranging from 2 (for HA) to 4 times (for CR). In detail, H_2O_2 and PS had similar performance in the degradation of HA and RR120 (ca.60 and 95%, respectively), while PS was much more effective than H_2O_2 in aiding the degradation of CR (ca.90 and 67%, respectively). In fact, PS has been shown to better degrade organic contaminants than H_2O_2 in several water treatment processes [473] due to i) a slightly higher oxidation potential than $\text{OH}^\cdot$ radicals (i.e., ca.3.1 vs. 2.8V, respectively) [474]; ii) higher selectivity during reaction via electron transfer with organic compounds that contain unsaturated bonds or aromatic π electrons since $\text{OH}^\cdot$ radicals are non-selective radicals and may also react with other functional groups by hydrogen abstraction or electrophilic addition [475], iii) longer half-life than $\text{OH}^\cdot$ radicals (ca.35 μs vs 20ns, respectively), ensuring better mass transfer and higher probability to interact with the molecule to be degraded [476].

Dehghani et al. (2024) [138] also reported a marked degradation increase (i.e., from ca.12 to 63%) when using PS for CR removal in an HC system (P=4bar and 4L of treated water). The effect of PS was lower than the one found in the present study (i.e., from 22 to 93%), but this could be attributed to the lower oxidant:target chemical ratio used with respect to our experiment (i.e., 10.5:1 vs. 230:1)

and to the different scales of the cavitation system (i.e., 4L vs. 14L). PS was also used by Wang et al. (2015) [448] to increase the performance of an ultrasonic cavitation system (i.e., power of 200W, 200mL of treated water, and oxidant:target chemical ratio of 230:1) applied to degrade HA in acidic conditions. In detail, they showed the importance of PS thermal activation by comparing two tests at 35°C and 50°C, showing a degradation increase of ca.30% (i.e., from 60 to 90% mineralization). However, as reported by Yeneneh et al. (2024) [122], above ca.40°C the vapor pressure inside the bubbles produced using HC can reach values high enough to resist compression and prevent the walls from collapsing, absorbing some of the shock and energy released, thus reducing the radical generation and cavitation effect at the same time. For this reason and to preserve the pumping system, HC applications are usually limited to lower temperature ranges (e.g., up to 35°C) through the use of heat exchangers [477]. Their results at 35°C were also quite similar to those obtained in this study for HA mineralization in the presence of PS (ca.60%), showing a similar performance of HC and ultrasonic cavitation in PS activation.

Furthermore, the effect of H₂O₂ on the degradation of CR was investigated by Askarniya et al. (2020) [446] in an HC system (i.e., P=6bar, pH=3), using an orifice plate and higher oxidant:target chemical ratio than the present work (i.e., 530:1 vs 230:1) but reaching lower degradation of CR after 60 minutes compared to the this study (i.e., 35% vs 64%). This shows that our nanobubble-based HC system using a Venturi tube combined with a lower pH (i.e., 2 vs. 3) was more efficient in activating the H₂O₂ inside the cavitation chamber than an orifice plate, thus resulting in higher degradation of CR.

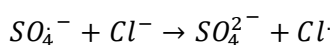
The use of HC in the presence of H₂O₂ for the degradation of RR120 was also investigated in the work of Saharan et al. (2011) [258] (i.e., P=6bar, 6L, and pH=2) and they reached similar decolorization to the one in the present study (i.e., ca.95%), using a lower oxidant:target chemical ratio (90:1 vs 230:1). This shows also the need for further optimization of our HC system, with the aim of minimizing reagent consumption.

5.3.3.3 HC in the presence of salt

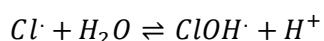
The presence of salts has been shown to interfere with several AOPs [478]. However, to the best of the authors' knowledge no previous work has been done to investigate the effect of salinity on Venturi-based cavitation systems for the degradation of the selected chemicals. In detail, the only work dealing with the effects of salinity on Venturi-based cavitation systems was carried out by Innocenzi and co-workers (2022) [479] for a solution of methyl-orange (i.e., P=5bar, pH=2, 1L of treated water, and initial concentration of dye and NaCl of 5mg/L).

The results (Figure 5.9) showed that the presence of salt markedly decreased the degradation of HA and RR120 with both of the tested oxidants, while results on the effect of salt on CR degradation were not available due to precipitation of particulate matter during the tests.

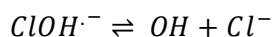
In detail, adding salt decreased HA's overall degradation by ca.7% with both H₂O₂ and PS, while the effect on RR120 elimination was slightly more noticeable (reduction of 12 and 17% in the presence of H₂O₂ and PS, respectively). The effect of salinity could be linked to the scavenging of reactive radicals (i.e., OH[•] and SO₄^{•-}) by chlorine atoms, as shown in Equation 26-Equation 29 [480]. Furthermore, self-quenching reactions of chlorine radicals (Equation 29) can result in the formation of the less-reactive chlorine dimer radical (Cl₂^{•-}, E°=2.126 V), which may further inhibit degradation. It is worth noting that after the treatment the pH should be brought back up to meet the discharge regulations (i.e., pH=5.5-8) [481]. Moreover, the reported degradation percentages are mineralization for NOM and primary degradation for dyes.



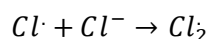
Equation 26



Equation 27



Equation 28



Equation 29

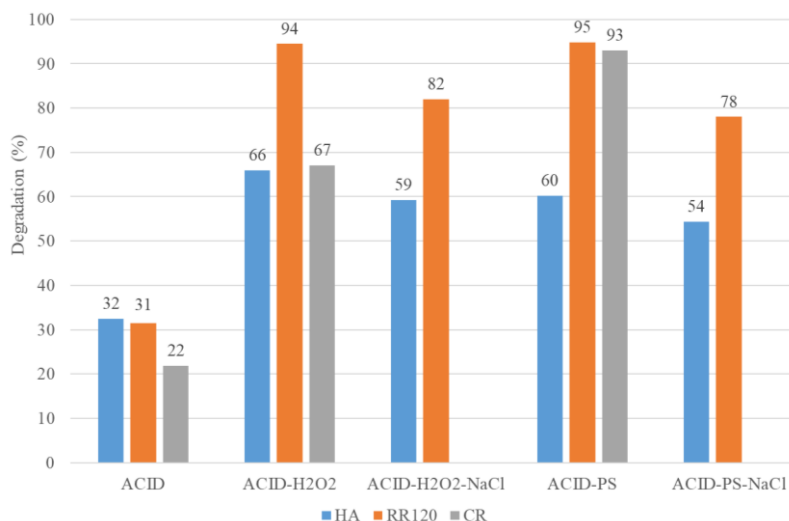


Figure 5.9: Degradation (%) comparison between humic acid, Reactive Red 120, and Congo Red in 120 min

5.3.3.4 Batch tests in the absence of HC

To better identify the contribution of cavitation to the observed degradation of the target chemicals, a series of batch tests were conducted by preparing a separate 50mL sample of each tested condition and heating it for 120 minutes within a temperature range comparable to the average temperature of the pilot-scale tests. The results (Figure 5.10) showed low degradation (i.e., below 5%) for all tested conditions, showing the fundamental role of the cavitation chamber in the oxidants' activation.

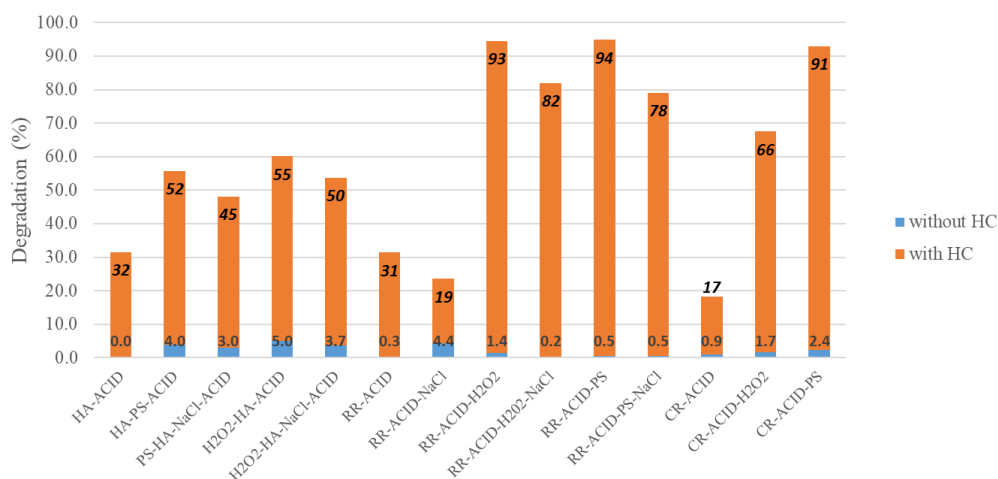


Figure 5.10: Batch test results for all tested substances in the absence of HC and comparison with the respective degradation test with HC.

5.3.3.5 *Degradation kinetics*

Kinetic analysis is essential to elucidate the underlying reaction mechanisms and rate-determining steps of a degradation process, allowing to more effectively optimize the removal [482] and better compare the effects of different testing conditions. In fact, several studies have shown that degradation occurs rapidly at the start of the process due to the high availability of radicals and target chemical molecules [483,484], then the reaction slows down due to mass transfer phenomena as the reactants become less concentrated [485–487]. In detail, the parameters (i.e., reaction constant (k) and regression coefficient (R^2)) of the estimated kinetics for each experiment are shown in Table 5-10 and Figure 5.11-5.13. The results showed marked differences in the degradation rate of the studied chemicals when only HC was employed, with HA and CR showing similar kinetic constants (i.e., 0.2 and 0.4hr⁻¹, respectively) while RR120 was degraded one order of magnitude faster (i.e., $k = 5.36\text{hr}^{-1}$). It should be noted that, while the degradation of RR120 was much faster than CR, the final decolorization reached a difference of about 10%, suggesting the presence of additional factors than only kinetic limitations, such as i) low availability of OH[•] radicals, ii) slow mass transfer between bubbles and bulk solution, and iii) different stability of the dyes.

The addition of PS showed a marked increase in the degradation rate for all tested substances, especially in the case of HA. In detail, the HA kinetic constant increased by ca. 50 times (i.e., up to 9.6hr⁻¹) while the increase for CR and RR120 was more limited (i.e., kinetic constants up to 2.4 and 9.9hr⁻¹, respectively). In addition, the results showed that the use of H₂O₂ also increased the degradation rate for all investigated substances, showing a faster reaction only in the case of HA and RR120 (i.e., kinetic constants of 15.9 and 25.6hr⁻¹, respectively) while the effect was lower than PS in the case of CR (i.e., kinetic constant of ca. 1.1hr⁻¹). Similarly to the results obtained regarding the overall degradation, this shows that PS is more effective in degrading CR than H₂O₂ (Figure 5.9), while both oxidants show a similar performance with HA and RR120.

The results showed that in the presence of H_2O_2 , the addition of salt decreased the kinetic constants of HA and RR degradation from 15.9 to 5.5hr^{-1} and from 25.6 to 2.5hr^{-1} , respectively (i.e., a decrease of ca. 3 and 10 times). This could be ascribed to chloride ions quenching a substantial fraction of hydroxyl radicals or converting them into chlorine radicals and related species (Equation 23-25), which exhibit lower oxidative potential [474]. On the contrary, when PS was used, salt showed a much more limited effect. In detail, the kinetic constant calculated for RR120 removal in the presence of PS and salt decreased only about 12% (i.e., kinetic constant from 9.9 to 8.8hr^{-1}), while it did not show a marked decrease in the case of HA (i.e., kinetic constant from 9.6 to 9.3hr^{-1}).

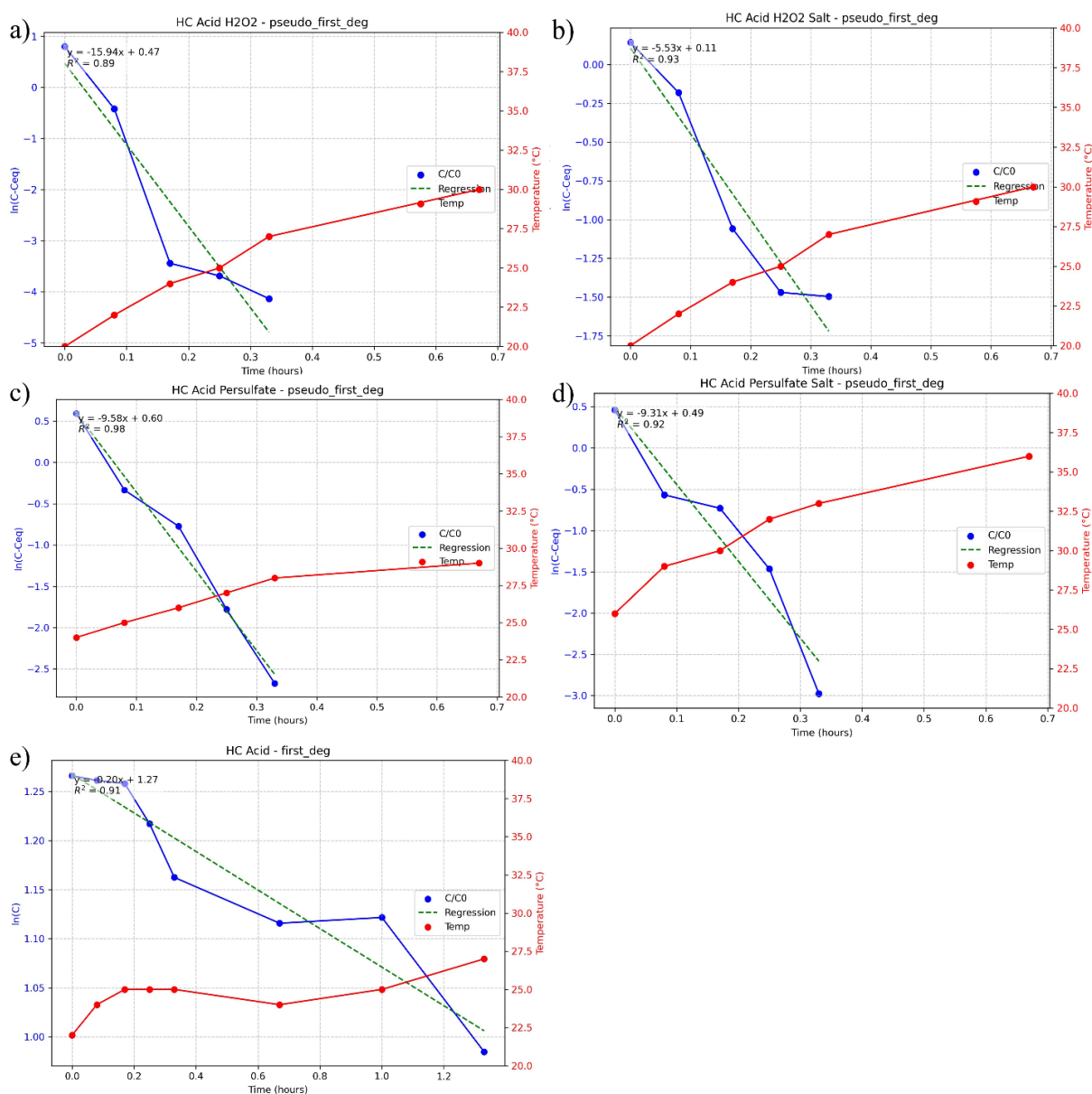


Figure 5.11: HA degradation kinetic models: H_2O_2 (a), H_2O_2 and salt (b), PS and Salt (c), PS (d), and HC alone (e)

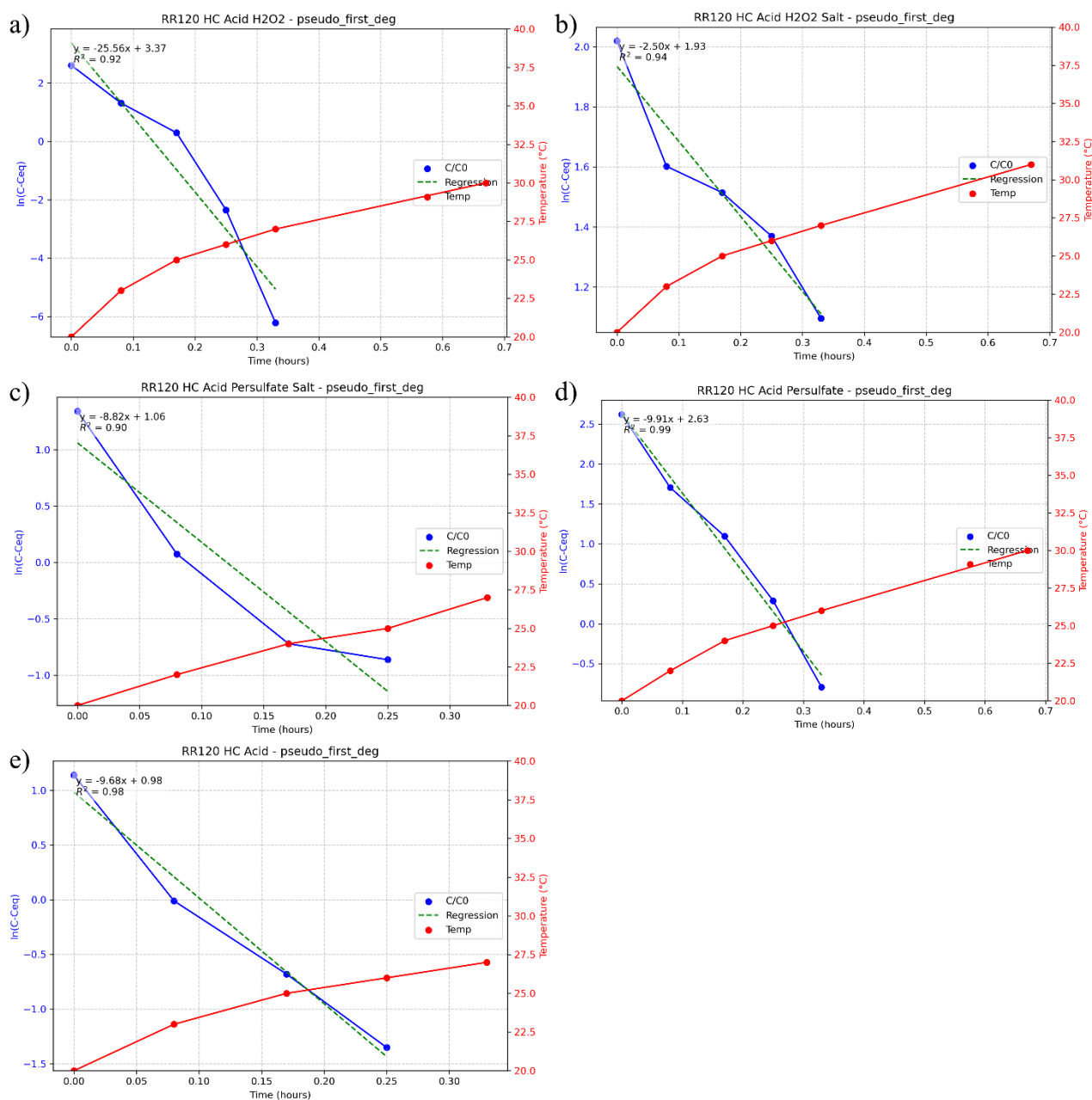


Figure 5.12: RR120 degradation kinetic models: H₂O₂ (a), H₂O₂ and salt (b), PS and salt (c), PS (d), and HC alone (e)

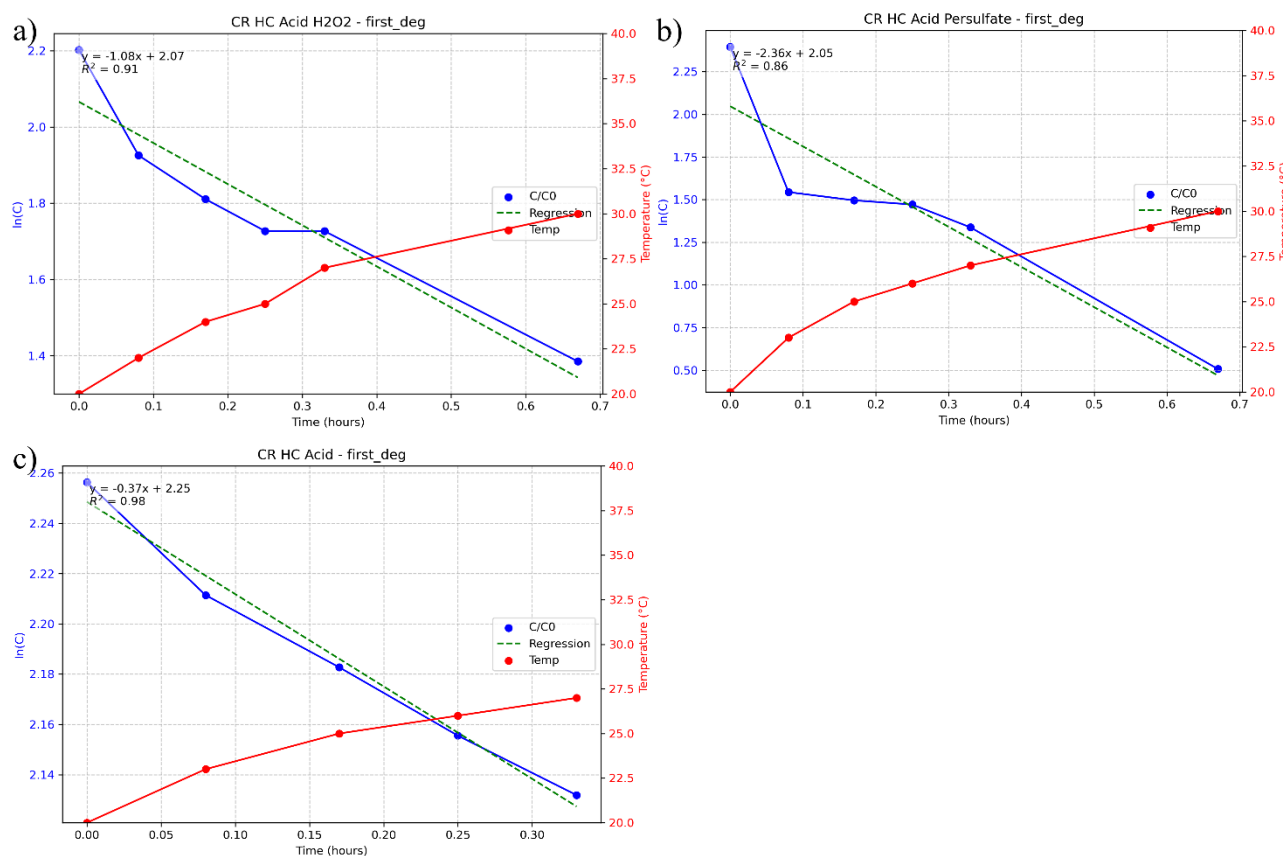


Figure 5.13: CR degradation kinetic models: H₂O₂ (a), PS (b), HC alone (c)

Table 5-10: Kinetic parameters of the advanced oxidation process for various testing conditions Kinetic parameters of the advanced oxidation process for various testing conditions

Test	Model	Rate (1/hr)	R ²	Reaction time (min)*
HA-ACID	First order	0.2	0.91	80
HA-ACID-PS	Pseudo-first order	9.6	0.98	40
HA-ACID-PS-NaCl	Pseudo -first order	9.3	0.92	40
HA-ACID-H ₂ O ₂	Pseudo -first order	15.9	0.89	40
HA-ACID-H ₂ O ₂ -NaCl	Pseudo -first order	5.5	0.93	40
RR120-ACID	Pseudo -first order	5.4	0.94	40
RR120-ACID-PS	Pseudo -first order	9.91	0.99	40
RR120-ACID-PS-NaCl	Pseudo -first order	8.8	0.9	20
RR120-ACID-H ₂ O ₂	Pseudo -first order	25.6	0.92	40
RR120-ACID-H ₂ O ₂ -NaCl	Pseudo -first order	2.5	0.94	40
CR-ACID	First order	0.4	0.98	20
CR-ACID-PS	First order	2.4	0.86	40
CR-ACID-H ₂ O ₂	First order	1.1	0.91	40

*The reaction time (min) indicates the time interval in which the kinetic model was assumed to be valid due to the restrictions of temperature (i.e., $T < 35^{\circ}\text{C}$ and $\Delta T \leq 5^{\circ}\text{C}$).

5.3.4 Economical evaluation

In order to assess the scalability and practical feasibility of the hydrodynamic cavitation process, an economic analysis was conducted by extrapolating experimental data to industrial-scale volumes. This involved calculating energy requirements, chemical reagent costs, and flow parameters under various operating conditions to evaluate cost-efficiency and guide real-world implementation [488].

In the initial stage, theoretical flow parameters were calculated based on Bernoulli's equation. Assuming negligible upstream velocity and setting the throat pressure equal to the vapor pressure of water (3171 Pa), it was determined that under a 4bar inlet pressure, the required theoretical velocity at the throat is approximately 28.2m/s. Using this velocity and a throat diameter of 1mm, the theoretical flow through the Venturi could be calculated. This target velocity is theoretically independent of the throat diameter, provided that cavitation occurs; however, in practice, achieving this velocity depends heavily on the flow rate and the geometry of the Venturi. As the diameter of the throat increases, the flow required to reach the same velocity also increases proportionally. Although the pressure necessary for cavitation does not change with the throat size, larger throat diameters result in reduced velocities for any given flow rate, making it increasingly unlikely to induce cavitation unless the flow is increased accordingly. For this reason, achieving efficient cavitation requires a substantial difference between the pipe and throat diameters to ensure the necessary pressure drop and high shear conditions.

Following this theoretical basis, pilot-scale measurements were incorporated into the evaluation. At pH=2, the actual flow through the 1mm Venturi was found to be approximately 75 L/h, or 0.075 m³/h. Given that the working volume of the test system was 14L, it was possible to calculate the number of turnovers per hour (tph) by dividing the volumetric flow rate by the system volume. To explore scale-up scenarios, an industrial-scale cavitation system was hypothesized with a volume of 10m³. By multiplying the pilot-derived tph value by the new system volume, the total flow required for the scaled process was estimated. Assuming the use of Venturi tubes with a throat diameter of 10 mm, the total number of Venturis needed could then be calculated by dividing the required total flow by the flow capacity of a single Venturi under equivalent conditions.

In order to estimate energy consumption, the power demand of the required pump was determined using the standard hydraulic power formula. In detail, energy consumption in kilowatt-hours (kWh) was then calculated by multiplying the pump's power output by the operational duration in hours necessary for a single batch treatment using Equation 30 to quantify energy costs, real-world

electricity prices (kWh cost) were retrieved from online sources [489], allowing the calculation of energy expenditure per batch.

$$P = \frac{Q_r \cdot \Delta P}{\eta_{tot} \cdot 1000} \quad \text{Equation 30}$$

Where P is the power (kw), Q_r is the flow (m/s), ΔP is the pressure difference (Pa), and η_{tot} is the total efficiency (i.e., pump, motor, and system efficiency).

Similarly, the cost of the chemical reactants was evaluated. This involved scaling the quantities used in the pilot system to match the industrial-scale volume by applying a volumetric factor equal to $V_2/V_1 = 10\text{m}^3 / 0.014\text{m}^3 = 714.3$. The mass of each required reactant was multiplied by this factor to project usage at the larger scale. Subsequently, cost per kilogram values for each chemical (retrieved from supplier websites) [490] were used to calculate the total cost per batch by simple multiplication. This approach was applied consistently across all chemicals, including oxidants and pH-adjusting agents such as lime. The latter was quantified based on the hydroxide ion concentration needed to neutralize the solution back to pH=7.

The entire process was then replicated for different assumed system volumes (i.e., 100, 250, 500, and 1000m³). In all cases, both energy consumption and chemical costs increased approximately linearly with volume, as expected in a batch process, summarizes the total treatment costs under different operating conditions (i.e., variations in pH and oxidant type) for each system size. The resulting data were visualized through a series of graphs, which showed that shortening the reaction time reduces the overall treatment cost, and that lowering oxidant dosages, when feasible, also contributes to economic efficiency. Despite its oxidative effectiveness, persulfate was found to be particularly costly and therefore less attractive for large-scale applications, highlighting the importance of balancing efficacy with affordability.

Regarding the pH, pH=5 and pH=7 follow the same methodological approach as described for pH=2. The only parameters that differ are the treatment durations and the mass of reagents applied, both of which affect the overall cost projections. These variations were incorporated into the overall economic model to assess how different chemical conditions influence process feasibility.

Table 5-11: Economical analysis of each scenario of various volumes of industrial effluent

Variables	Unit of Measure	Actual Pilot	Scenario 1	Scenario 2	Scenario 3	Scenario 4	Scenario 5
V ₁	m ³	0.01	0.01	0.01	0.01	0.01	0.01
V ₂	m ³	500.00	10.00	100.00	250.00	500.00	1000.00
d(throat)	m		0.01	0.01	0.01	0.01	0.01
Q _r *	m ³ /h		53.91	539.08	1347.71	2695.42	5390.84
Q _v **	m ³ /h		7.97	7.97	5.10	5.10	5.10
N. of Venturies		1	7	68	265	529	1058
P(pump)	kW	1.30	9.79	97.87	244.68	489.36	978.73
Energy consumption	kWh	0.43	3.26	32.62	81.56	163.12	326.24
Energy cost	€/kWh	0.12	0.12	0.12	0.12	0.12	0.12
Energy cpb***	€/batch	0.05	0.39	3.93	9.83	19.66	39.31
H ₂ SO ₄ (98%)	kg	30.00	2.94	2.94	2.94	2.94	2.94
H ₂ SO ₄ cost	€/kg		0.20	0.20	0.20	0.20	0.20
H ₂ SO ₄ cpb	€/batch	0.66	0.59	0.59	0.59	0.59	0.59
Lime Ca(OH) ₂	kg	0.01	3.70	37.00	92.50	185.00	370.00
Lime cost	€/kg	0.10	0.10	0.10	0.10	0.10	0.10
Lime cpb	€/batch	0.00	0.37	3.70	9.25	18.50	37.00
H ₂ O ₂ (30%)	kg	5.15	3.68	36.79	91.96	183.93	367.86
H ₂ O ₂ cost	€/kg	46.70	0.32	0.32	0.32	0.32	0.32
H ₂ O ₂ cpb	€/batch	0.24	1.18	11.77	29.43	58.86	117.71
PS	kg	11.00	7.86	78.57	196.43	392.86	785.71
PS cost	€/kg	89.80	1.44	1.44	1.44	1.44	1.44
PS CPB	€/batch	0.99	11.31	113.14	282.86	565.71	1131.43
Batch cost (without oxidants)	€/batch	0.71	1.35	8.22	19.67	38.74	76.90
Batch cost with H ₂ O ₂	€/batch	0.95	2.53	19.99	49.09	97.60	194.61
Batch cost with PS	€/batch	1.70	12.66	121.36	302.52	604.46	1208.33

*Q_r: total flow of the system**Q_v: the flow through each Venturi tube

***cpb: cost per batch

5.4 Conclusions

In this work, the design of a Venturi tube favoring the formation of nanobubbles was carried out, and the optimal configuration was used to build a hydrodynamic cavitation system used to degrade humic acid and two azo-dyes. In particular, nanobubbles' Z-potential was considered for the first time in the optimization process, providing insights into the relationship between the Venturi tube's geometry and nanobubbles' characteristics. The study identified the optimal Venturi tube design through the use of Design of Experiment (DoE), with the following parameters: i) $D_t=1\text{mm}$, ii) $L_1=9.85\text{mm}$, iii) $L_2=67.15\text{mm}$, and iv) $\alpha=1.96^\circ$. This configuration produced nanobubbles with an average diameter of 151 nm, a frequency of 89%, and a Z-potential of -15.4mV.

A neural network was developed to explore further Venturi tube configurations that were beyond our manufacturing capability. However, model testing highlighted the need for more experimental data with additional prototypes to train the model better and obtain more reliable predictions, especially regarding nanobubble diameter.

The developed HC system was tested for the degradation of three humic acids (HA), Reactive Red 120 (RR120), and Congo Red (CR)) in the presence of both additional oxidants (i.e., hydrogen peroxide (H_2O_2) and persulfate (PS)) and salt. In detail, the best results were obtained in acidic conditions ($\text{pH}=2$) using PS (for HA and CR) and H_2O_2 (for RR120), while the presence of salt markedly decreased the degradation of all studied chemicals. This study also investigated degradation kinetics, providing fundamental information to more efficiently manage treatment time, especially in the case of future scale-up efforts.

To the authors' best knowledge, this is the first work to investigate the efficiency of a nanobubble-enhanced HC system in the degradation of HA, RR120, and CR at the pilot scale (i.e., 14L) while considering the simultaneous effect of additional oxidants and salt.

6. Chapter 6: Developing and testing of a pilot scale plant for ammonia and PFAS removal from landfill leachate

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6.1 Introduction

As reported in Chapter 1.4.1, landfill leachate is a significant source of environmental emission of compounds that can pose a risk to aquatic environments and drinking water sources, particularly regarding perfluoroalkyl substances (PFAS) [491,492].

Although there are no specific regulations for PFAS concentrations in landfill leachate, regulatory limits regarding water resources that could be negatively affected by landfill leachate contamination are very strict. For example, according to the European Parliament Directive 2020/2184 [493], the sum of PFAS concentrations for 20 specific PFAS in drinking water must not exceed 0.1 µg/l.

This necessitates the use of suitable treatments for leachate, for which multiple treatment techniques are employed to meet legal requirements regarding the disposal of this complex mixture of organic and inorganic compounds [494]. In detail, the best practice is to construct the landfills in remote locations far from urban areas and more importantly from water resources [495]. However, the leachate may seep out of the landfill and contaminate groundwater or surface waters due to the following reasons i) inadequate liner system, ii) excessive leachate generation, iii) clogged collection system, iv) permeable geological condition such as fractured bedrock or permeable soil, and v) aging infrastructure [496,497].

Many different combinations of methods are available, including biological treatment, chemical oxidation, adsorption, precipitation, evaporation, drying, stripping, ultrafiltration (UF), nanofiltration (NF), and reverse osmosis (RO) [498]. If membrane filtration processes are used, it is also crucial to treat the concentrated fraction where most pollutants are present/accumulated, which may require specific and more aggressive treatments that usually lead to the production of contaminated sludge. Disposing of such sludges creates long-term problems, such as groundwater contamination, but various processes can be used to obtain reusable materials from these wastes (e.g., solidification/stabilization and chemical precipitation). Along with a series of treatments for the concentrated effluent to meet discharge criteria (e.g., Fenton, granulation) [499].

Among the treatment methods, nanofiltration (NF) has emerged as a potential process for treating landfill leachate. Membrane processes play a crucial role in water and wastewater treatment due to their adaptability to various contaminants and plant configurations [500,501]. However, if both organic and inorganic substances contaminate the landfill leachate, a more complex combination of treatment technologies is necessary to meet all the criteria for discharging landfill leachate. However, if nitrogenous compounds (e.g., ammonia) are present, it is necessary to incorporate additional procedures (e.g., biological oxidation treatment) to remove nitrogenous compounds and, depending on specific regulatory criteria, other monovalent salts. Particularly, due to the low effectiveness of NF for nitrogenous compounds, a second biological oxidation treatment may also be required [502–

504]. The total phosphorous levels (P) in the initial leachate was low (i.e., 12.7 mg/L), therefore it was not necessary to add additional components in the biological treatment to also address the phosphorous.

Given this context, this chapter reports the manuscript “Developing and testing of a pilot scale plant for ammonia and PFAS removal from landfill leachate”, describing the development of a landfill leachate treatment system. In particular, the process considers the removal of i) high levels of ammoniacal nitrogen, ii) PFAS, and iii) other conventional landfill leachate components (COD, BOD, heavy metals). Furthermore, a dedicated treatment of the concentrate fractions resulting from membrane filtration processes was developed.

6.2 Experimental

6.2.1 Leachate characterization

The leachate used in this study was collected from the Bossarino plant and the Ca' Felissine landfill (Verona, Italy). In detail, it is a non-hazardous landfill that was constructed in 2009. The landfill has remained inactive for 10 years. Even though it is a landfill for non-hazardous waste the leachate was tested to be PFAS-contaminated, necessitating a remediation plan. Moreover, the leachate composition was reported in Table 6-1. The initial volume of leachate was ca.1.3m³ and in order to obtain the necessary amount of granules (i.e., 1kg). In detail, 12 treatment cycles were concluded, resulting in 120L of treated leachate. Moreover, from the obtained granules, 100g was used for one leaching test, done in triplicate for reproducibility, resulting in 300g used for the leaching test. In detail, its composition is reported in. The results showed the presence of common long-chain PFAS (e.g., PFOA and PFOS), legacy PFAS (e.g., PFHxS and PFHxA), PFAS precursors (e.g., 8:2 FTS and 6:2 FTA), high levels of COD and ammoniacal nitrogen (i.e., ca.2000 and 1600mg/L respectively), and metals (e.g., Calcium, Zinc, and Copper).

Table 6-1: Leachate characteristics used in this study

Parameters	U. M.	Leachate
Perfluorooctanoic Compounds		
PFOS (Perfluorooctane Sulfonate)	ng/L	3500
PFOA (PerfluoroOctanoic Acid)	ng/L	19000
PFBA (PerfluoroButyric Acid)	ng/L	3800
PFBS (PerfluoroButane Sulfonate)	ng/L	6000
PFAS		
PFDeA (PerfluoroDecanoic Acid)	ng/L	< 10
PFDoA (PerfluoroDodecanoic Acid)	ng/L	< 10
PFHpA (PerfluoroHeptanoic Acid)	ng/L	1280
PFHxA (PerfluoroHexanoic Acid)	ng/L	1970
PFHxS (PerfluoroHexane Sulfonate)	ng/L	800
PFNA (PerfluoroNonanoic Acid)	ng/L	< 10
PFPeA (PerfluoroPentanoic Acid)	ng/L	820

PFUnA (PerfluoroUndecanoic Acid)	ng/L	< 10
Total PFAS	ng/L	37000
Total PFOA e PFOS e rispettivi derivati	ng/L	22500
Total PFAS esclusi PFOA, PFOS, PFBA e PFBS	ng/L	4900
Total PFAS esclusi PFOA e PFOS	ng/L	14600
Gen X (Perfluoro-2metil-3oxahexanoic acid)	ng/L	< 100
C ₆ O ₄ [CAS 1190931-41-9]	ng/L	< 100
Fluorotelomers		
4:2 - FTS	ng/L	< 10
6:2 - FTS	ng/L	< 10
8:2 - FTS	ng/L	< 10
10:2 - FTS	ng/L	< 100
6:2 - FTA	ng/L	< 100
8:2 - FTA	ng/L	< 100
10:2 - FTA	ng/L	< 100
Biological Oxygen Demand (BOD ₅)	mg/L of O ₂	338
Chemical Oxygen Demand (COD)	mg/L of O ₂	2040
Total Suspended Solids (TSS)	mg/L	201
Total Phosphorous (P)	mg/L	12.7
Ammoniacal nitrogen (ammonium ion)	mg/L	1580
Nitrous nitrogen (as N)	mg/L	< 0.02
Nitric nitrogen (as N)	mg/L	< 0.1
Total Nitrogen (as N)	mg/L	2240
Fluorides (fluoride ion)	mg/L	< 0.1
Chlorides (chloride ion)	mg/L	1350
Sulfates (sulfate ion)	mg/L	102
Total cyanides (cyanide ion)	mg/L	< 0.02
Metals		
Arsenic	mg/L	0.0500
Cadmium	mg/L	0.0100
Chromium	mg/L	0.424
Hexavalent Chromium	mg/L	< 0.01
Mercury	mg/L	< 0.0005
Nichel	mg/L	0.171
Lead	mg/L	< 0.01
Copper	mg/L	0.00600
Zinc	mg/L	0.0500
Iron	mg/L	2.60
Manganese	mg/L	0.0450
Calcium	mg/L	46.0
Magnesium	mg/L	61.0

6.2.2 Leachate treatment process and economic evaluation

In order to address each contaminant, the treatment procedure (Figure 6.1) described in the following paragraphs was employed. The system was fed from the pool of leachate that gets collected and transported to the treatment plant. Then the pumps are used to feed the system with a volume of 10L per day. Therefore, the system configuration can be considered as continuous. Moreover, it should be

noted that the landfill has not been operational for 10 years and has been capped and covered. Therefore, it is not actively producing leachate.

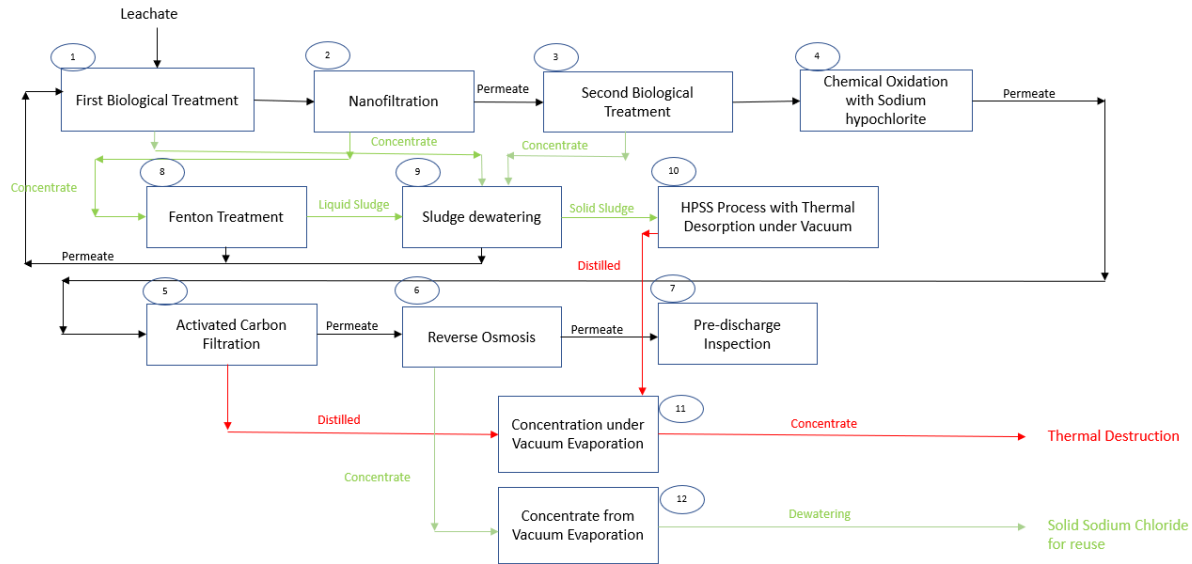


Figure 6.1: Multi-stage leachate treatment process schematics

Moreover table Table 6-2, shows the operational costs of the T.E.V. functioning treatment plant. This treatment plant was designed to treat 40,000m³ of landfill leachate in year with a total PFAS concentration of 65,000 ng/L, with the objective to decrease the final effluent concentration of PFAS to less than 300 ng/L. Moreover, as shown in Table 6-2, the highest annual cost is attributed to the energy consumption (i.e., 262,800.00) of the system and the highest cost per treated cubic meter to operators (labor) (i.e., 12 €/m³). In detail these two factors together account for ca. 78% of the total operation cost (i.e., €23.57/m³), indicating that the treatment process is both energy- and manpower-intensive. Therefore, automation of various treatment units could result in lower labour costs and renewable energy sources such as solar panels may be used to reduce the high electricity costs. However, it should be noted that the cost analysis is for treating industrial landfill leachate with high concentrations of PFAS, and the costs to ensure the PFAS will not enter the environment are justified.

Table 6-2: Economic evaluation of the landfill leachate treatment plant

Cost Item	Unit Cost	Annual Cost	€/m ³
Depreciation over 5 years		€ 80,000.00	2.00
Electricity	€ 0.20/kWh	€ 262,800.00	6.57
Reagents		€ 96,000.00	2.40
Labor (2 operators)	€ 9,600.00/month	€ 115,200.00	12.00
Rentals	€ 2,000.00/month	€ 24,000.00	0.60
Total			€ 23.57/m³

6.2.2.1 *First biological treatment*

The treatment plant utilized the patented MMF (Membrane Bio-System Mix Flow) technology developed by TEV GROUP SRL (patent no.10201600001818), an advanced version of the MBR (Membrane Bioreactor). This system employs tubular side-stream membranes with a porosity of 0.1 μm for ultrafiltration, reducing bacterial load and preventing the escape of suspended solids. The side-stream configuration keeps the membranes external to the oxidation tank, with activated sludge recirculated through a 0.6 mm automatic filter to prevent clogging. Pressurized air injection (2bar) enhances oxygenation, eliminating the need for conventional aeration systems.

The system consisted of two interconnected tanks, each with a volume of approximately 300L. The first tank was dedicated to the oxidation process, while the second tank was designed for denitrification. After filtration, the leachate was pumped into the first tank at a flow rate of 10L/day and a mean residence time of 10 days for the removal of biodegradable organic substances. It was then continuously introduced (10L/day) into the second denitrification tank, where nitrogen was removed via nitrification and denitrification. During the denitrification phase, an external carbon source (glucose) was added to convert nitrates into N_2 and CO_2 .

Both systems had a volume of about 300 liters (diameter = 0.7m, height = 0.8m) and operated at a temperature of 22–23°C, maintained using a water heat exchanger. The pH of the tanks during the treatment process was maintained at an average of around 9. Dissolved oxygen (DO) was supplied via a blower and kept at approximately 5mg/L. A sludge transfer pump was employed to maintain a constant hydraulic retention time (HRT) of 15 minutes.



Figure 6.2: Nitrification (a) and denitrification (b) tanks.

6.2.2.2 *Nanofiltration*

Nanofiltration was tested using two different membranes: one with a molecular weight cutoff of 270Da (FilmTec™ NF270-440 Element, 41m² filtration surface area) and another with a cutoff of 400Da (NX Filtration MP025 dNF40, 0.05m² filtration surface area). These membranes were employed to remove toxic substances from the leachate, including non-biodegradable organic compounds, PFAS, phenols, hydrocarbons, heavy metals, high-molecular-weight saline substances,

and divalent ions. However, this stage allows sodium chloride and ammoniacal nitrogen permeation and is ineffective for short-chain PFAS.

Two streams were obtained: a concentrate, representing about 7-10% of the influent, which was sent for physicochemical treatment, and a permeate, which was directed to the denitrification phase of the second biological process. The test with the NX Filtration MP025 dNF40 membrane was performed using a bench-scale system that treated 30L of permeate extracted from the UF outlet of the first biological treatment, while the FilmTec™ NF270-440 Element membrane was tested using a pilot-scale system that treated 120L.

The liquid was recirculated through the nanofiltration system at 5L/min and 6bar for the NX Filtration MP025 dNF40 membrane, while a flow of 15L/min at 31bar was used for the FilmTec™ NF270-440 Element membrane. The pressure drop for the NF membrane was 9bar and during both experiments, the pH of the treated liquid was maintained at 6.8.

Moreover, in order to manage calcium carbonate fouling, the membranes were cleaned daily with a diluted HCl solution, which converted the carbonate into CO₂ and removed other metal precipitates and mineral scales. Additionally, the membranes were washed every two days with a 1-1.5% aqueous solution of tetrasodium EDTA, a highly effective chelating agent for removing organic matter and silt. This combination of cleaning methods ensured optimal membrane performance and minimized clogging risks during the tests [505,506] due to the fact that a simple hydraulic backwash is only able to remove high molecular weight organic compounds, thus not providing sufficient fouling prevention for the membrane [507]. It is worth mentioning that this cleaning procedure was not intended to be used on a larger scale and was implemented in the current treatment plant to minimize membrane fouling and maximize its lifetime, thus preventing additional costs in the future.



Figure 6.3: Nanofiltration using the membrane NF270

6.2.2.3 Second biological treatment and oxidation with sodium hypochlorite

The second biological treatment, following nanofiltration, employs a single-stage nitrification and denitrification process to remove residual ammonia. A side-stream MMF (Multi-Media Filtration) system was used, and advanced removal ensures compliance with an ammoniacal nitrogen limit of 0.5mg/L. Sodium hypochlorite may be added to the permeate from this process to oxidize any residual ammonia.



Figure 6.4: Second biological treatment

6.2.2.4 Activated carbon filtration

The permeate containing monovalent salts and low molecular weight contaminants not removed by the NF process was treated in an activated carbon filtration setup consisting of two interconnected tubes. The first was filled with Micropore CARBOSORBNC1240® and the second with CARBOSORBNC1250®. The experimental setup used two interconnected HDPE columns, each with a diameter of 2cm and a height of 30cm. The effluent exiting the first column, filled with CARBOSORBNC1240®, flowed directly into the second column containing CARBOSORBNC1250®. Also, to ensure a constant and controlled process, the inlet was supplied drop by drop using a peristaltic pump, maintaining a flow of 1L per hour. The temperature was maintained at $25 \pm 2^\circ\text{C}$ throughout the process, and no changes in pH were reported. The calculated residence time in the system was 4.5 hours, which allowed sufficient contact time for effective adsorption of contaminants through both columns [508].

6.2.2.5 Reverse osmosis

The reverse osmosis phase was tested using a FilmTec™ SW30-2514 membrane in the same apparatus used for the FilmTec™ NF270-440 Element membrane, with a pressure of 6.5bar. The process was used to remove the remaining salts and any residual PFAS (e.g., Gen-X and other short-chain PFAS). The test was conducted at a temperature of 25°C . Samples of permeate and concentrate were collected. After completing each test, the membrane was washed with 1% EDTA solution (pH=10) and NaOH solution (pH=11) solutions and rinsed with deionized water [509]. The concentrate was vacuum-concentrated to recover the salt [510].



Figure 6.5: Reverse osmosis membrane

6.2.2.6 *Fenton treatment of the concentrated part of the nanofiltration*

The concentrate obtained from nanofiltration with the FilmTec™ NF270-440 Element underwent a Fenton treatment to convert non-biodegradable organic substances into biodegradable molecules and precipitate dissolved metals. This process is based on generating $\text{OH}^\cdot$ radicals from the catalytic decomposition of hydrogen peroxide (H_2O_2) by iron in an acidic medium ($\text{pH} \approx 3$).

Hydrochloric acid (HCl, 25%), hydrogen peroxide (H_2O_2 , 20%), and ferrous chlorate ($\text{Fe}(\text{ClO}_3)_2$) were used to treat 30 liters of nanofiltration concentrate collected in an HDPE barrel. The process was conducted as follows: (i) the initial pH of the concentrate was measured, showing a value of 8.8; (ii) 2.5L of ferrous chlorate was added, bringing the pH to 6.7; (iii) then 2L of hydrochloric acid was added, further lowering the pH to 2.9; (iv) 6L of hydrogen peroxide was introduced, reducing the pH to 2.6; (v) finally, 1.8L of sodium hydroxide (NaOH, 20%) was added to neutralize the solution and bring the pH back to 8. In this case, the cost of bringing the pH back to 8 is justified since the pH should be brought back up in order to precipitate the metals as hydroxides. In detail, $\text{Fe}(\text{ClO}_3)_2$ was used specifically to act as a source of Fe and to provide it for the Fenton process, which was coagulated when the pH was brought back up to 8, thus facilitating the treatment process. The reason chloride was used instead of sulphate is due to the fact that chloride can be recovered as salt and is therefore economically efficient while sulphate cannot. Moreover, the liquid part of the Fenton process is always re-introduced inside the system as shown in Figure 6.1.

Throughout the process, the solution was continuously stirred using a mechanical mixer. An antifoaming agent (i.e., Glensol 5919) was also added at 0.25% v/v during reagent dosing to control foam development. After the mixing phase, the solution was allowed to rest for 24 hours to allow the sludges to settle. Samples were taken from the inlet, the clarified solution, and the sludge from the bottom of the tank for analysis. The sludges were sent to a filter press for dehydration and granulation, while the clarified solution was reintroduced into the initial biological oxidation process [511,512].



Figure 6.6: Fenton process agitation phase (a) and precipitation phase (b)

6.2.2.7 Fenton sludge granulation and thermal desorption

The solids obtained from the dehydration section are granulated using the HPSS process (the granulate contains 27% Portland Cement 52.5, 73% sludge, and additives MapeplastECO1A and MapeplastECO1B equal to 2% of the cement weight). The granular material is subjected to a vacuum thermal desorption process to remove PFAS, and the distilled PFAS solution is sent for further concentration and thermal destruction. Specifically, the thermal desorption process was carried out by heating the granulate to 230°C (temperature reached in 3 hours and maintained for 1 hour to ensure uniform heating of the granules). Subsequently, a vacuum treatment phase was applied for 35 minutes, during which it took 20 minutes to reach a pressure of 40mmHg, then maintained for another 15 minutes.

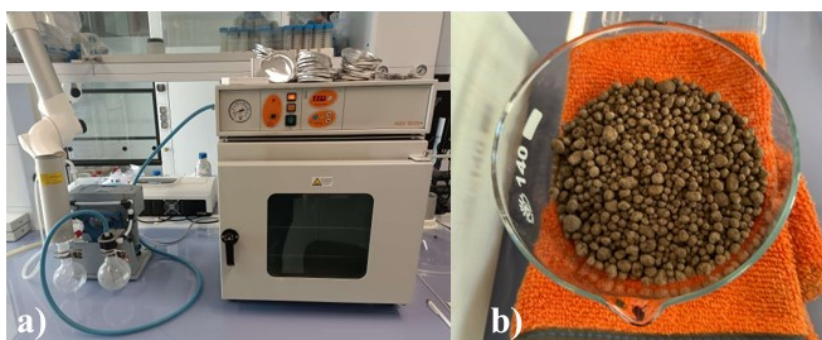


Figure 6.7: Apparatus used for the thermal desorption test (a) and treated granules (b)

Leaching tests on the granulate after the thermal desorption process were carried out according to the UNI EN 12457-2:2004 standard, as required by Italian regulations on reusable materials [513]. Specifically, the samples were placed in an HDPE bottle with MilliQ water (solid/liquid ratio=1/10) and shaken with an end-over-end tumbler (10rpm) for 24 hours, and then the resulting eluate was filtered at 0.45μm and analyzed.

6.2.2.8 Reverse osmosis concentrates under vacuum

The concentrate from the reverse osmosis phase is vacuum concentrated to recover the salt present, heating the solution to about 50°C after reaching a pressure of about 70mmHg.

6.3 Results and discussion

6.3.1 Biological treatment

The analyses conducted for each biological treatment (Table 6-3 and Table 6-4) included: i) Chemical Oxygen Demand (COD) levels (mg/L), ii) dissolved oxygen (DO) levels in the oxidation tanks (mg/L), iii) ammonia levels at the outlet of both ultrafiltration units (mg/L), iv) nitrate and nitrite levels, v) temperature, and vi) sludge content.

Table 6-3: Monitoring and performance of the first biological treatment (average values)

Parameter	U.M.	Influent	Effluent UF
COD	mg/L	2040	3113
Ammonia	mg/L	1540	40.3
Nitrates (NO ₃)	mg/L	<3	<3
Nitrites (NO ₂)	mg/L	<0.5	<0.5

The results (Table 6-3) showed that the first biological treatment removed most of the ammonia present in the leachate, while the remaining nitrogen was removed by the second biological treatment (Table 6-4).

The temperature of both biological treatments remained between 25 and 32°C, favoring the presence of mesophilic bacteria, which have shown good performance in degrading organic matter and transforming ammonia into nitrates and then nitrogen [514]. The dissolved oxygen (DO) levels were also always maintained at suitable levels in the first oxidation tank and during the aerobic phase of the second biological treatment [515] with values of ca. 5.2mg/L and 3.6mg/L, respectively. The volatile substance content of the sludge in both treatments (i.e., 42 and 40%d.w., respectively) also indicates a healthy bacterial population.

The permeate from the first biological treatment was treated via NF and then subjected to the second biological oxidation treatment. The results are shown in Table 6-4, highlighting the near-complete removal of ammoniacal nitrogen and a drastic reduction in organic matter.

Table 6-4:Monitoring and performance of the second biological treatment (average value)

Parameter	U.M.	Influent	Effluent
COD	mg/L	1400	670
Ammonia	mg/L	27.3	1.5
Nitrates (NO ₃)	mg/L	<3	<3
Nitrites (NO ₂)	mg/L	<0.5	<0.5

Furthermore, as shown in Table 6-5, the residual ammonia present after the second biological treatment was successfully removed in the oxidation process with sodium hypochlorite, reaching a level below 0.5mg/L in line with the process objectives.

Table 6-5: Performance of oxidation with sodium hypochlorite

Parameter	U.M.	Influent	Effluent
Ammonia	mg/L	1.5	<0.5
Nitrates (NO ₃)	mg/L	<3	<3
Nitrites (NO ₂)	mg/L	<0.5	<0.5

6.3.2 Nanofiltration

The analysis was conducted on samples obtained using NF400 and NF270 membranes, and the results are shown in Table 6-6, Table 6-7, and Figure 6.8. Good removal was obtained for both NF400 and NF270 in the case of long-chain PFAS (e.g., N-MeFOSAA, N-EtFOSAA, PFUnDA, PFDoDA, PFDS, FOSA, 11Cl-PF3OUdS, and PFOA) while short-chain PFAS were removed only using NF270 (e.g., PFBA, PFHxA, PFHpA). In detail, PFAS removal ranged between 65-90% for all compounds in the case of NF270, while the efficiency of the process was significantly lower for short-chain PFAS (i.e., 5–10%) when using NF400 since these PFAS have a molecular weight close to the cut-off of the NF400 membrane [516].

Regarding metals' removal in the NF270 treatment, copper, nickel, and chromium had the highest removal percentages. However, significant concentrations of copper, magnesium, zinc, and calcium were still detected in the permeate. Filtration with NF400 showed similar results but with lower efficiency, particularly for copper (36% removal) and magnesium (20%), while it performed more effectively in separating iron (92%) and chromium (79%). In the concentrate from both NF trials, PFAS concentrations ranged between 1000 and 10000ng/L, with notable values for PFHxA (1.617ng/L) and PFUnDA (7.871ng/L) in the case of NF270 and for PFPeA (116ng/L) and PFBS (2.624ng/L) in the case of NF400.

Based on the type of the NF membrane used (i.e., NF90, 270, 400), not all the contaminants can be efficiently stopped, as the experimental results showed for the short-chain PFAS. Moreover, the economic feasibility of the project is contingent upon salt production. Therefore, nano membranes

NF400 and NF270 were selected over NF90 since this membrane would have stopped so much salt. Meanwhile, in order to address the problem of remaining low-molecular-weight contaminants in the next treatment stages, an additional activated carbon filtration was included inside the treatment train, to ensure both the quality of salt (deprived of any PFAS or metals) and to prevent fouling of the subsequent RO process.

Table 6-6: Concentration of PFAS and metals in the feed, permeate, and concentrate during the NF filtration with the NF270 membrane

Parameter	U.M.	Influent	Effluent	Concentrate	Removal %
PFAS					
6-2 FTS	ng/L	<1	<1	<1	-
8-2FTS	ng/L	<2.5	<2.5	<2.5	-
9Cl-PF3ONS	ng/L	<0.5	<0.5	<0.5	-
ADONA	ng/L	<0.5	<0.5	<0.5	-
FBSA	ng/L	<0.5	<0.5	<0.5	-
FHxSA	ng/L	<0.5	<0.5	<0.5	-
FOSA	ng/L	178	37	3976	80
HFPO-DA	ng/L	<0.5	<0.5	<0.5	-
N-EtFOSAA	ng/L	252	64	3288	75
PFBA	ng/L	1388	203	4574	85
PFBS	ng/L	1369	405	4765	70
PFDA	ng/L	91	18	1679	80
PFDODA	ng/L	44	8	1099	82
PFHPA	ng/L	61	14	567	77
PFHpS	ng/L	<0.5	<0.5	<0.5	-
PFHXA	ng/L	379	76	1617	80
PFHXS	ng/L	<0.5	<0.5	<0.5	-
PFNA	ng/L	142	17	3495	88
PFNS	ng/L	<0.5	<0.5	<0.5	-
PFOA	ng/L	365	45	4523	88
PFOS	ng/L	<0.5	<0.5	<0.5	-
PFPeA	ng/L	266	47	747	82
PFPeS	ng/L	<0.5	<0.5	<0.5	-
PFTriDA	ng/L	<10	<10	<10	-
PFunDA	ng/L	205	41	7871	80
Metals					
Arsenic	mg/L	0.060	0.030	0.080	50
Cadmium	mg/L	0.00200	0.00200	0.0090	0
Calcium	mg/L	41	20.0	80	51.2
Chromium	mg/L	0.151	<0.005	0.36	100
Iron	mg/L	0.69	0.103	2.1	85.1
Magnesium	mg/L	64	11.0	109	83
Manganese	mg/L	0.072	0.0200	0.110	72.2
Nickel	mg/L	0.42	0.0130	0.97	97
Lead	mg/L	0.040	0.0100	0.040	75
Copper	mg/L	56	4.4	91	92.1
Zinc	mg/L	7.5	1.35	7.6	82

Table 6-7: Concentration of PFAS and metals in the feed, permeate, and concentrate during the NF filtration with the NF400.

Parameter	U.M.	Influent	Effluent	Concentrate	Removal %
PFAS					
6-2 FTS	ng/L	<1	<1	<1	-
8-2FTS	ng/L	<2.5	<2.5	<2.5	-
9Cl-PF3ONS	ng/L	<0.5	<0.5	<0.5	-
ADONA	ng/L	<0.5	<0.5	<0.5	-
FBSA	ng/L	<0.5	<0.5	<0.5	-
FHxSA	ng/L	<0.5	<0.5	<0.5	-
FOSA	ng/L	169	32	1777	81
HFPO-DA	ng/L	<0.5	<0.5	<0.5	-
N-EtFOSAA	ng/L	181	60	3141	66
PFBA	ng/L	1421	1350	2060	<5%
PFBS	ng/L	1404	1333.8	2035	<5%
PFDA	ng/L	117	20	977	82
PFDoDA	ng/L	47	10	181	79
PFHPA	ng/L	120	115	430	4.2
PFHpS	ng/L	<0.5	<0.5	<0.5	-
PFHXA	ng/L	535	530	1059	0.9
PFHXS	ng/L	<0.5	<0.5	<0.5	-
PFNA	ng/L	357	160	2369	55
PFNS	ng/L	<0.5	<0.5	<0.5	-
PFOA	ng/L	786	746.7	1139.7	<5%
PFOS	ng/L	<0.5	<0.5	<0.5	-
PFPeA	ng/L	232	220.4	336.4	<5%
PFPeS	ng/L	<0.5	<0.5	<0.5	-
PFTriDA	ng/L	<10	<10	<10	-
PFunDA	ng/L	397	36	2091	91
Metals					
Arsenic	mg/L	0.050	0.0200	0.100	60
Cadmium	mg/L	0.00200	0.00100	0.0060	50
Calcium	mg/L	45	22	125	51
Chromium	mg/L	0.137	0.029	0.77	79
Iron	mg/L	0.94	0.071	5.5	92
Magnesium	mg/L	55	44	119	20
Manganese	mg/L	0.110	0.052	0.41	53
Nickel	mg/L	0.35	0.164	1.60	53
Lead	mg/L	0.030	0.0100	0.170	66.7
Copper	mg/L	25	16.0	96	36
Zinc	mg/L	4.0	1.36	18.2	66

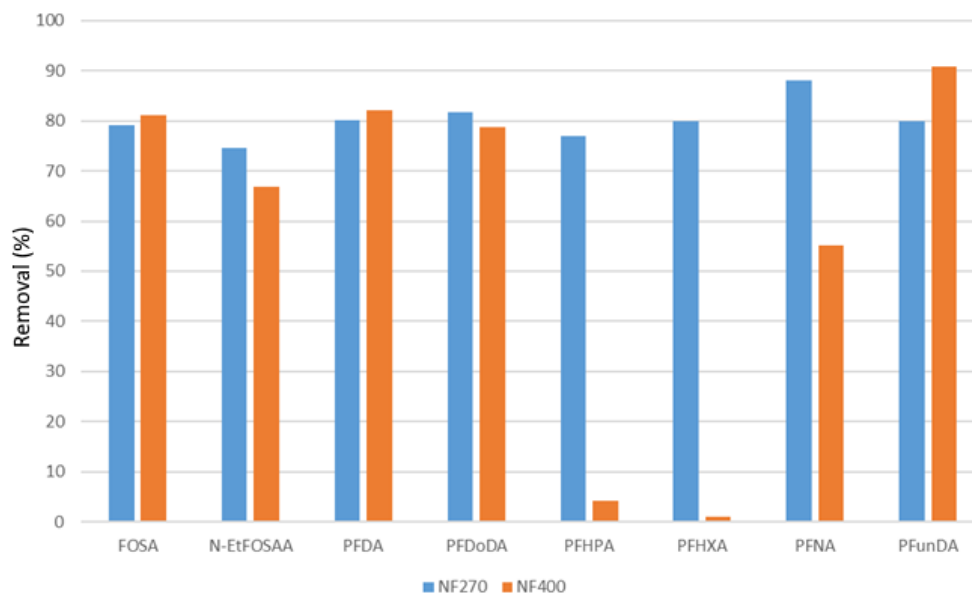


Figure 6.8: Comparison of removal percentage between NF270 and NF400

6.3.3 Activated carbon filtration

Activated carbon filtration was necessary to remove PFAS that were not retained by the nanofiltration process, as well as substances that could lead to clogging of the subsequent reverse osmosis membrane. Furthermore, it was necessary to stop all the PFAS that were not remaining from previous stages in order to ensure they will not appear in the salt, yield from the RO filtration. The results are reported in Table 6-8, showing that the process completely removed all residual PFAS that were not eliminated by the NF270 process, ensuring the quality of the salt obtained from the next reverse osmosis process. Moreover, concentrations of copper (0.108mg/L), calcium (14.3mg/L), and magnesium (32mg/L) were detected, but at levels that do not exceed the total limits established by regulations. Iron was removed with an efficiency of 96%, while only 3.7% of Nickel was removed.

Table 6-8: Concentration of PFAS and metals in the influent and effluent of the activated carbon filtration process.

Parameter	U.M.	Influent	Effluent	Removal
PFAS				
6-2 FTS	ng/L	2.34	<1	100
8-2FTS	ng/L	<2.5	<2.5	100
9Cl-PF3ONS	ng/L	<0.5	<0.5	100
ADONA	ng/L	<0.5	<0.5	100
FBSA	ng/L	7.04	<0.5	100
FHxSA	ng/L	<0.5	<0.5	100
FOSA	ng/L	<0.5	<0.5	100
HFPO-DA	ng/L	<0.5	<0.5	100
N-EtFOSAA	ng/L	<25	<25	100
PFBA	ng/L	760	<0.5	100
PFBS	ng/L	1260	<1	100
PFDA	ng/L	<2.5	<25	100
PFDoDA	ng/L	<0.5	<0.5	100
PFHPA	ng/L	31.8	<1	100
PFHpS	ng/L	<2.5	<2.5	100
PFHXA	ng/L	208	<5	100
PFHXS	ng/L	3.8	<0.5	100
PFNA	ng/L	66.6	<0.5	100
PFNS	ng/L	<0.5	<0.5	100
PFOA	ng/L	113.4	<0.5	100
PFOS	ng/L	2.94	<1	100
PFPeA	ng/L	134	<0.5	100
PFPeS	ng/L	3.28	<0.5	100
PFTriDA	ng/L	<0.5	<0.5	100
PFunDA	ng/L	25.8	<0.5	100
Metals				
Arsenic	mg/L	<0.01	<0.01	0
Cadmium	mg/L	<0.001	<0.001	0
Calcium	mg/L	14.0	12.0	14.3
Chromium	mg/L	0.0070	<0.005	28.6
Iron	mg/L	2.9	0.126	96
Magnesium	mg/L	23	32	-
Manganese	mg/L	0.0180	0.0050	72
Nickel	mg/L	0.053	0.051	3.7
Lead	mg/L	0.030	<0.01	66.7
Copper	mg/L	6.3	0.108	98
Zinc	mg/L	0.76	0.029	96

6.3.4 Reverse Osmosis filtration and salt recovery.

The reverse osmosis process was used to recover the salts still present in the effluent after the activated carbon filtration and to ensure the removal of other organic and inorganic compounds from the final effluent. The results shown in Table 6-9 indicate that all PFAS were successfully removed from the system, while for the remaining metals, some residual concentration was found in the permeate despite the high removal efficiency (i.e., between 70-100%).

The reverse osmosis concentrate was concentrated under vacuum, achieving a salt yield (primarily sodium chloride) of approximately 25g/L. In detail, the metal content of the salt (Table 6-10) was with regulatory limits for its use as road salt [517], thus contributing to the economic feasibility of

this process. However, in the case of possible contaminations from other process steps, an additional activated carbon filtration step before concentration should be considered.

Table 6-9: Concentration of PFAS and metals in the influent, permeate, and concentrate during the RO process.

Parameter	U.M.	Influent	Permeate	Concentrate	Removal
PFAS					
6-2 FTS	ng/L	<1	<1	<1	-
8-2FTS	ng/L	<2.5	<2.5	<2.5	-
9Cl-PF3ONS	ng/L	<0.5	<0.5	<0.5	-
ADONA	ng/L	<0.5	<0.5	<0.5	-
FBSA	ng/L	<0.5	<0.5	<0.5	-
FHxSA	ng/L	<0.5	<0.5	<0.5	-
FOSA	ng/L	<0.5	<0.5	<0.5	-
HFPO-DA	ng/L	<0.5	<0.5	<0.5	-
N-EtFOSAA	ng/L	<25	<25	<25	-
PFBA	ng/L	<0.5	<0.5	<0.5	-
PFBS	ng/L	<1	<1	<1	-
PFDA	ng/L	<25	<25	<25	-
PFDODA	ng/L	<0.5	<0.5	<0.5	-
PFHPA	ng/L	<1	<1	<1	-
PFHpS	ng/L	<2.5	<2.5	<2.5	-
PFHXA	ng/L	<5	<5	<5	-
PFHXS	ng/L	<0.5	<0.5	<0.5	-
PFNA	ng/L	<0.5	<0.5	<0.5	-
PFNS	ng/L	<0.5	<0.5	<0.5	-
PFOA	ng/L	<0.5	<0.5	<0.5	-
PFOS	ng/L	<1	<1	<1	-
PFPeA	ng/L	<0.5	<0.5	<0.5	-
PFPeS	ng/L	<0.5	<0.5	<0.5	-
PFTriDA	ng/L	<0.5	<0.5	<0.5	-
PFunDA	ng/L	<0.5	<0.5	<0.5	-
Metals					
Arsenic	mg/L	0.040	< 0.01	<0.01	75
Cadmium	mg/L	< 0.001	< 0.001	<0.001	-
Calcium	mg/L	13.0	1.00	9.0	92
Chromium	mg/L	< 0.005	< 0.005	<0.005	-
Iron	mg/L	0.32	0.041	0.54	87
Magnesium	mg/L	29	5.0	155	82
Manganese	mg/L	0.008	<0.005	0.017	100
Nickel	mg/L	0.069	0.0180	0.528	74
Lead	mg/L	<0.01	<0.01	<0.01	-
Copper	mg/L	0.36	0.039	1.72	89
Zinc	mg/L	0.40	0.031	0.44	92

Table 6-10: Concentration of metals in the salt obtained after vacuum evaporation of the concentrate from the reverse osmosis process.

Parameter	U.M.	Salt	Regulatory limit*
Arsenic	mg/Kg d.w.	0.04	2.5
Cadmium	mg/Kg d.w.	0.004	2
Calcium	mg/Kg d.w.	360	-
Chromium	mg/Kg d.w.	0.004	5
Iron	mg/Kg d.w.	21.6	-
Magnesium	mg/Kg d.w.	5160	-
Manganese	mg/Kg d.w.	0.68	-
Nickel	mg/Kg d.w.	3.2	5
Lead	mg/Kg d.w.	0.084	5
Copper	mg/Kg d.w.	3.8	5
Zinc	mg/Kg d.w.	17	20

*Requirements of EN 16811-1 standard for de-icing salt

6.3.5 Fenton process on nanofiltration concentrate

The concentrate resulting from the nanofiltration process with NF270 was treated with a Fenton oxidation process, and the results are reported in Table 6-11. The results show a complete removal of FhxSA, OFSA, PFDA, PFDODA, PFHXS, PFNA, PFOS, PFTriDA, and PFunDA, due to both their adsorption onto the sludge produced and their degradation, while an increase in the concentration of 6-2 FTS, FBSA, and PFBA was observed. This increase may be due to the degradation of more complex PFAS. The treatment also led to the precipitation and removal of arsenic (83%), chromium (90%), copper (87%), and zinc (99%) with high efficiency, while magnesium, nickel, and lead were removed with minimal efficiency of 2%, 33%, and 33%, respectively.

Table 6-11: Concentration of PFAS and metals in the influent and effluent of the Fenton process for the treatment of the concentrate from the NF270 process.

Parameter	U.M.	Influent	Effluent	Removal%
PFAS				
6-2 FTS	ng/L	23.2	27	
8-2FTS	ng/L	<2.5	<2.5	-
9Cl-PF3ONS	ng/L	<0.5	<0.5	-
ADONA	ng/L	<0.5	<0.5	-
FBSA	ng/L	24.2	45.4	
FHxSA	ng/L	2.94	<0.5	100
FOSA	ng/L	5.8	<1	100
HFPO-DA	ng/L	<0.5	<0.5	-
N-EtFOSAA	ng/L	<25	<25	-
PFBA	ng/L	5500	5920	
PFBS	ng/L	8700	6560	24.6
PFDA	ng/L	4.66	<2.5	100
PFDoDA	ng/L	<5	<5	100
PFHPA	ng/L	572	9.72	98.3
PFHpS	ng/L	<0.5	<0.5	-
PFHXA	ng/L	2460	182.2	92.6
PFHXS	ng/L	41.6	<0.5	100
PFNA	ng/L	82.8	<1	100
PFNS	ng/L	<0.5	<0.5	-
PFOA	ng/L	3940	113	97.1
PFOS	ng/L	6.3	<0.5	100
PFPeA	ng/L	1136	938	17.3
PFPeS	ng/L	20.2	1.944	90.4
PFTriDA	ng/L	26.2	<10	100
PFunDA	ng/L	11.68	<2.5	100
Metals				
Arsenic	mg/L	0.060	< 0.01	83
Cadmium	mg/L	< 0.001	< 0.001	0
Calcium	mg/L	63	41	35
Chromium	mg/L	0.26	0.026	90
Iron	mg/L	1.98	1.61	18.7
Magnesium	mg/L	97	95	2
Manganese	mg/L	0.105	1.20	-
Nickel	mg/L	0.61	0.41	33
Lead	mg/L	0.030	0.0200	33
Copper	mg/L	41	5.4	87
Zinc	mg/L	7.1	0.073	99



Figure 6.9: Fenton process dosage (a), influent, and clarified effluent of the Fenton process (b).

6.3.6 Granulation of sludge from the Fenton process and thermal desorption

The sludge obtained from the Fenton process contains a high concentration of PFAS (Table 6-12), making it unsuitable for reuse as an agricultural amendment or as an inert material for cementitious products. Therefore, the effectiveness of a vacuum treatment for removing these contaminants was investigated to obtain a material that does not pose risks to human health or the environment if reused. The results show that the PFAS concentration in the granulate remained consistently below the limit of quantification, both before and after the thermal treatment (TT) (Table 6-12). For this reason, it was not possible to estimate the process efficiency, but the presence of PFAS in both the condensates (Table 6-13) and the eluates from the leaching test (Table 6-14) indicates that these substances were removed from the granulate, though not to a level where they were undetectable in the leachate from the UNI 12457-2 test. Further analytical investigations will be necessary to determine the removal percentages of PFAS from the granulate during TT.

The results of the UNI EN 12457-2 leaching test show significant concentrations of 6-2 FTS (99ng/L), PFBA (173.6ng/L), and PFBS (17.2ng/L) in the leachate, along with metal concentrations (As, Cd, Pb, and Ni) slightly below the limits established by Italian regulations on reusable materials [518].

Table 6-12: Concentration of PFAS in granules before and after thermal desorption treatment.

Parameter	U.M.	Granules before thermal desorption	Granules after thermal desorption	Removal %
PFAS				
6-2 FTS	µg/Kg	< 1	< 1	--
8-2FTS	µg/Kg	< 1	< 1	--
9Cl-PF3ONS	µg/Kg	< 1	< 1	--
ADONA	µg/Kg	< 1	< 1	--
FBSA	µg/Kg	< 1	< 1	--
FHxSA	µg/Kg	< 1	< 1	--
FOSA	µg/Kg	< 1	< 1	--
HFPO-DA	µg/Kg	< 1	< 1	--
N-EtFOSAA	µg/Kg	< 1	< 1	--
PFBA	µg/Kg	< 1	< 1	--
PFBS	µg/Kg	< 1	< 1	--
PFDA	µg/Kg	< 1	< 1	--
PFDoDA	µg/Kg	< 1	< 1	--
PFHPA	µg/Kg	< 1	< 1	--
PFHpS	µg/Kg	< 1	< 1	--
PFHXA	µg/Kg	< 1	< 1	--
PFHXS	µg/Kg	< 1	< 1	--
PFNA	µg/Kg	< 1	< 1	--
PFNS	µg/Kg	< 1	< 1	--
PFOA	µg/Kg	< 1	< 1	--
PFOS	µg/Kg	< 1	< 1	--
PFPeA	µg/Kg	< 1	< 1	--
PFPeS	µg/Kg	< 1	< 1	--



Figure 6.10: Sludge resulting from the Fenton process after dehydration (a) and granulation (b).

Table 6-13: Concentration of PFAS in the condensate obtained during the TT process. Condensate 1 refers to the condensation system along the vacuum line, while Condensate 2 refers to the water condenser placed after the pump outlet.

Parameter	U.M.	Condensate 1	Condensate 2
PFAS			
6-2 FTS	ng/L	15.1	145
8-2FTS	ng/L	<2.5	<2.5
9Cl-PF3ONS	ng/L	1.0	<0.5
ADONA	ng/L	<0.5	71
FBSA	ng/L	1.6	<0.5
FHxSA	ng/L	22.6	<0.5
FOSA	ng/L	108	8.8
HFPO-DA	ng/L	<0.5	32
N-EtFOSAA	ng/L	2220	<25
PFBA	ng/L	538	31000
PFBS	ng/L	41.2	46
PFDA	ng/L	38.2	21.4
PFDoDA	ng/L	<5	14.44
PFHPA	ng/L	366	218
PFHpS	ng/L	41	<0.5
PFHXA	ng/L	364	228
PFHXS	ng/L	185	26.2
PFNA	ng/L	2840	242
PFNS	ng/L	<0.5	<0.5
PFOA	ng/L	2120	684
PFOS	ng/L	1300	27.2
PFPeA	ng/L	1028	6740
PFPeS	ng/L	32.2	<0.5
PFTriDA	ng/L	<10	22
PFunDA	ng/L	83.8	12.7

Table 6-14: Concentration of PFAS and metals in the leachate obtained from the UNI EN 12457-2 leaching test performed on the granules after thermal desorption treatment (TT).

Parameter	U.M.	Eluate of the granulate after thermal desorption treatment	Regulatory Limit*
6-2 FTS	ng/L	99	-
8-2FTS	ng/L	<2.5	-
9Cl-PF3ONS	ng/L	<0.5	-
ADONA	ng/L	<0.5	-
FBSA	ng/L	1.6	-
FHxSA	ng/L	<0.5	-
FOSA	ng/L	<1	-
HFPO-DA	ng/L	<0.5	-
N-EtFOSAA	ng/L	<25	-
PFBA	ng/L	173.6	-
PFBS	ng/L	17.2	-
PFDA	ng/L	<2.5	-
PFDoDA	ng/L	<5	-
PFHPA	ng/L	4.1	-
PFHpS	ng/L	<0.5	-
PFHXA	ng/L	4.3	-
PFHXS	ng/L	5.5	-
PFNA	ng/L	<1	-
PFNS	ng/L	<0.5	-
PFOA	ng/L	6	-
PFOS	ng/L	5.4	-
PFPeA	ng/L	5.3	-
PFPeS	ng/L	1.4	-
PFTriDA	ng/L	<10	-
PfunDA	ng/L	<2.5	-
Metals			
Arsenic	µg/L	40	50
Barium	µg/L	25	1000
Beryllium	µg/L	< 0.1	10
Cadmium	µg/L	1.0	5
Cobalt	µg/L	0.5	250
Chromium	µg/L	40	50
Nickel	µg/L	8	10
Lead	µg/L	0.1	50
Copper	µg/ L	35	50
Selenium	µg/ L	3.1	10
Vanadium	µg/ L	18.5	250
Zinc	µg/ L	< 5	3000
pH		11.5	5.5-12

*Regulatory limit: Table 1 of Annex III of Ministerial Decree No 186 of 05th April 2006.

6.4 Conclusions

In this work, a pilot-scale process for the treatment of landfill leachate characterized by a high concentration of ammoniacal nitrogen and perfluoroalkyl substances (PFAS), as well as by the presence of several metals, was developed and tested. The treatment process combined several technologies to address the different contaminants present, using membrane filtration (i.e., nanofiltration and reverse osmosis), coagulation/precipitation, and filtration on activated carbon processes for organic chemicals removal and employed a two-phase biological treatment (biological oxidation and anaerobic nitrification/denitrification) to remove the high levels of ammonia. Reverse osmosis was used to recover the salts present in the leachate to be used as road salt. HPSS technology was applied to the sludge obtained from the Fenton process carried out on the NF concentrate. In detail, a thermal desorption process was used to remove PFAS from the granular stabilized material. However, it was not possible to calculate detailed removal rates as the concentration of PFAS in the material was always below the detection limit. Leaching tests were carried out on the final granules, showing concentrations of metals in the eluates within regulatory limits, but also the presence of PFAS, which indicates that additional steps will be needed to ensure that no PFAS remains in the granules.

7. Chapter 7. Conclusions

This Ph.D. thesis work investigated the efficiency of several water and wastewater treatment processes, including a Venturi tube cavitation system for organic contaminants removal and a multi-stage treatment train to remove organic and inorganic compounds from landfill leachate.

Since it is very time-consuming for scholars to study all the publications related to a specific field to identify the overall trends and gaps in knowledge, scientometric analysis plays a vital role in assessing and integrating published works, enhancing researchers' information, and analyzing the future directions of scientific disciplines. Within this context, a scientometric analysis on the works published in the last twenty years on cavitation applied to water and wastewater treatment was carried out, showing an increasing interest in both hydrodynamic and ultrasonic cavitation, while laser and particle cavitation remained marginal. In particular, hydrodynamic cavitation has gained attention for its industrial uses, particularly when combined with other advanced oxidation processes (AOP). Ultrasonic cavitation, despite energy and cost limitations, is becoming more relevant due to recent technological advancements, especially in developed countries. Key research gaps include the need for improved energy efficiency, sustainability, and economic viability of both cavitation methods. In detail, integrating hydrodynamic or ultrasonic cavitation into conventional treatment methods presents a promising yet currently under-explored area of research.

Moreover, another scientometric study carried out on the application of Life Cycle Assessment (LCA) on the topic of water use and management elucidated that this field has evolved significantly over the past 12 years, as shown by advancements in frameworks, databases, and analytical software tools. However, integrating AI and machine learning into LCA remains an essential research opportunity, promising optimized water management processes and a deeper understanding of environmental, economic, and social interconnections. Additionally, studies on the environmental impacts of innovative water treatment technologies are still limited due to inadequate data and the need to adapt current LCA methodologies. The investigation of the economic aspects of the studied processes (i.e., Life Cycle Costing) also requires greater attention and standardization to enhance decision-making processes by clearly identifying trade-offs between environmental sustainability and financial feasibility.

Regarding the experimental activities of the present thesis, the results have shown substantial influence of Venturi tube's configuration on the generated bubbles' characteristics. In detail, the bubbles with the smallest average diameter, (i.e., 151nm), the highest frequency (i.e., 89%), and the lowest Zeta potential (i.e., -15.4) were realized with a specific Venturi tube with a throat diameter of 1mm, throat length of 9.85mm, divergence length of 67.15mm and a divergence angle of 1.96°. The

developed pilot-scale (i.e., 14L of treated water) hydrodynamic cavitation system, enhanced with nanobubbles, effectively degraded Humic Acid (HA), Reactive Red 120 (RR120), and Congo Red (CR) using oxidants, (i.e., Hydrogen Peroxide (H_2O_2) or Sodium Persulfate (PS)). Optimal degradation occurred under acidic conditions (i.e., pH=2), using PS for HA and CR, while the best results for RR120 were obtained using H_2O_2 . The observed primary degradation for CR and RR120 was about 95%, while mineralization of HA was about 66%. However, the presence of salt significantly hindered degradation efficiency for all substances by 10-20%.

Regarding landfill leachate, this study developed and tested a pilot-scale (i.e., 10L/day of treated leachate) treatment system for removing high concentrations of ammoniacal nitrogen, PFAS (e.g., PFOS, PFOA, PFBA, PFBS, PFHpA, PFHxA, PFHxS, and PFPeA), and metals (e.g., Arsenic, Lead, Copper, and Chromium). The process combined membrane filtration (i.e., nanofiltration and reverse osmosis), coagulation/precipitation, activated carbon filtration, and a two-phase biological treatment to ensure PFAS, ammonia and metals removal. Moreover, the High-Performance Solidification/Stabilization (HPSS) technology was applied to the sludge obtained from the Fenton process carried out on the nanofiltration concentrate, and leaching tests on the stabilized granular material showed metal concentrations within regulatory limits but indicated residual PFAS (e.g., PFBA, PFBS, PFHxA, and PFHxS), suggesting that further treatment of the granules will be necessary. Salts (mainly Sodium Chloride) present in the leachate were recovered using reverse osmosis and concentration under vacuum to be repurposed as road salt.

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