STRATEGY FOR REMOVING MICROPOLLUTANTS FROM INDUSTRIAL EFFLUENTS

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ABSTRACT

This study addresses strategies for the removal of micropollutants from industrial effluents, with emphasis on the characterization and treatment of water contaminated by microplastics. In light of increasing water pollution and the challenges associated with its mitigation, the research integrates a bibliographic review and a laboratory-scale experimental approach, compiling technical, regulatory, and methodological information applicable to sustainable water resource management. The main sources, characteristics, and behavior of microplastics in industrial environments are discussed, along with identification and quantification methods, considering the analytical limitations associated with the morphological and compositional diversity of these particles. Regarding treatment technologies, the study evaluates physicochemical and advanced processes, including coagulation–flocculation, filtration, and membrane separation, highlighting their efficiencies and limitations in the removal of particles of different sizes. Additionally, a critical assessment of adsorbent materials, such as activated carbon, is presented, emphasizing their greater applicability for dissolved compounds and their limitations when applied to particulate contaminants such as microplastics. Finally, the study highlights the importance of integrated treatment systems, as well as the need for methodological standardization and regulatory advancements to support the control of emerging contaminants and the protection of aquatic ecosystems.

Keywords: micropollutants, microplastics, industrial effluents, water treatment.

RESUMO

A presente pesquisa aborda estratégias para a remoção de micropoluentes em efluentes industriais, com ênfase na caracterização e no tratamento de águas contaminadas por microplásticos. Diante do aumento da poluição hídrica e dos desafios associados à sua mitigação, o estudo integra revisão bibliográfica e abordagem experimental em escala laboratorial, reunindo informações técnicas, regulatórias e metodológicas aplicáveis à gestão sustentável dos recursos hídricos. São discutidas as principais fontes, características e comportamento dos microplásticos em ambientes industriais, bem como os métodos de identificação e quantificação, considerando as limitações analíticas associadas à diversidade morfológica e composicional dessas partículas. No que se refere às tecnologias de tratamento, o trabalho analisa processos físico-químicos e avançados, incluindo coagulação-floculação, filtração e separação por membranas, destacando suas eficiências e limitações na remoção de partículas de diferentes dimensões. Adicionalmente, é realizada uma avaliação crítica do uso de materiais adsorventes, como o carvão ativado, evidenciando sua maior aplicabilidade na remoção de compostos dissolvidos e suas limitações frente a contaminantes particulados, como os microplásticos. Por fim, o estudo ressalta a necessidade de adoção de sistemas de tratamento integrados, bem como de avanços na padronização metodológica e nos marcos regulatórios, visando ao controle de contaminantes emergentes e à proteção dos ecossistemas aquáticos.

Palavras-chave: micropoluentes, microplásticos, efluentes industriais, tratamento de água.

LIST OF FIGURES

LIST OF TABLES

LIST OF ABBREVIATIONS AND ACRONYMS

ACS – American Chemical Society

ANA – Brazilian National Water and Basic Sanitation Agency

APA – Portuguese Environment Agency

BOPP – Biaxially Oriented Polypropylene

GAC – Granular Activated Carbon

VOCs – Volatile Organic Compounds

BOD – Biochemical Oxygen Demand

COD – Chemical Oxygen Demand

EPS – Expanded Polystyrene

DWTP – Drinking Water Treatment Plant

WWTP – Wastewater Treatment Plant

ETP – Effluent Treatment Plant

EU – European Union

EUR-Lex – European Union Legislation Access Portal

DAF – Dissolved Air Flotation

FTIR – Fourier Transform Infrared Spectroscopy

GC/MS – Gas Chromatography coupled with Mass Spectrometry

MF – Microfiltration

MP – Microplastic

NF – Nanofiltration

RO – Reverse Osmosis

PE – Polyethylene

PET – Polyethylene Terephthalate

AOP – Advanced Oxidation Process

PP – Polypropylene

PS – Polystyrene

PVC – Polyvinyl Chloride

Py-GC/MS – Pyrolysis coupled with Gas Chromatography and Mass Spectrometry

Raman – Raman Spectroscopy

TSS – Total Suspended Solids

UF – Ultrafiltration

UN – United Nations

UNESCO – United Nations Educational, Scientific and Cultural Organization

1. INTRODUCTION

Water is an essential resource for maintaining ecosystems, socioeconomic development, and human health, and it plays a central role in environmental sustainability (TUNDISI; MATSUMURA-TUNDISI, 2017). However, population growth combined with the intensification of industrial activities has significantly increased pressure on water bodies, leading to deterioration in water quality. International reports indicate that urban, agricultural, and industrial expansion have heightened the risks of water scarcity and contamination on a global scale (UNESCO, 2023; UN, 2022).

Several industrial sectors—including plastics, paints, petrochemicals, pharmaceuticals, food processing, and textiles—generate effluents containing persistent and recalcitrant contaminants that can cause long-term environmental impacts (PEREIRA; LOPES, 2021). Among these contaminants, microplastics have emerged as a primary environmental concern due to their widespread distribution and environmental persistence (ERIKSEN *et al.*, 2018).

Microplastics are defined as synthetic polymer particles measuring less than 5 mm in size, originating either from industrial production processes or from the fragmentation of larger plastic materials. Studies have demonstrated their presence across multiple environmental compartments, including sediments, soils, marine environments, drinking water, and industrial and municipal effluents (WANG; LIN; CHEN, 2020). In aquatic systems, these particles are associated with physicochemical changes, impacts on biodiversity, and potential risks to human health. Moreover, they may act as vectors for heavy metals, persistent organic pollutants, pharmaceuticals, and pathogenic microorganisms (COUCEIRO, 2019; PAN *et al.*, 2023).

Within the industrial context, microplastic generation is linked to the production and handling of polymer resins, mechanical abrasion, paint formulation, equipment wear, and the degradation of improperly discarded plastic waste. Studies indicate that plastic processing, paint, rubber, packaging, and cosmetic industries contribute significantly to microplastic loads in highly polluted effluents (OLIVEIRA, BARBOSA, MENDONÇA, 2023; BARRETO, 2024). Although treatment plants can partially remove these particles, smaller fractions—particularly those below 20 µm—are often not retained by conventional treatment processes and may ultimately reach natural water bodies (WANG *et al.*, 2020).

In this context, the demand for effective technologies to remove microplastics from industrial and municipal effluents has grown substantially. Conventional processes such as coagulation, flocculation, sedimentation, and filtration exhibit variable efficiency, depending on particle type and size as well as effluent composition (CRISTÓVÃO *et al*., 2014). In drinking water treatment, removal efficiencies ranging from 40% to 98% have been reported, whereas in industrial effluents, the high variability of organic loads may compromise process performance (BARRETO, 2024).

Accordingly, advanced methods—including adsorption using modified biochar, dissolved air flotation, advanced oxidation processes, and hybrid technologies—have been widely investigated. Low-cost adsorbents, particularly biochar derived from agricultural residues and chemically modified, have demonstrated promising results, achieving efficiencies exceeding 70% under laboratory conditions (LI; SHI, 2022; OLIVEIRA; BARBOSA; MENDONÇA, 2023).

Despite these advances, the characterization and quantification of microplastics continue to face significant challenges, primarily due to the lack of international methodological standardization, especially for smaller particles. Techniques such as Fourier Transform Infrared Spectroscopy (FTIR), Raman spectroscopy, optical microscopy, and pyrolysis coupled with gas chromatography/mass spectrometry (Py-GC/MS) present specific limitations, often requiring the combination of multiple methods to ensure more robust identification.

In this framework, understanding the occurrence and removal methods of microplastics in industrial effluents is essential to support public policy development and guide technological adaptations. The European Union and Portugal have advanced in incorporating directives related to microplastics and micropollutants into their regulatory frameworks (Regulation (EU) 2025/2365; Regulation (EU) 2023/2055; Decree-Law No. 78/2021; APA, 2025). Therefore, this literature review aims to analyze the presence of microplastics in industrial effluents and to critically assess the available removal technologies, identifying gaps and future perspectives for mitigating this emerging contaminant.

2. THEORETICAL FRAMEWORK AND LITERATURE REVIEW

2.1 Microplastics: Concepts, Sources, and Impacts in Industrial Effluents

The growing presence of microplastics in aquatic environments has attracted substantial scientific, regulatory, and industrial attention worldwide. These emerging contaminants pose a significant risk due to their environmental persistence, morphological diversity, chemical complexity, and capacity to interact with other pollutants. In the industrial context, microplastics are particularly noteworthy because of the frequency with which they are generated and discharged into effluents, either directly or indirectly, throughout production processes (UNESCO, 2023; UN, 2022).

This chapter presents the fundamental concepts related to microplastics, including their classification, formation pathways, occurrence in industrial effluents, and environmental implications, integrating both national and international studies.

2.1.1 Definition and Characteristics of Microplastics

Microplastics are solid particles composed of synthetic polymers measuring less than 5 mm in size and may occur in various forms, including pellets, fragments, fibers, films, and spheres (ERIKSEN *et al*., 2018). Their high durability and physicochemical resistance hinder environmental degradation, ensuring persistence across different environmental compartments.

The literature distinguishes between primary microplastics, which are intentionally manufactured at this scale—such as cosmetic microbeads and industrial pellets—and secondary microplastics, which result from the fragmentation of larger plastic items due to mechanical abrasion, ultraviolet radiation, and oxidative processes (COUCEIRO, 2019). Secondary microplastics are generally more heterogeneous, exhibiting wide variation in particle size distributions and additive compositions.

Chemically, microplastics are composed of conventional polymers such as polyethylene (PE), polypropylene (PP), polystyrene (PS), polyvinyl chloride (PVC), and polyethylene terephthalate (PET), in addition to plasticizers, stabilizers, and colorants that may leach over time, thereby increasing associated toxicological risks (BARRETO, 2024). Furthermore, microplastics may act as carriers of adsorbed contaminants, including polycyclic aromatic hydrocarbons, pesticides, and metals (HORTON et al., 2017).

2.1.2 Classification of Microplastics

Regarding origin, microplastics are commonly classified as primary or secondary. Primary microplastics are particles intentionally manufactured at microscopic dimensions, such as industrial pellets used as raw materials in the plastics industry, microabrasives used in industrial processes, and microbeads incorporated into cosmetic products. In contrast, secondary microplastics result from the fragmentation and degradation of larger plastic materials, including packaging debris, plastic films, textile fibers released during washing processes, and residues originating from urban areas (HORTON *et al*., 2017).

In terms of morphology, microplastics exhibit considerable diversity and may occur as irregular fragments, elongated fibers, thin films, spherical or cylindrical pellets, expanded polystyrene (EPS) foams, and uniform spheres typically associated with industrial or cosmetic applications. This morphological variability directly influences their behavior in aquatic environments, affecting transport dynamics, sedimentation patterns, and interactions with organisms (HORTON *et al*., 2017).

Regarding size, microplastics span a broad range, from particles measuring approximately 1 mm to progressively smaller fractions, including 100 to 1000 μm, 20 to 100 μm, and particles smaller than 20 μm. The smallest fractions pose the greatest analytical challenge, as they require the application of advanced characterization techniques (BARRETO, 2024; RUFINO *et al*., 2023).

Regarding polymer composition, polyethylene (PE) and polypropylene (PP) are the most frequently identified polymers, primarily due to their extensive use in packaging and consumer products and their relatively low density. This property favours their persistence within the water column, enhancing their dispersion and long-term presence in aquatic environments (HORTON *et al*., 2017).

2.1.3 Industrial Sources of Microplastics

Plastic processing industries are among the primary direct sources of microplastics to the environment. During stages such as extrusion, grinding, cutting, mixing, and pigmentation, pellet losses and the generation of fine plastic dust are recurrent, and these materials can be easily dispersed. In specific processes, including bi-axially oriented polypropylene (BOPP) film production, injection moulding, and thermoforming, small fractions of plastic material may be conveyed into industrial drainage systems, thereby contributing to microplastic loads in effluents (ANDRADY, 2011; GESAMP, 2015; HORTON et al., 2017).

Beyond the plastics processing sector, other industrial segments are also recognized as significant potential sources. The paints and coatings industry, for instance, releases polymeric particles primarily during the cleaning of equipment and production lines (PREUSS *et al*., [n.d.];

RESMINI, 2023). In the textile sector, microfibers are released extensively during fabric washing and dyeing. The cosmetic industry has historically incorporated plastic microbeads into exfoliating products, serving as a direct source of primary microplastics. The petrochemical sector, in turn, stands out as a major producer of polymer resins, while the rubber industry contributes through mold wear and the use of abrasive materials (GESAMP, 2015; HORTON *et al*., 2017; COUCEIRO, 2019; RESMINI, 2023; BARRETO, 2024).

From an environmental perspective, microplastics may enter industrial effluents either as lost raw material—characterizing primary microplastics—or, due to degradation processes, leaching, and mechanical abrasion throughout production chains, as discussed by TAE Magazine (2019).

2.1.4 Occurrence in Industrial and Municipal Effluents

The occurrence of microplastics in industrial and municipal effluents has been widely documented in the scientific literature, although reported values exhibit considerable variability due to methodological differences, facility scale, and the specific characteristics of the productive sectors analyzed (PEREIRA; LOPES, 2021; PAN *et al.*, 2023). The absence of standardized protocols for sampling, pre-treatment, and quantification hinders direct comparisons among studies; nevertheless, there is a broad consensus regarding the recurrent presence of these particles in industrial discharges and wastewater treatment plants.

At the international level, Pan et al. (2023) report elevated concentrations of microplastics in effluents from plastic processing, textile, cosmetic, rubber, and paint industries, with a predominance of synthetic fibers and irregular fragments, generally below 500 µm. In many cases, the most frequently identified polymers are polyethylene (PE), polypropylene (PP), and polyester, reflecting the production profile of the industrial chains evaluated. Barreto (2024) emphasizes that, although water treatment plants (WTPs) and wastewater treatment plants (WWTPs) can achieve significant removal of particles larger than 50 µm through coagulation, sedimentation, and filtration, fine fractions—particularly those smaller than 20 µm—often remain in the treated effluent and may be discharged into receiving water bodies.

In the Brazilian context, studies conducted in university and urban environments have demonstrated the presence of microplastics in institutional and municipal effluents. Oliveira (2023), in an analysis of laboratory and institutional effluents from the Federal University of Rio Grande do Norte (UFRN), identified a predominance of fibers and fragments smaller than 100 µm, highlighting marked morphological heterogeneity. The author further notes that smaller particles tend to escape conventional treatment processes, reinforcing the need for complementary technologies to enhance retention.

In addition, national reports, such as those published by the National Water and Basic Sanitation Agency (ANA, 2023), indicate that increasing urbanization and industrial expansion in Brazil have intensified pressure on treatment systems, which were not always designed to remove emerging contaminants, including microplastics. Although Brazilian legislation has not yet established specific discharge limits for microplastics in effluents, concern regarding these contaminants has been progressively incorporated into regulatory and scientific discussions.

Within the European and Portuguese context, concern about emerging contaminants has been integrated into environmental policies and water quality directives, although specific regulatory limits for microplastics in industrial effluents remain insufficiently defined. Reports from the Portuguese Environment Agency (APA, 2022) and European Union guidelines emphasize the need to monitor and mitigate micropollutants in drainage and treatment systems. European studies indicate that conventional WWTPs may retain between 70% and 95% of influent microplastics; however, the remaining fraction—particularly fine particles and synthetic fibers—continues to be discharged into rivers and estuaries, contributing to diffuse environmental contamination (WANG et al., 2020; PAN *et al*., 2023).

Specific sectors, such as the paints and varnishes industry, exhibit a particularly critical emission profile. Resmini (2023) reports high concentrations of polymeric particles associated with pigments and synthetic resins in effluents from this segment, complicating separation by conventional methods due to interactions between hydrophobic particles and dissolved organic matter. Similarly, textile effluents are recognized as important sources of synthetic microfibers, which may represent a substantial proportion of total microplastic loads in municipal systems.

In the context of domestic wastewater, the release of microfibers from the washing of synthetic textiles constitutes one of the main pathways of microplastic entry into sanitation systems. Wang et al. (2020) note that, although biological activated sludge processes promote partial retention of these particles through incorporation into sludge, smaller fractions remain in treated effluents and may ultimately reach surface waters.

Overall, the literature converges on the understanding that, despite technological advances and operational improvements in treatment systems, microplastics continue to occur in significant quantities in industrial and municipal effluents, particularly in smaller particles. This scenario underscores the need for systematic monitoring, the development of standardized methodologies, and the adoption of integrated technologies to enhance the removal efficiency of these emerging contaminants.

2.1.5 Behaviour and Persistence in Aquatic Environments

The persistence of microplastics in the environment is directly related to their high chemical, thermal, and biological resistance, which significantly hinders natural degradation. Unlike organic materials, synthetic polymers do not undergo substantial mineralization under typical environmental conditions. Studies indicate that conventional plastics, such as polyethylene (PE) and polypropylene (PP), may persist in the environment for decades to centuries, depending on environmental conditions and exposure to ultraviolet radiation (ANDRADY, 2011; ERIKSEN *et al.*, 2018).

Environmental processes such as photodegradation, mechanical abrasion, and oxidation do not result in complete polymer mineralization; rather, they promote progressive fragmentation into increasingly smaller particles. This fragmentation increases the proportion of microplastics and nanoplastics, which exhibit greater specific surface area and enhanced potential for interaction with contaminants and living organisms (ERIKSEN *et al.*, 2018). In surface marine environments, continuous exposure to UV radiation may accelerate physical degradation; however, chemical degradation remains extremely slow, with rates varying according to polymer type and radiation intensity (ANDRADY, 2011).

The environmental behaviour of microplastics is governed by a combination of physicochemical and hydrodynamic factors. Polymer density is a determining parameter: low-density materials, such as PE (≈ 0.91 to 0.96 $g \cdot cm^{-3}$) and PP (≈ 0.90 to 0.92 $g \cdot cm^{-3}$), tend to remain at the surface or within the water column for extended periods and may be transported over long distances by currents. In contrast, higher-density polymers, such as polyvinyl chloride (PVC) and polyethylene terephthalate (PET), tend to sediment more readily, accumulating in riverbeds, lakes, and coastal zones (HORTON *et al.*, 2017).

Another relevant factor is biofilm formation on particle surfaces. Microbial colonization can increase the apparent density of microplastics over time, promoting their sinking even when originally buoyant. This process alters the vertical dynamics of particles, favouring successive cycles of flotation and sedimentation, thereby prolonging their residence time within aquatic systems (ERIKSEN *et al.*, 2018).

Furthermore, interactions with natural organic matter, dissolved salts, and mineral particles influence aggregation, transport, and deposition processes. In low-energy hydrodynamic environments, such as reservoirs and estuarine zones, microplastics may accumulate in sediments for extended periods, acting as secondary reservoirs and potentially being remobilized during flood events or physical disturbances (UNESCO, 2023).

In terms of spatial dynamics, irregularly shaped fragments tend to deposit preferentially in low-current zones, whereas fibrous particles exhibit a greater capacity to remain suspended for prolonged periods, facilitating long-distance transport. Synthetic fibers, due to their high length-to-diameter ratio, display distinct hydrodynamic behaviour and may remain in suspension even when their density exceeds that of water (HORTON *et al*., 2017; UNESCO, 2023).

Overall, the literature indicates that once introduced into aquatic environments, microplastics may persist for periods far exceeding the hydraulic residence time of water in natural systems, progressively accumulating in sedimentary and biological compartments. This prolonged persistence, combined with continuous fragmentation, explains the observed tendency for increasing environmental concentrations over time, even under scenarios involving partial emission reductions.

2.1.6 Interactions with Other Contaminants

Microplastics exhibit a high affinity for toxic compounds due to their large specific surface area, predominantly hydrophobic nature, and the presence of chemical additives incorporated into the polymer matrix during manufacturing. These characteristics favour the adsorption of a wide range of contaminants, including pharmaceuticals, pesticides, polycyclic aromatic hydrocarbons (PAHs), heavy metals, surfactants, industrial dyes, and volatile organic compounds (VOCs) (BARLOW *et al*., 1996; COUCEIRO, 2019; HORTON *et al*., 2017).

Interactions between microplastics and contaminants occur primarily through hydrophobic partitioning, electrostatic interactions, and Van der Waals forces. Polymers such as polyethylene (PE) and polypropylene (PP), owing to their high hydrophobicity, demonstrate significant adsorption capacity for persistent organic compounds. Furthermore, environmental aging processes—driven by ultraviolet radiation, oxidation, and mechanical abrasion—increase surface roughness and promote the formation of oxygen-containing functional groups, thereby further enhancing adsorption capacity (ANDRADY, 2011; ERIKSEN *et al*., 2018).

Studies report that microplastics can concentrate contaminants to levels exceeding those found in the surrounding water, effectively functioning as mobile micro-reservoirs of pollutants. Horton *et al*. (2017) emphasize that this adsorption capacity is particularly relevant in urban and industrial environments, where dissolved micropollutant loads are elevated. Similarly, Wang et al. (2020) demonstrate that plastic particles can adsorb hydrophobic organic compounds and metals, altering their mobility and potential for biological exposure.

In this context, microplastics act as vectors of pollutants in aquatic systems, concentrating potentially toxic substances and facilitating their dispersion over long distances, particularly when suspended in the water column. Pan *et al*. (2023) highlight that such interactions may increase the bioavailability of emerging pollutants, intensifying toxicological risks to aquatic organisms through the direct ingestion of contaminated particles. Following ingestion, physicochemical changes in the gastrointestinal tract may promote the partial release of adsorbed contaminants, thereby increasing internal exposure.

In addition to externally adsorbed contaminants, it is important to consider that many polymers contain additives incorporated during production, such as plasticizers, thermal stabilizers, flame retardants, and pigments, which may leach over time. Couceiro (2019) and Eriksen *et al*. (2018) note that these additives may possess intrinsic toxic potential, contributing to combined effects when associated with the adsorption of environmental pollutants.

Thus, the role of microplastics as contaminant carriers increases the complexity of environmental assessment, as their impacts extend beyond their mere physical presence of particles to include processes of transport, concentration, and the potential release of toxic substances along the trophic chain.

2.1.7 Environmental and Human Health Risks

Microplastics pose a multifactorial environmental risk involving physical, chemical, and ecotoxicological impacts. In aquatic environments, these particles are ingested across multiple trophic levels, including zooplankton, molluscs, crustaceans, and fish. Studies demonstrate that the presence of microplastics in the gastrointestinal tract may cause intestinal obstruction, reduced food intake, behavioural alterations, and decreased energy efficiency, ultimately impairing growth and reproduction (ERIKSEN *et al*., 2018; HORTON *et al*., 2017).

Beyond physical effects, microplastics may induce inflammatory responses, oxidative stress, and histological alterations in exposed tissues. Mechanical abrasion caused by particles and their interactions with epithelial cells can trigger localized inflammatory processes, while smaller particles can cross biological barriers and reach internal tissues (WANG; LIN; CHEN, 2020). Experimental evidence indicates that particles smaller than 20 µm have a greater capacity for cellular translocation, potentially reaching circulatory and lymphatic systems.

Another critical factor is associated with chemical additives incorporated into polymers, such as plasticizers, stabilizers, and flame retardants. These compounds may leach within the gastrointestinal tract after ingestion, increasing toxicological risks. Barreto (2024) emphasizes that the combined presence of microplastics and adsorbed contaminants may potentiate adverse effects, creating multiple exposure pathways. Complementarily, Eriksen et al. (2018) note that microplastics can function as vectors for persistent organic pollutants and heavy metals, thereby enhancing their bioavailability.

At the ecosystem level, trophic transfer represents one of the primary environmental concerns. Particles ingested by organisms at the base of the food web may be transferred to predators, promoting bioaccumulation and potential biomagnification along the food chain (HORTON *et al.*, 2017). Although the magnitude of biomagnification remains under scientific debate, evidence suggests that microplastics and associated contaminants may persist in biological tissues and interfere with metabolic functions.

Regarding human health, exposure occurs mainly through the consumption of drinking water, seafood, and sea salt, as well as through inhalation of airborne particles. Wang, Lin, and Chen (2020) highlight that smaller particles pose a greater risk because they can cross epithelial cell barriers that protect body surfaces, potentially triggering systemic inflammatory responses, oxidative stress, and immunological alterations. Although significant gaps remain concerning chronic effects in humans, recent studies have identified microplastics in human tissues and biological fluids, underscoring the need for further investigation.

The complexity of these risks is amplified by particle heterogeneity, as microplastics vary in size, shape, composition, and degree of environmental aging, complicating the standardization of toxicological assessments. The absence of specific regulatory limits for microplastics in effluents and drinking water represents a significant normative challenge. The European Union has progressively incorporated directives related to emerging micropollutants into its regulatory framework (EUROPEAN UNION, 2024), reflecting growing recognition of the need for monitoring and mitigation.

In this context, environmental risks and potential human health impacts underscore the urgency of developing standardized analytical methodologies, implementing systematic monitoring, and adopting effective removal technologies—particularly in industrial effluents, which serve as important primary emission points for these contaminants.

3. CHARACTERIZATION OF INDUSTRIAL EFFLUENTS CONTAINING MICROPLASTICS

The characterization of industrial effluents containing microplastics constitutes an essential step in understanding the extent of contamination, identifying physicochemical properties that influence particle removal, and selecting appropriate treatment technologies. Unlike other pollutants, microplastics exhibit substantial heterogeneity and may be associated with organic compounds, surfactants, pigments, solvents, and suspended solids, rendering their detection and quantification particularly complex (PEREIRA; LOPES, 2021). Furthermore, the variable composition of industrial effluents directly affects separation and identification processes.

This chapter presents the principal parameters involved in the characterization of effluents containing microplastics, as well as the analytical methods employed for their morphological identification and determination of polymer composition.

3.1 Importance of Characterization in Industrial Effluents

Industrial effluents differ substantially from domestic wastewater due to greater compositional variability, the presence of sector-specific chemicals, elevated solid content, and significant concentrations of organic matter (APA, 2022; ANA, 2023). Polymeric particles may be associated with oils, emulsions, pigments, resins, or organic aggregates, which complicates their analytical determination.

Studies indicate that microplastics may be embedded within complex matrices or adhered to paint particles, synthetic fibers, and inorganic materials, thereby hindering direct identification (RESMINI, 2023). In addition, surfactants and solvents present in industrial effluents may alter the colloidal behaviour of microplastics, stabilizing fine particles and preventing sedimentation (RESMINI, 2023).

Accordingly, detailed characterization is indispensable not only to quantify the presence of microplastics but also to understand their hydrodynamic behaviour and interactions with the liquid matrix—information that is essential for the proper design and sizing of removal technologies (BARRETO, 2024).

Table 1 presents parameters associated with industrial wastewater discharge standards in public drainage systems, based on European regulations (EUR-LEX, 2014; PORTUGAL, 1993; PORTUGAL, 1995).

Table 1 – Regulatory parameters for the discharge of industrial wastewater into public drainage systems.

Parameters	**Emission Limit Value**
pH	6.0 – 9.0
Temperature (°C)	30
Biochemical Oxygen Demand after 5 days, 20°C (mg/L O_2)	500
Chemical Oxygen Demand (mg/L O_2)	1100
Total Suspended Solids (mg/L)	650
Aluminum (mg/L Al)	10
Total Iron (mg/L Fe)	2.0
Manganese (mg/L Mn)	2.0
Odor	Not detectable at a 1:20 dilution
Color	Not detectable at a 1:20 dilution
Phenols (mg/L C_6H_5OH)	0.5
Oils and Grease (mg/L)	100
Sulfides (mg/L S)	1.0
Sulfites (mg/L SO_3)	1.0
Sulfates (mg/L SO_4)	400
Total Phosphorus (mg/L P)	15
Ammoniacal Nitrogen (mg/L NH_4)	50
Total Nitrogen (mg/L N)	70
Nitrates (mg/L NO_3)	50
Aldehydes (mg/L)	1.0
Arsenic (mg/L As)	1.0
Lead (mg/L Pb)	1.0
Cadmium (mg/L Cd)	0.2
Chromium (mg/L Cr)	2.0
Hexavalent Chromium (mg/L Cr VI)	0.1
Copper (mg/L Cu)	1.0
Nickel (mg/L Ni)	2.0
Mercury (mg/L Hg)	0.05
Mineral Oils (mg/L)	15
Detergents (mg/L, sodium lauryl sulfate)	10

Source: Adapted from EUR-Lex (2014) and Portugal (1993; 1995).

In the Brazilian context, industrial effluent discharge standards are primarily established by CONAMA Resolution No. 430/2011, which defines conditions and limits for discharge into water bodies, complementing CONAMA Resolution No. 357/2005, responsible for the classification of water bodies and the definition of their respective uses (BRASIL, 2011; BRASIL, 2005). Unlike Portuguese legislation, which establishes specific parameters for discharges into public drainage systems, Brazilian federal regulation predominantly focuses on direct discharge into surface receiving waters.

Table 2 – Brazilian regulatory parameters for industrial effluent discharge into water bodies

Parameter	Emission limit value (Brazil – CONAMA 430/2011)
pH	5.0 – 9.0
Temperature (°C)	≤ 40
BOD_5 (20 °C) (mg/L O_2)	60 mg/L or a minimum 60% removal
Oils and grease (mg/L)	100
Settleable solids	1 mL/L
Floating materials	Absent
Arsenic (mg/L As)	0.5
Lead (mg/L Pb)	0.5
Cadmium (mg/L Cd)	0.2
Total chromium (mg/L Cr)	1.0
Hexavalent chromium (mg/L Cr VI)	0.1
Copper (mg/L Cu)	1.0
Nickel (mg/L Ni)	2.0
Mercury (mg/L Hg)	0.01
Zinc (mg/L Zn)	5.0

Source: Adapted from Brasil (2011).

Compared with certain Brazilian parameters, BOD_5 is more stringent, with a limit significantly lower than that set by European regulations for discharges into public sewer networks. However, as observed in both Portuguese and European regulatory frameworks, Brazilian legislation does not yet establish specific limits for microplastics in industrial effluents. Instead, microplastics are addressed only indirectly through general parameters such as total suspended solids, turbidity, and floating materials (BRASIL, 2011; BRASIL, 2005).

In addition to federal legislation, state-level regulations and sanitation utility standards may impose additional criteria for discharges into sewer systems, varying by regional jurisdiction and the classification of the receiving water body. This regulatory heterogeneity underscores the need for normative updates to address emerging contaminants such as microplastics, whose presence in industrial effluents has been extensively documented in recent scientific literature (BRASIL, 2011; BRASIL, 2005).

3.2 Relevant Physicochemical Parameters

The characterization of effluents containing microplastics should be conducted using physicochemical parameters defined in national and international technical standards, which provide essential support for interpreting particle behaviour in aqueous environments and selecting appropriate treatment technologies. As discussed by Tundisi and Matsumura-Tundisi (2017), the integrated analysis of these parameters is indispensable for assessing effluent quality and its potential environmental impacts.

pH constitutes one of the most relevant parameters, as it directly influences the stability of colloidal systems, the ionization state of additives present in polymers, and the efficiency of treatment processes such as coagulation and flocculation. Variations in pH may alter the surface charge of both microplastics and coagulants, thereby affecting floc formation and fine particle removal. Moreover, certain polymer additives exhibit pH-dependent acidic or basic behaviour, which may modify interactions among microplastics, organic matter, and other dissolved contaminants (TUNDISI; MATSUMURA-TUNDISI, 2017; COUCEIRO, 2019; PAN *et al*., 2023).

Turbidity is widely used as an indirect indicator of suspended solids in effluents. In industrial systems, elevated turbidity values are often associated with the presence of polymer fragments, synthetic fibers, and colloidal particles, including microplastics. However, turbidity measurements may mask the presence of finer microplastics, particularly those smaller than 20 µm, hindering visual detection and requiring complementary analytical techniques for specific identification (HORTON *et al.*, 2017; BARRETO, 2024; PAN *et al*., 2023).

Total suspended solids (TSS) represent another fundamental parameter in the characterization of effluents containing microplastics. In certain industrial sectors, such as plastics processing, paints, and textiles, fine polymeric particles may contribute significantly to the TSS fraction. Oliveira (2023) emphasizes that the presence of microplastics may increase TSS values without necessarily reflecting an increase in organic or mineral matter, underscoring the need for caution when interpreting this parameter in isolation.

Chemical Oxygen Demand (COD) and Biochemical Oxygen Demand (BOD) are traditionally used as indicators of biodegradable organic load in effluents. However, in systems containing microplastics, interpreting these parameters may become more complex. Although microplastics do not directly contribute to BOD due to their polymeric nature and low biodegradability, several studies demonstrate that microplastics exhibit a high capacity to adsorb hydrophobic organic compounds, such as oils, surfactants, pharmaceuticals, and other micropollutants, potentially leading to increased measured COD values in effluents (HODSON *et al*., 2017; WANG *et al*., 2018).

In addition, microplastics may serve as reactive surfaces for microbial biofilm formation, indirectly influencing biological treatment processes and clarification mechanisms. Such interactions may alter the availability of organic matter to microorganisms and affect the efficiency of conventional removal processes, contributing to nonconventional interpretations of COD and BOD parameters, particularly in complex industrial effluents (RUMMEL *et al*., 2017; LI; LIU; CHEN, 2018).

Color and associated compounds are particularly important parameters in effluents from the paint, coating, and graphic industries. In these cases, pigmented microplastics and polymeric particles coated with dyes contribute significantly to apparent and true color values, affecting not only the aesthetic quality of the effluent but also the efficiency of physicochemical treatment processes (BARRETO, 2024; PAN *et al*., 2023). Resmini (2023) notes that pigmented polymer particles exhibit distinct behaviour in adsorption and filtration processes, reinforcing the need for integrated parameter assessment.

Finally, effluent density and viscosity directly influence the transport and separation mechanisms of microplastics. At elevated concentrations, these particles may alter the viscosity of the medium, interfering with sedimentation, flotation, and the hydraulic performance of filtration and membrane systems. Changes in these parameters may compromise the efficiency of treatment units designed for conventional effluents, making their consideration essential in system design and operation (TUNDISI; MATSUMURA-TUNDISI, 2017).

Overall, the integrated analysis of parameters such as pH, turbidity, total suspended solids (TSS), COD, and BOD enables a more precise understanding of microplastic behaviour in industrial effluents, particularly with respect to colloidal stability, aggregation tendency, and interaction with dissolved organic matter. Tundisi and Matsumura-Tundisi (2017) emphasize that the combined evaluation of these parameters is essential for interpreting particle dynamics in complex aquatic systems, while Couceiro (2019) highlights that the physicochemical characterization of the liquid matrix directly influences the efficiency of microplastic separation and identification methods.

Pan *et al*. (2023) demonstrate that variations in these parameters significantly affect the performance of treatment technologies—especially in coagulation, flotation, and membrane separation processes—since particle surface charge and hydrodynamic behaviour determine removal efficiency. Complementarily, Barreto (2024) shows that inadequate characterization may compromise the design of technologies such as granular activated carbon and advanced filtration systems, thereby reducing operational efficiency.

Thus, as evidenced by these studies, the integrated analysis of physicochemical parameters not only contributes to understanding the environmental behaviour of microplastics but also provides fundamental technical support for the selection, design, and optimization of treatment technologies for industrial effluents.

3.3 Sampling and Sample Preparation Methods

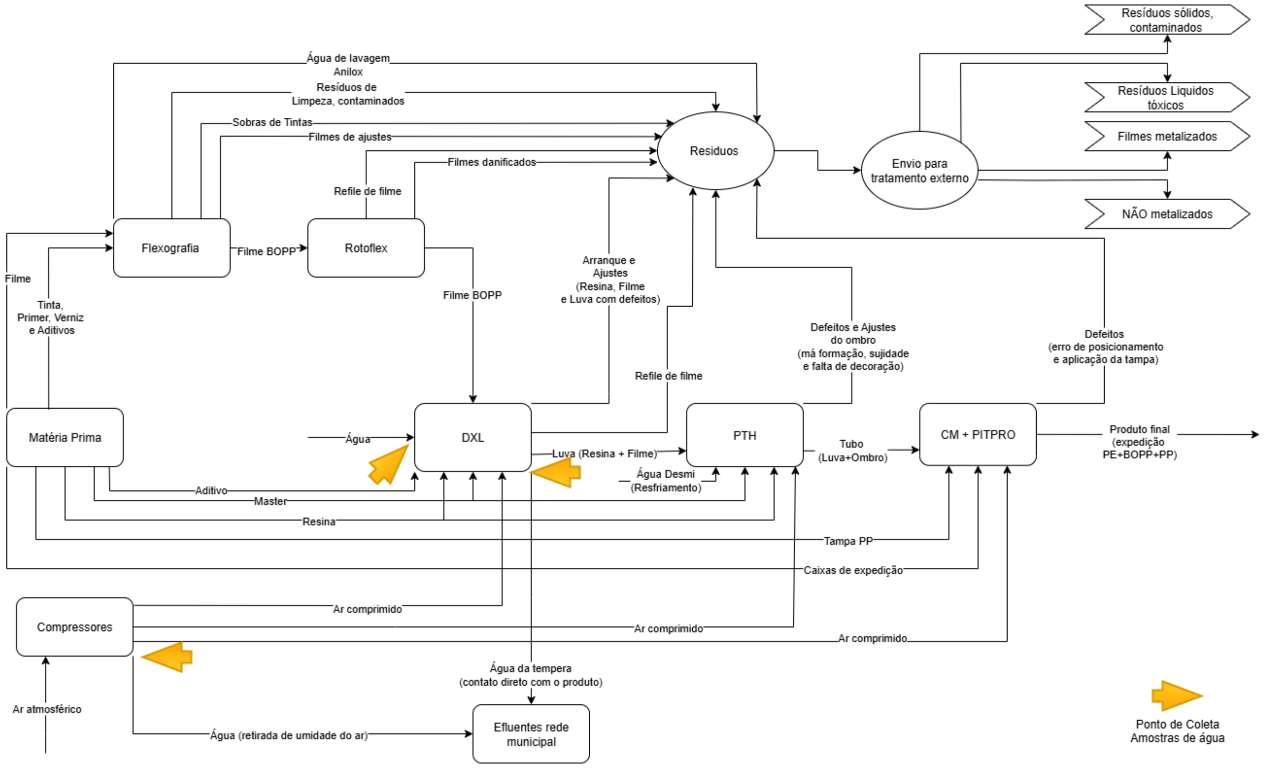
The characterization of microplastics in environmental matrices and industrial effluents generally begins with controlled sample collection, using glass or stainless-steel containers to minimize secondary contamination from conventional plastic materials. In studies involving liquid effluents, sample volumes typically range from 1 to 50 L, depending on the expected microplastic concentration and the analytical sensitivity required by the selected identification method (COUCEIRO, 2019; CAMPANALE *et al.*, 2020).

Following collection, the sample undergoes pre-treatment, which includes sequential filtration or sieving, usually with mesh sizes ranging from 5 mm to 20 µm, to separate particles into different size classes. In matrices with high organic matter content, chemical or enzymatic digestion procedures are applied. Hydrogen peroxide (H_2O_2), Fenton’s reagent, or proteolytic enzymes are frequently used to remove organic material while avoiding significant degradation of synthetic polymers (MAES *et al.*, 2017; COUCEIRO, 2019).

Subsequently, density separation is performed using saline solutions such as NaCl, $ZnCl_2$, or NaI, allowing the flotation of polymers with densities lower than that of the aqueous matrix. The recovered material is then dried, stored under controlled conditions, and forwarded to analytical stages for identification and quantification (CAMPANALE *et al.*, 2020).

The selection of sampling points should follow industrial process flow diagrams, as illustrated in Figure 1.

Figure 1: Flowchart of the plastic industry production process.



Source: Author’s own elaboration (technical information provided by the company).

3.4 Methods for Identification and Quantification

The identification and quantification of microplastics in environmental matrices and industrial effluents are among the main analytical challenges associated with this emerging contaminant. This complexity arises from the wide diversity of particle sizes, shapes, densities, and polymer compositions, as well as from the heterogeneity of the analyzed matrices, which frequently contain high organic loads, suspended solids, oils, and other interfering substances.

Accordingly, the technical and scientific literature recommends adopting integrated methodologies that combine morphological and chemical analyses to improve the reliability of particle identification (HORTON *et al.*, 2017).

3.4.1 Optical Microscopy

Optical microscopy is widely employed as an initial screening step for the visual identification and preliminary quantification of microplastics, particularly for particles larger than 50 µm. The method is based on direct observation of particles retained on filters, enabling morphological classification according to shape (fragments, fibers, films, and pellets) and color (LEFEBVRE *et al.*, 2019).

Although it is a rapid, accessible, and low-cost technique, optical microscopy does not confirm the chemical composition of the analyzed particles and is therefore prone to identification errors, especially when natural particles exhibit morphologies similar to those of synthetic polymers. For this reason, the literature recommends its use only as a preliminary method, to be complemented by spectroscopic techniques for validation of the results (LEFEBVRE *et al.*, 2019; CAMPANALE *et al.*, 2020).

3.4.2 Infrared Spectroscopy (FTIR)

Fourier Transform Infrared (FTIR) spectroscopy is considered one of the most robust and widely applied techniques for the chemical identification of microplastics. The FTIR-ATR (Attenuated Total Reflectance) and transmission FTIR modes are the most used and are suitable for the analysis of particles larger than 20 µm (CAMPANALE *et al.*, 2023).

The method is based on the interaction between infrared radiation and the chemical bonds within the polymer structure, generating characteristic spectra that can be compared with reference libraries. Oliveira (2023) demonstrated strong spectral similarity between reference synthetic microplastics and actual particles isolated from industrial effluents, validating the applicability of FTIR for analyses in complex matrices.

Among the main advantages of this technique are its high reliability in polymer identification and its potential for automation via imaging systems. Micro-FTIR (µ-FTIR) imaging combines optical microscopy with infrared spectroscopy, enabling automated surface mapping of the sample and simultaneous chemical identification of multiple particles. This approach generates two-dimensional images of the spatial distribution of polymers, enabling the concurrent analysis of a large number of particles. However, analytical efficiency may decrease for very small particles or those heavily coated with biofilms (CAMPANALE *et al.*, 2023).

3.4.3 Raman Spectroscopy

Raman spectroscopy offers high spatial resolution and is particularly suitable for analyzing microplastics smaller than 20 µm. The technique enables the identification of the base polymer, as well as pigments, mineral fillers, and additives incorporated into plastic particles (CHEN *et al.*, 2023).

The method is based on the inelastic scattering of incident laser radiation, generating highly specific spectral signatures. Studies indicate that Raman spectroscopy is a complementary technique to FTIR, especially when small particles or pigments hinder infrared analysis. However, fluorescence from residual organic matter may interfere with spectral acquisition, requiring appropriate sample pre-treatment or adjustments in experimental conditions (CHEN *et al*., 2023).

3.4.4 Pyrolysis Coupled to GC/MS (Py-GC/MS)

Pyrolysis coupled with gas chromatography–mass spectrometry (Py-GC/MS) is considered one of the most definitive techniques for identifying and quantifying microplastics, particularly in complex environmental matrices. The method involves the thermal decomposition of polymers under an inert atmosphere, generating characteristic volatile fragments that are subsequently separated and identified by GC/MS (XU *et al*., 2023).

Unlike spectroscopic techniques, Py-GC/MS does not depend on particle size or morphology, allowing direct analysis of solid samples and quantification by mass. Pan et al. (2023) emphasize its high reliability for unequivocal polymer identification and for determining the total microplastic load. The main limitations of the technique include its destructive nature and the absence of morphological information regarding the analyzed particles.

3.4.5 Fluorescent Dyes

The use of fluorescent dyes, particularly Nile Red, has been widely explored as a rapid, low-cost method for preliminary detection and quantification of microplastics. This dye exhibits a high affinity for hydrophobic surfaces, promoting fluorescence of plastic particles when observed under ultraviolet light or fluorescence microscopy (MAES *et al*., 2017).

Despite its applicability in initial screenings and large-scale analyses, the literature highlights important limitations, primarily related to interference from natural organic matter, which may also be stained and lead to false-positive results. For this reason, Nile Red is generally employed as a complementary technique, and confirmation of identified particles by spectroscopic methods, such as FTIR or Raman spectroscopy, is recommended (MAES *et al*., 2017; CAMPANALE *et al*., 2020).

3.5 Challenges in the Characterization of Microplastics

The characterization of microplastics in environmental matrices and industrial effluents is widely recognized as a complex task, hindered by a combination of physicochemical, analytical, and methodological factors. One of the primary challenges is matrix complexity, particularly in industrial effluents, which often contain organic solvents, pigments, oils, greases, and other chemical additives. These constituents may interfere with both pre-treatment procedures and instrumental analyses, compromising separation efficiency, spectral quality, and overall data reliability (COUCEIRO, 2019; RESMINI, 2023).

Another significant challenge is the adsorption of pollutants onto microplastic surfaces. Due to their high specific surface area and the hydrophobic nature of many polymers, these particles act as sorbents. for persistent organic compounds, heavy metals, surfactants, and pharmaceuticals. The presence of such contaminants may mask or alter spectra obtained through techniques such as FTIR and Raman spectroscopy, hindering unequivocal identification of the base polymer and requiring additional cleaning procedures or analytical validation steps (BARRETO, 2024).

The lack of international standardization is a major bottleneck in this field, as different studies employ distinct methodologies for sampling, preparation, separation, and identification of microplastics. This methodological heterogeneity limits direct comparability among results, reduces study reproducibility, and hampers the consolidation of reliable quantitative data at regional and global scales (COUCEIRO, 2019; BARRETO, 2024).

Additionally, aggregate formation constitutes a significant challenge, particularly in aquatic environments rich in organic matter. Microplastics may aggregate with each other or with mineral particles and natural organic matter, forming complex structures that alter hydrodynamic behaviour and hinder density-based separation, while also compromising morphological and spectroscopic analyses (RESMINI, 2023).

Interference from surfactants—commonly present in industrial and domestic effluents—further complicates analytical characterization. These compounds may modify surface tension, promote particle emulsification, and interfere with fluorescent staining methods such as Nile Red, increasing the likelihood of false positives or hindering the distinction between microplastics and organic matter (COUCEIRO, 2019; RESMINI, 2023).

Finally, low analytical efficiency for particles smaller than 20 µm represents one of the most pressing current challenges. Widely used techniques such as optical microscopy and conventional FTIR exhibit significant limitations in this size range, requiring the application of more sophisticated methods, such as micro-Raman spectroscopy or Py-GC/MS. Although more precise, these techniques involve higher costs, greater operational complexity, and lower analytical throughput (RESMINI, 2023).

Considering these challenges, the scientific literature emphasizes the need for multi-technique approaches and combined protocols that integrate visual, spectroscopic, thermal, and chemical methods to enhance the reliability of results and reduce analytical uncertainty. International organizations such as UNESCO highlight that methodological harmonization and the development of integrated protocols are essential for advancing research and ensuring consistent evaluation of the occurrence and impacts of microplastics in the environment (UNESCO, 2023).

3.6 Implications for the Selection of Treatment Technologies

Proper characterization of microplastics is a fundamental step in selecting and designing treatment technologies for industrial and environmental effluents. The identification of predominant particle size, morphology (fragments, fibers, films, or pellets), and granulometric distribution allows anticipation of the performance of physical and physicochemical processes, since these properties directly influence removal mechanisms such as sedimentation, flotation, adsorption, and retention by physical barriers (PEREIRA; LOPES, 2021).

Moreover, knowledge of polymer density and hydrodynamic behaviour is essential to understand the tendency of particles to remain in the water column or deposit in sediments, thereby guiding the choice between gravitational processes, dissolved air flotation, or membrane separation systems. Low-density polymers, such as polyethylene and polypropylene, tend to remain suspended for extended periods, requiring technologies capable of capturing floating or dispersed particles (PEREIRA; LOPES, 2021; PAN *et al.*, 2023).

Another critical aspect concerns the interactions between microplastics and natural or anthropogenic organic matter. Biofilm formation and the adsorption of organic compounds alter particle surfaces, modifying hydrophobicity, surface charge, and affinity for specific adsorbent materials. These interactions may either enhance or hinder removal efficiency, depending on the technology employed, making detailed prior characterization indispensable (PAN et al., 2023).

From a technological standpoint, studies indicate that larger particles—particularly fragments and pellets—are removed with relatively high efficiency by conventional coagulation–flocculation processes followed by sedimentation or flotation. These methods promote particle aggregation, increasing effective size and facilitating physical separation (PEREIRA; LOPES, 2021). Conversely, small-sized, highly hydrophobic microplastics exhibit lower removal efficiency in these processes and are more effectively removed by adsorption onto materials such as granular activated carbon

(GAC) or by membrane-based separation technologies, including microfiltration and ultrafiltration (PAN *et al*., 2023).

Resmini (2023) reports that pigmented microplastics, due to the presence of organic dyes, mineral fillers, and additives incorporated into the polymer matrix, display distinct surface behavior compared to non-pigmented particles. In effluents from the paints and coatings industry, polymeric particles associated with pigments showed greater interaction with adsorbent materials, particularly GAC, owing to their increased hydrophobicity and functional groups that promote intermolecular interactions. Complementarily, Pan *et al*. (2023) emphasize that polymer surface characteristics—such as ageing, adsorbed organic compounds, and additives—significantly influence adsorption mechanisms, thereby enhancing retention efficiency in carbonaceous materials. Accordingly, pigmented microplastics tend to exhibit greater affinity for adsorptive surfaces, rendering adsorption processes particularly relevant in industrial effluents from graphic, plastics, and cosmetic sectors (RESMINI, 2023; PAN *et al*., 2023).

In textile effluents, the high load of synthetic microfibers—often associated with dyes, surfactants, and dissolved organic matter—poses additional challenges to conventional treatment. Pan et al. (2023) note that the simultaneous presence of organic compounds and fibrous particles reduces the efficiency of isolated physical separation processes, requiring integrated approaches. Resmini (2023) highlights that the application of advanced oxidation processes, such as ozonation or Fenton processes, may promote the degradation of dissolved organic matter and alter polymer surface characteristics, thereby enhancing subsequent coagulation, filtration, or adsorption stages. Thus, the literature recommends combined systems in which oxidative processes are followed by physical separation steps to maximize microplastic removal in complex matrices (RESMINI, 2023; PAN *et al*., 2023).

More broadly, Pereira and Lopes (2021) emphasize that the selection of effective treatment technologies depends directly on comprehensive effluent characterization, including physicochemical parameters and particle morphology. Pan *et al*. (2023) further reinforce that properties such as density, size, shape, and degree of surface ageing directly influence the performance of coagulation, adsorption, and membrane separation processes. Accordingly, the specialized literature converges on the need for integrated approaches that simultaneously consider the physical, chemical, and hydrodynamic properties of microplastics to optimize removal efficiency in industrial effluents (PEREIRA; LOPES, 2021; PAN *et al*., 2023).

4. EXPERIMENTAL METHODOLOGY

4.1 Study Design

The present study adopted a methodological approach grounded in experimental protocols, aiming to propose a replicable model for the identification of microplastics. The methodological framework was defined based on widely used guidelines in environmental and industrial studies, taking into account the complexity of liquid matrices and the heterogeneity of microplastics in terms of size, shape, and polymer composition (CAMPANALE *et al.*, 2020; PAN *et al.*, 2023).

Conventional physicochemical characterization of the effluent was not included at this methodological stage, as the study's focus is exclusively on the identification and quantification of microplastics, consistent with the approach adopted in several reference studies in the scientific literature.

4.2 Sampling Strategy

The sampling strategy was designed to ensure the representativeness of the analyzed industrial effluent and to minimize contamination from external sources. Samples were collected at strategic points within the production process, previously defined based on the industrial flow diagram, as recommended in studies addressing the characterization of complex effluents (CAMPANALE et al., 2020; PAN et al., 2023).

Collection was performed exclusively using previously decontaminated glass containers, and the use of plastic materials was strictly avoided throughout all procedural stages. This practice is widely recommended in the literature to reduce the introduction of secondary microplastics originating from sampling equipment and utensils (HORTON et al., 2017; MAES et al., 2017).

A total volume of 5 L of effluent was collected from the C-Pack facility, which manufactures polyethylene (PE) packaging for cosmetic applications. The selected volume was defined based on the expected microplastic concentration and the analytical sensitivity of the identification methods employed, in accordance with the procedures described by Couceiro (2019) and Campanale et al. (2020). Samples were stored and analyzed as quickly as possible after collection to preserve their physicochemical characteristics.

4.3 Pretreatment and Separation of Microplastics

Sample pretreatment was performed to remove organic matter, biofilms, and other interfering substances that could compromise the identification and quantification of microplastics. Oxidative chemical digestion using hydrogen peroxide (H_2O_2) is widely recommended due to its high efficiency in degrading organic matter while exerting minimal interference on the structural integrity of polymers such as polyethylene (PE), polypropylene (PP), and polystyrene (PS) (COUCEIRO, 2019; MAES et al., 2017; CAMPANALE et al., 2020).

For the procedure, 1 L of sample was digested with 30% (v/v) H_2O_2 under controlled heating at 40-50 °C, a temperature range considered suitable for accelerating oxidation without promoting thermal deformation of polymers (CAMPANALE et al., 2020; BARRETO, 2024). Heating began at 16:15 and reached 40 °C at 16:40, at which point 100 mL of 30% H_2O_2 was slowly added. The sample was heated and gently agitated for 1 hour, then left at rest for 24 hours to ensure complete oxidation, in accordance with protocols described in the literature (MAES et al., 2017; COUCEIRO, 2019).

After this period, density separation was carried out using a saturated NaCl solution (360 $g \cdot L^{-1}$). A total of 1 L of saturated solution was prepared, achieving an approximate density of 1.2 $g \cdot cm^{-3}$. This method is based on the flotation of lower-density polymers, such as PE (≈ 0.91 to 0.96 $g \cdot cm^{-3}$) and PP (≈ 0.90 to 0.92 $g \cdot cm^{-3}$), which are frequently predominant in industrial effluents from the plastics sector (ANDRADY, 2011; CAMPANALE et al., 2020). The sample was left undisturbed for 28 hours to allow sedimentation of denser fractions.

However, no clear visual separation between supernatant and sedimented phases was observed, possibly due to the high presence of fine particles and residual organic matter. Consequently, the entire sample was filtered through nylon membranes with a pore size of 0.43 µm.

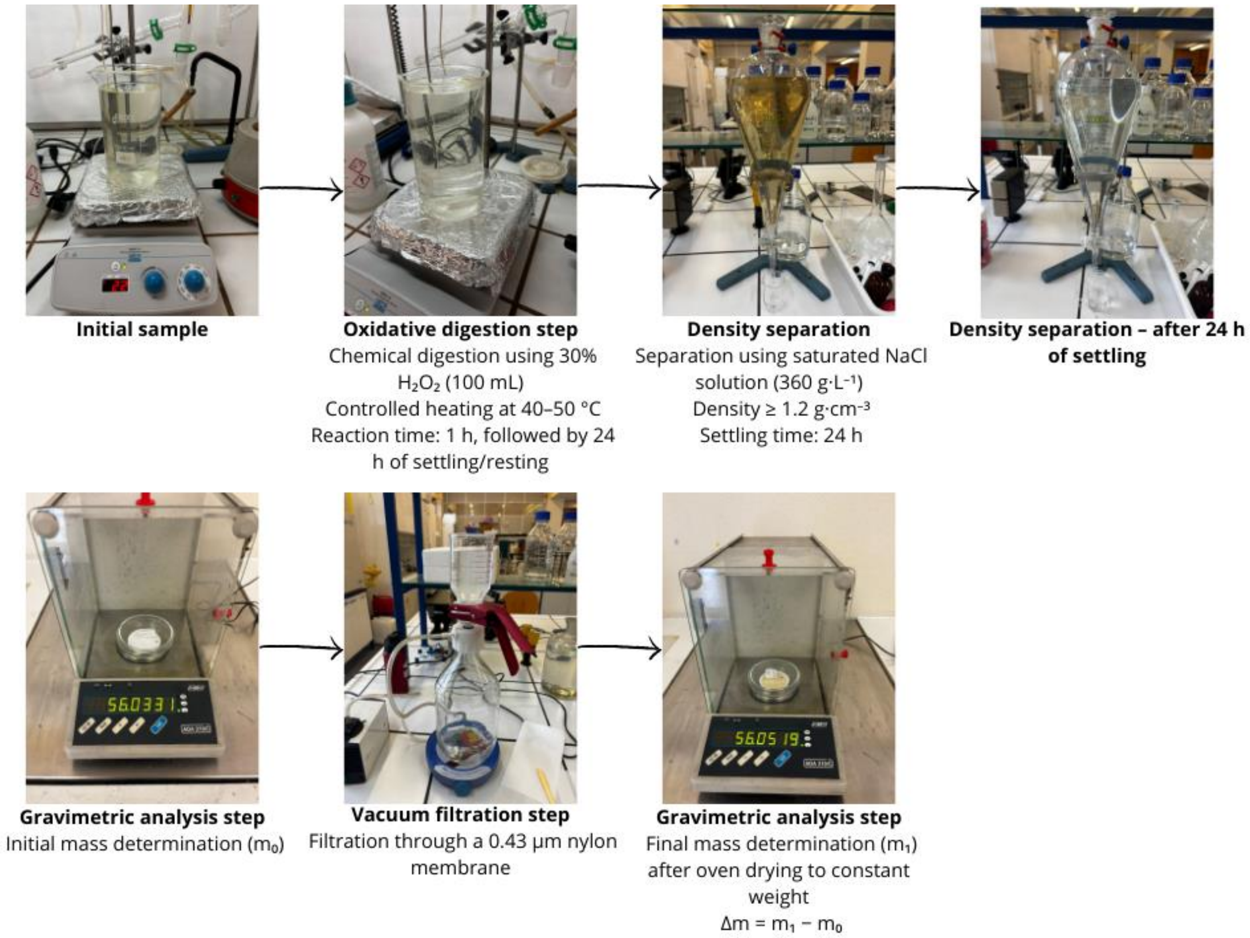
As a gravimetric control and to verify particulate retention, filters were weighed on an analytical balance prior to filtration. After filtration, the filters were dried in an oven at 50 °C until constant mass and weighed again, allowing determination of mass difference and confirmation of significant solid retention on the membrane. A retained mass of 0.0188 g was measured for the analyzed 1 L sample. The use of gravimetric control is recommended as a complementary procedure in microplastic studies, particularly when density separation is not efficient, as it enables estimation of the recovered particulate fraction (COUCEIRO, 2019; BARRETO, 2024)

4.4 Experimental Procedure Flowchart

The experimental procedure for sample pretreatment and gravimetric evaluation was structured into sequential stages: oxidative digestion, density separation, filtration, and mass control of the retained material. The organization of these steps aimed to ensure the removal of organic interferents, the concentration of particles present in the sample, and the quantitative determination of the mass retained on the filter.

The flowchart in Figure 2 summarizes the methodological sequence, highlighting the operational conditions at each stage and the procedure used to calculate gravimetric retention.

Figure 2 – Technical flowchart of the experimental procedure for microplastic analysis in industrial effluent.



Source: Author (2026).

The determination of the mass retained on the filter was performed by gravimetric difference, according to Equation 1:

$$\Delta m = m_1 - m_0$$

where:

Δm = retained mass (g);
m_0 = initial filter mass (g);
m_1 = final filter mass (g).

For the analyzed volume (1 L), the following value was obtained:

$$\Delta m = 0.0188\, g/L$$

corresponding to an estimated concentration of:

$$C = 18.8\, mg/L$$

4.5 Critical Discussion of Gravimetric Results

The gravimetric analysis yielded a retained mass of 0.0188 g for the analyzed 1 L sample, corresponding to an estimated particulate concentration of 18.8 $mg \cdot L^{-1}$. This value is considerably higher than concentrations typically reported in municipal wastewater effluents, which are often expressed in particle counts ranging from 10^1 to 10^4 $particles \cdot m^{-3}$, generally corresponding to much lower mass concentrations when converted to gravimetric terms (TALVITIE et al., 2017; WANG et al., 2020). The elevated value observed in the present study may be attributed to the industrial origin of the effluent, particularly considering the handling of polyethylene (PE) materials in the production process.

It is important to emphasize that gravimetric quantification measures total retained particulate matter and does not discriminate between polymeric particles and other suspended solids. Industrial effluents, especially from plastic transformation processes, may contain polymer fragments, pigment-associated particles, inorganic fillers, and residual additives. Barreto (2024) highlights that gravimetric approaches tend to overestimate microplastic concentrations when applied without complementary spectroscopic validation, particularly in matrices with significant organic or mineral content.

The lack of visible stratification during density separation suggests that part of the particulate fraction either possessed densities close to or above 1.2 $g \cdot cm^{-3}$ or remained stabilized in suspension due to fine particle size and possible surfactant interactions. Pan et al. (2023) report that particles below 20–50 µm frequently exhibit reduced settling efficiency and may remain colloidally dispersed even after extended resting periods. This behavior may explain why complete filtration was necessary and supports the hypothesis that a substantial portion of the retained mass corresponds to fine particulate matter.

When compared with literature values for industrial effluents, the concentration obtained falls within a plausible range for facilities involved in polymer processing, although direct comparisons remain challenging due to methodological variability. Studies emphasize that differences in mesh size, digestion protocol, density solution, and filter pore size significantly affect recovery rates and reported concentrations (COUCEIRO, 2019; PAN et al., 2023). The use of a 0.43 µm membrane in this study likely increased retention of fine particles, contributing to the measured mass.

From an environmental perspective, even moderate concentrations in low-volume discharges may become significant when extrapolated over operational periods. For instance, assuming a monthly discharge of 500 L, the retained mass would correspond to approximately 9.4 $g \cdot month^{-1}$ of particulate material. Over an annual cycle, this would represent more than 110 g of potentially polymeric material entering downstream systems, reinforcing concerns regarding cumulative environmental loading.

Therefore, while the gravimetric result provides a valuable first-order estimate of particulate retention, it should be interpreted cautiously. The absence of polymer-specific confirmation limits definitive attribution to microplastics. Future analytical refinement using FTIR, Raman spectroscopy, or Py-GC/MS would allow differentiation between polymeric and non-polymeric fractions and improve quantitative reliability. Nonetheless, the elevated retained mass observed highlights the relevance of implementing optimized treatment strategies capable of removing fine particulate matter in industrial effluents.

5. METHODS FOR THE REMOVAL OF MICROPLASTICS FROM INDUSTRIAL EFFLUENTS

The removal of microplastics from industrial effluents is a growing challenge for the environmental sector, particularly due to the diversity of particle sizes, shapes, and chemical compositions. Unlike many other contaminants, microplastics exhibit complex physicochemical behaviour, as they may float, remain suspended, or associate with organic matter, emulsions, oils, and pigments, thereby significantly complicating treatment processes (ERIKSEN et al., 2018; BARRETO, 2024). In industrial environments characterized by high concentrations of suspended solids, surfactants, and recalcitrant compounds, conventional treatment methods are often insufficient to remove particles smaller than 20 µm (PAN et al., 2023; WANG et al., 2020).

In response to this scenario, various physical, chemical, physicochemical, and hybrid technologies have been evaluated for the removal of microplastics. This chapter presents the main methods, their efficiencies, limitations, and applicability in industrial contexts, based on experimental studies, recent reviews, and technical reports (BARRETO, 2024; OLIVEIRA; BARBOSA; MENDONÇA, 2023).

The removal of microplastics (MPs) in water and wastewater treatment systems has been widely investigated due to increasing regulatory stringency and growing evidence of environmental impacts (UNESCO, 2023; EUROPEAN UNION, 2024). Applied methods vary in terms of efficiency, cost, operational applicability, and capacity to remove particles of different sizes and characteristics. Table 2 summarizes the most commonly used methodologies, their advantages, and limitations, compiled from technical literature and case studies (BARRETO, 2024; GÜNEȘ et al., 2018).

Table 3 – Methods for the removal of microplastics from water.

Method	Average efficiency	Advantages	Limitations	Reference
Coagulation–Flocculation–Sedimentation	40–55%	Simple, low cost, already applied in DWTPs	Low efficiency for MPs < 20 µm	Cristóvão et al. (2014); Barreto (2024); Pan et al. (2023)
Sedimentation / Clarification	60–67%	Good removal of large particles	Inefficient for very small MPs	Pan et al. (2023)
Sand Filtration	29–85%	Consolidated technology, low cost	Variable efficiency; risk of fine MP breakthrough	Hidayaturrahman & Lee (2019); Enfrin et al. (2019)
Granular Activated Carbon (GAC)	56–98%	High adsorption capacity; effective for MPs and organic micropollutants	Requires regeneration or replacement	Barreto (2024)

Method	Average efficiency	Advantages	Limitations	Reference
Membrane Filtration (MF/UF)	> 90% (MPs > 0.1 µm)	Efficient retention of small particles	High cost and membrane fouling	Talvitie et al. (2017); Pan et al. (2023)
Nanofiltration (NF) / Reverse Osmosis (RO)	~100%	Removes MPs and even nanoplastics	High energy and operational cost	Mintenig et al. (2019); Barreto (2024)
Biological Processes (activated sludge, biofilters)	30–70%	MPs retained via biofilms; applied in WWTPs	MPs accumulate in sludge; risk of reintroduction	Talvitie et al. (2017); Pan et al. (2023)
Standalone Ozonation	Inefficient / may fragment MPs	Oxidizes organic matter and disinfects	May generate smaller MPs and toxic by-products	Resmini (2023)
Pre-ozonation + Sedimentation	~76%	Improves MP aggregation	Does not achieve >90% alone	Pan et al. (2023)
Combined processes (coagulation + membranes or carbon)	> 90%	Higher overall efficiency	Higher cost and operational complexity	Talvitie et al. (2017); Barreto (2024); Pan et al. (2023)

Source: Author's own work, based on the cited authors, 2025.

5.1 Traditional Physicochemical Processes

Physicochemical processes—such as coagulation, flocculation, sedimentation, and filtration—are well-established steps in treatment plants and are frequently used as pretreatment steps for industrial effluents. Larger particles (> 50 µm) tend to respond more effectively to these processes, whereas finer fractions require additional barriers to ensure efficient removal (BARRETO, 2024).

5.1.1 Coagulation

Coagulation operates by neutralizing the surface charges of colloidal particles, typically using aluminium or iron salts, thereby destabilizing the system and facilitating the formation of larger flocs amenable to sedimentation. Cristóvão et al. (2014a) describe that process efficiency is directly related to pH control, coagulant dosage, and matrix characteristics, emphasizing that high concentrations of organic matter and surfactants may compete for active sites, thereby reducing aggregation efficiency.

Complementarily, Cristóvão et al. (2014b) demonstrate that in complex industrial effluents, the presence of dissolved organic compounds interferes with charge neutralization mechanisms and interparticle bridging, requiring operational adjustments to optimize removal performance. Although these studies were not specifically conducted on microplastics, the mechanisms described are applicable to polymeric particles exhibiting colloidal behaviour.

In the specific context of microplastics, Barreto (2024) reports that recent reviews indicate removal efficiencies of 40% to 55% for larger particles, with improved performance for particles between 50 and 500 µm. Pan et al. (2023) further emphasize that coagulation efficiency depends on the nature of the polymer surface, the degree of environmental aging, and the presence of associated organic matter. In pigmented effluents, Resmini (2023) observes that inorganic coagulants may exhibit variable performance, particularly in the presence of high dye and additive loads, requiring careful optimization of dosage and pH to maximize floc formation.

5.1.2 Flocculation

Polymeric flocculants (anionic, cationic, or nonionic) are widely employed as coagulation aids to promote interparticle bridging, increase floc size and density, and, consequently, facilitate sedimentation. In jar test experiments using real effluents and synthetic waters containing microplastics, Jachimowicz et al. (2022) observed that the addition of polymeric flocculants following ferric or aluminium-based coagulants significantly enhanced particle transfer to sludge, with removal gains associated with increased floc size and improved clarification.

Rajala et al. (2020) emphasize that performance strongly depends on particle surface characteristics and matrix composition, including the presence of organic matter and surfactants, which compete for adsorption sites and modify aggregation kinetics. In complex industrial matrices, Resmini (2023) reports that anionic flocculants tend to form denser, structurally more stable flocs that can incorporate synthetic fibers and polymer fragments, although precise dosage control is critical to prevent system restabilization.

Wiśniowska et al. (2024) further demonstrate that excessive polymer dosing may lead to charge reversal and redispersion of hydrophobic particles into the liquid phase, thereby reducing overall process efficiency. Therefore, the selection of flocculant type (anionic, cationic, or nonionic), as well as careful control of pH and optimal dosage, constitutes a critical step in maximizing microplastic removal in industrial treatment systems (RAJALA et al., 2020; JACHIMOWICZ et al., 2022; WIŚNIOWSKA et al., 2024; RESMINI, 2023).

5.1.3 Sedimentation and Flotation

Conventional sedimentation is more efficient for particles with higher density and larger size, particularly those denser than water, which tend to settle under gravity. However, polymers commonly predominant in industrial effluents, such as polyethylene (PE) and polypropylene (PP), have densities lower than that of water (≈0.90–0.96 $g \cdot cm^{-3}$), favoring their persistence at the surface or within the water column and consequently reducing removal efficiency by simple decantation (ANDRADY, 2011; HORTON et al., 2017).

Given this limitation, flotation processes are generally more suitable for removing hydrophobic particles. Pan et al. (2023) highlight that in dissolved air flotation (DAF), microbubbles adhere to the surfaces of polymeric particles, increasing their buoyant force and promoting their removal at the reactor surface. The authors emphasize that process efficiency depends on particle hydrophobicity, bubble size, and operational conditions such as pH and coagulant dosage.

In industrial matrices with high concentrations of oils, greases, and hydrophobic organic compounds, Resmini (2023) reports that combining coagulants with surfactant-based auxiliaries can enhance the formation of stable aggregates and increase particle adhesion to air bubbles, thereby improving flotation performance. In such cases, interactions among microplastics, oil droplets, and air bubbles promote more efficient removal compared to sedimentation alone.

5.1.4 Conventional Filtration

Sand or granular media filtration is a well-established technology, characterized by low operational cost and widespread application in water and wastewater treatment plants. Barreto (2024) emphasizes that the removal efficiency of microplastics by granular filtration depends strongly on the grain size of the filter media, the filtration velocity, and the particle size distribution in the effluent. In field studies compiled by the author, removal rates ranged approximately from 29% to 85%, reflecting differences in operational conditions and liquid matrix composition.

Pan et al. (2023) further note that larger particles are more likely to be retained by straining and interception mechanisms, whereas fine particles and fibers may pass through the filter bed, particularly under high dissolved organic matter loads. Campanale et al. (2020) also indicate that process efficiency is influenced by particle morphology and by the presence of biofilms within the filter bed, which may either enhance or reduce retention depending on operational conditions.

In industrial applications characterized by significant concentrations of pigments, resins, and hydrophobic compounds, Resmini (2023) reports that filtration using low-porosity cartridges followed by a polishing stage with granular activated carbon (GAC) tends to achieve superior performance compared to granular filtration alone. The author highlights that the combination of physical retention and surface adsorption enhances the removal of fine particles and microplastics associated with organic compounds.

5.2 Granular Activated Carbon (GAC) Filtration

Granular activated carbon (GAC) filtration has been investigated as a complementary polishing technology for the removal of microplastics from industrial effluents and wastewater, primarily due to its combination of physical retention and surface adsorption mechanisms. Barreto (2024), in a systematic review of removal technologies, describes that GAC operates through both mechanical mechanisms—particle entrapment within the interstitial spaces of the bed—and hydrophobic interactions and adsorption forces on the highly porous carbon surface. Pan et al. (2023) further note that the hydrophobic nature of many polymers enhances interaction with carbonaceous surfaces, particularly when organic matter is associated with the particles.

The performance of GAC is directly related to the physicochemical characteristics of microplastics. Barreto (2024) emphasizes that size, morphology, and the degree of surface aging influence removal efficiency, with particles in the 20–100 µm range being more readily retained compared to ultrafine fractions. Pan et al. (2023) report that, under appropriate hydraulic contact time and controlled filtration velocity, removal efficiencies exceeding 90% may be achieved for specific size ranges, particularly when microplastics are associated with dissolved or particulate organic matter that mediates adsorption.

From an operational perspective, parameters such as contact time, carbon granulometry, and bed saturation state are critical. Campanale et al. (2020) highlight that adsorbent materials with higher specific surface area enhance the retention of hydrophobic compounds, while Pan et al. (2023) stress that longer contact times promote diffusion and particle–surface interaction. However, finer carbon granulometry may increase head loss, requiring hydraulic optimization.

Resmini (2023), evaluating effluents from the paints and coatings industry, observed that pigmented microplastics and particles associated with resins exhibit a greater affinity for GAC due to adsorbed organic compounds and increased surface hydrophobicity. The author emphasizes that in complex industrial systems, GAC filtration is more effective when applied as a final polishing stage following coagulation–flocculation and sedimentation, enabling additional removal of fine particles and residual pigments.

Despite the high efficiencies reported, Barreto (2024) notes that progressive bed saturation reduces adsorption capacity over time, particularly under high organic loads. Pan et al. (2023) further emphasize that periodic regeneration or replacement of the carbon is essential to maintain performance, as occupation of active sites compromises the retention of hydrophobic particles.

Overall, the studies compiled by Barreto (2024) and the experimental analyses discussed by Pan et al. (2023) indicate that GAC filtration constitutes a robust technology for microplastic removal in complex effluents, particularly when integrated into multistage treatment systems.

5.2.1 Limitations of Granular Activated Carbon for Microplastic Removal

Although granular activated carbon (GAC) is widely recognized for its high efficiency in the removal of dissolved organic micropollutants, its applicability for microplastic removal presents important limitations, particularly when considering particle size and concentration levels.

From an analytical and operational perspective, GAC exhibits greater efficiency in the adsorption of compounds at concentrations in the order of nanograms per liter ($ng \cdot L^{-1}$), being especially effective for low molecular weight dissolved organic substances. In contrast, microplastics are particulate contaminants, often present at higher concentrations (typically in the order of micrograms per liter – $\mu g \cdot L^{-1}$ or above), for which removal mechanisms rely primarily on physical retention rather than true adsorption processes.

Furthermore, microplastics generally present dimensions and surface characteristics that hinder diffusion into the microporous structure of activated carbon, thereby reducing adsorption efficiency. In complex industrial effluents, the presence of natural organic matter, surfactants, oils, and suspended solids may compete for active adsorption sites, further decreasing process performance.

Therefore, the use of GAC as a standalone technology for microplastic removal is considered technically limited, particularly for particulate fractions. Its application is more suitable as a polishing step within integrated treatment systems, especially following processes such as coagulation–flocculation, filtration, or membrane separation, which are more effective for the removal of suspended particles.

5.3 Membrane Processes (MF, UF, NF, and RO)

Membrane separation processes have become established as highly effective approaches for the removal of microplastics (MPs) and nanoplastics (NPs), as they operate through selective physical barriers in which size-exclusion mechanisms predominate, along with electrostatic and hydrophobic interactions between particles and the membrane surface. Wang et al. (2020) note that microfiltration (MF), ultrafiltration (UF), nanofiltration (NF), and reverse osmosis (RO) present distinct retention ranges and may be applied complementarily along treatment trains. Pan et al. (2023) further emphasize that technology selection depends on particle size distribution and matrix complexity.

MF and UF are widely employed for the retention of micrometric and submicrometric particles. Pan et al. (2023) report that when preceded by pre-treatment steps such as coagulation, flocculation, or granular filtration, these technologies can achieve removal efficiencies exceeding 90% for MPs $\geq \sim 0.1\ \mu m$, significantly reducing particulate loads in the permeate. Wang et al. (2020) highlight that, beyond size exclusion, polymer hydrophobicity may promote retention via adsorption onto membrane surfaces, particularly in polymeric membranes.

For smaller particles, including nanoplastics, NF and RO demonstrate superior performance. Wang et al. (2020) show that these technologies can achieve near-complete removal of NPs by combining physical barrier mechanisms with rejection of organic solutes and ions—an attribute particularly relevant in water reuse systems and complex industrial effluents. However, the authors note that distinguishing between purely physical retention and surface adsorption remains an ongoing area of investigation.

Despite their high efficiencies, membrane fouling constitutes the primary operational constraint. Pan et al. (2023) report that the accumulation of organic matter, oils, suspended solids, and biofilms on membrane surfaces or within pores increases head loss and reduces permeate flux, thereby increasing the frequency of chemical cleaning. Wang et al. (2020) add that the hydrophobic nature of microplastics intensifies adhesion to polymeric membrane surfaces, exacerbating fouling and impacting operational costs and membrane lifespan.

Another critical aspect concerns the management of concentrate (retentate) streams in NF and RO systems. Wang et al. (2020) emphasize that the retentate concentrates salts, organic matter, and plastic particles, posing an additional environmental challenge regarding disposal or subsequent treatment. Pan et al. (2023) reinforce that the adoption of membrane technologies for MP removal must be accompanied by integrated strategies for concentrate management, otherwise merely transferring the pollutant load to another stream.

Overall, as discussed by Wang et al. (2020), Pan et al. (2023), and Campanale et al. (2020), membrane technologies are among the most effective approaches for removing micro- and nanoplastics, particularly when integrated with pretreatment stages that minimize fouling and stabilize influent matrix characteristics.

5.4 Advanced Oxidation Processes (AOPs)

Advanced Oxidation Processes (AOPs) have been extensively investigated for the treatment of industrial effluents due to their high capacity to degrade recalcitrant organic matter and remove dyes, primarily through the in situ generation of highly reactive species, such as hydroxyl radicals (•OH).

Güneş et al. (2018) describe that processes such as ozonation, hydrogen peroxide oxidation, and Fenton reactions promote non-selective oxidation of organic compounds and are particularly effective in degrading dyes and aromatic compounds present in complex industrial effluents.

In the context of microplastics, AOPs do not primarily function as physical separation mechanisms but play a relevant role in modifying particle surfaces and removing associated organic interferents. Barreto (2024) emphasizes that partial oxidation of biofilms and adhered organic matter may reduce interference in subsequent treatment stages, such as coagulation–flocculation, granular filtration, and membrane processes. Pan et al. (2023) further highlight that reducing the influent organic load contributes to minimizing membrane fouling and improving overall treatment efficiency.

However, the isolated application of AOPs may produce adverse effects. Güneş et al. (2018) demonstrate that prolonged exposure of polymers to oxidizing species promotes surface cracking, increased roughness, and progressive fragmentation, resulting in the formation of smaller particles. Barreto (2024) notes that this process may increase the total number of microplastics and favor the formation of nanoplastics, which exhibit greater bioavailability and are more difficult to remove using conventional technologies.

Beyond physique fragmentation, Campanale et al. (2020) indicate that partial polymer oxidation may generate oxygen-containing functional groups on particle surfaces, altering chemical properties and increasing particle reactivity. Güneş et al. (2018) also discuss that oxidation by-products of organic matter—such as aldehydes, ketones, and low-molecular-weight organic acids—may remain in the effluent, requiring complementary treatment steps.

Specifically regarding ozonation, Güneş et al. (2018) report that interactions with polymers such as polyethylene and polypropylene occur predominantly at the surface, leading to structural weakening without complete mineralization. Similarly, Barreto (2024) observes that Fenton and photo-Fenton processes significantly alter the surface morphology of microplastics, potentially facilitating subsequent removal by coagulation or filtration, although they may also increase the risk of fragmentation.

5.5 Adsorption Using Biochar and Modified Adsorbents

Adsorption using biochar and modified carbonaceous adsorbents derived from lignocellulosic residues has been investigated as a technically viable and environmentally sustainable alternative for removing microplastics (MPs) and associated contaminants from aqueous matrices. Li and Shi (2022) emphasize that interest in these materials stems from their low production costs, the valorisation of agro-industrial residues, and the high specific surface area achieved through controlled pyrolysis, which promotes the formation of a highly porous structure.

The removal mechanism involves a combination of hydrophobic interactions, Van der Waals forces, electrostatic interactions, and physical entrapment within the material pores. Li and Shi (2022) demonstrate that hydrophobic polymeric particles, such as polyethylene (PE) and polypropylene (PP), exhibit strong affinity for carbonaceous surfaces. Oliveira (2023) further notes that microplastics coated with dissolved organic matter or associated with persistent organic contaminants display an even greater tendency toward adsorption due to increased surface hydrophobicity.

Several studies indicate that adsorptive performance can be enhanced through physical and chemical modifications. Li and Shi (2022) report that biochar doped with metals, such as iron, acquires magnetic properties that facilitate post-treatment recovery and increase the availability of active sites. Nitrogen doping or surface treatments also increase the density of functional groups, promoting chemical interactions with micropollutants associated with MPs. Pan et al. (2023) reinforce that surface modifications expand removal efficiency in complex aqueous matrices.

In experimental assays compiled by Li and Shi (2022), removal efficiencies exceeding 70%–80% were observed, depending on pyrolysis temperature, granulometry, and biochar surface area. Oliveira (2023), in an applied experimental study, demonstrated that modified biochar exhibited significantly superior performance compared to untreated materials, particularly in the removal of fine microplastics and particles associated with dissolved organic matter.

Among the advantages of this approach are the potential reuse of agricultural residues and the flexibility to tailor surface properties. Barreto (2024) highlights that these characteristics make the technology attractive as a complementary polishing step in integrated treatment systems. However, Campanale et al. (2020) and Oliveira, Barbosa, and Mendonça (2023) identify limitations, including a finite adsorption capacity and the need to regenerate or dispose of saturated material in an environmentally appropriate manner.

Overall, as discussed by Li and Shi (2022), Oliveira (2023), and Pan et al. (2023), adsorption using modified biochar demonstrates high potential for microplastic removal, particularly when integrated with physicochemical or membrane-based processes, contributing to more sustainable and efficient treatment chains.

5.6 Removal by Hybrid Treatment Systems

Hybrid treatment systems, integrating complementary physicochemical and physical–mechanical processes, have been highlighted as an effective strategy for removing microplastics from complex industrial matrices. Pan et al. (2023) emphasize that standalone technologies often exhibit limitations when applied to effluents with high organic loads, the presence of surfactants, and hydrophobic particles. The combination of mechanisms—such as aggregation, physical retention, adsorption, and oxidation—is therefore essential to enhance overall performance and mitigate adverse effects, including fouling and overloading of final treatment stages.

Among the most frequently reported configurations is the coagulation–flocculation-granular activated carbon (GAC) filtration association. Oliveira (2023), in an experimental application, demonstrated that coagulation increases the effective size of fine microplastics and colloidal particles, while GAC acts as a polishing step, removing residual particles and associated organic compounds. Barreto (2024) emphasizes that this integration yields significant efficiency gains over isolated processes, particularly in effluents with high dye concentrations and organic matter loads.

Another widely investigated approach involves integrating microfiltration or ultrafiltration (MF/UF) with adsorption processes using biochar or modified adsorbents. Pan et al. (2023) report that the adsorptive stage reduces dissolved organic matter and hydrophobic compound concentrations, thereby decreasing the propensity for membrane fouling, while MF/UF ensures physical retention of remaining microplastics. The authors indicate that hybrid configurations can maintain removal efficiencies above 90% for micrometric particles while providing greater operational stability over time.

The combination of advanced oxidation processes (AOPs) and ultrafiltration has also been reported as an effective alternative. Barreto (2024) notes that AOPs contribute to the degradation of recalcitrant organic matter and the removal of biofilms, thereby reducing the organic load entering membrane systems. Pan et al. (2023) demonstrate that this integration helps mitigate fouling and improve hydraulic performance, particularly in industrial effluents characterized by high chemical complexity.

In systems with high concentrations of suspended solids and low-density particles, Oliveira (2023) describes that the association of dissolved air flotation (DAF) with membrane processes promotes preliminary removal of floating microplastics and aggregates, significantly reducing the particulate load reaching the membranes and extending their operational lifespan. Similarly, Resmini (2023) observes that combinations of adsorption and sedimentation are effective in capturing particles associated with particulate organic matter in pigmented industrial effluents.

Overall, Pan et al. (2023) and Barreto (2024) indicate that hybrid approaches frequently achieve overall removal efficiencies exceeding 90% in complex industrial effluents, where the simultaneous presence of microplastics, organic matter, oils, and additives compromises the performance of isolated technologies. However, Oliveira (2023) emphasizes that such systems entail greater operational complexity and higher capital costs, stemming from the integration of multiple treatment units and increased process control requirements. Nevertheless, the gains in robustness, stability, and efficiency tend to offset additional costs in medium- and large-scale applications.

5.7 Overall Comparison of Technologies

The integrated analysis of the scientific literature and pilot- and industrial-scale case studies indicates that the performance of microplastic removal technologies is strongly conditioned by effluent characteristics and by the objectives of the treatment system. In general, granular activated carbon (GAC) filtration and membrane processes stand out for their high overall removal efficiencies, particularly when used as polishing stages or in water reuse systems. These technologies demonstrate high capacity for retaining fine particles and microplastics associated with organic matter and micropollutants, provided that their implementation is technically and economically feasible within the operational context (BARRETO, 2024; PAN et al., 2023).

Conversely, coagulation–flocculation remains a widely adopted and effective technology for the removal of larger particles, acting by aggregating microplastics and suspended solids, thereby facilitating separation by sedimentation or flotation. However, the literature highlights significant limitations of this process for particles smaller than 20 µm, for which removal efficiency tends to decrease, particularly in chemically complex matrices or in the presence of surfactants (BARRETO, 2024).

Advanced oxidation processes (AOPs), although frequently investigated in wastewater treatment contexts, are not sufficient for the direct removal of microplastics when applied in isolation. Their primary contribution lies in pre-treatment, promoting the degradation of recalcitrant organic matter, biofilm removal, and surface modification of plastic particles, thereby enhancing the efficiency of subsequent physical separation or adsorption stages. Nevertheless, the isolated application of these processes may result in fragmentation of larger particles, reinforcing the need for integration with complementary technologies (PAN et al., 2023).

Adsorption using biochar and alternative adsorbents derived from lignocellulosic residues has been identified as a sustainable and promising alternative, particularly due to lower cost, waste valorisation potential, and the possibility of tailoring adsorbent surface properties. Experimental studies indicate favourable performance in the removal of microplastics and associated contaminants, especially when this technology is integrated with physical or membrane processes, thereby enhancing overall system efficiency (LI; SHI, 2022; OLIVEIRA, 2023).

In this context, hybrid treatment systems combining complementary technologies emerge as the most robust solutions for treating complex industrial effluents. Process integration enables compensation for individual limitations, reduces adverse effects such as fouling, and enhances operational stability, often resulting in efficiencies superior to those achieved with standalone technologies. On the other hand, such systems require higher initial investment, greater operational complexity, and specialized technical expertise—factors that must be carefully evaluated during the design phase (PAN et al., 2023; OLIVEIRA, 2023).

Overall, the selection of the most appropriate technology for microplastic removal depends on a series of interdependent factors, including effluent composition, predominant particle size, the presence of surfactants, pigments, and organic matter, the final treatment objective—whether discharge into receiving water bodies or water reuse—and the economic feasibility of the system. In this regard, the literature and technical documents strongly recommend conducting cost–benefit analyses and pilot-scale studies prior to full-scale industrial implementation to ensure the technical efficiency, economic sustainability, and environmental compliance of the proposed treatment system (TAE MAGAZINE, 2019; EUROPEAN UNION, 2024).

6. INTEGRATED DISCUSSION AND COMPARISON OF MICROPLASTIC REMOVAL TECHNOLOGIES

The integrated analysis of microplastic removal technologies applied to industrial effluents reveals a complex and multifactorial scenario. Despite significant advances in adsorption, advanced filtration, oxidative processes, and hybrid methods, removal efficiency remains strongly dependent on the particles' physicochemical characteristics and the properties of the effluent matrix. Factors such as density, hydrophobicity, size, shape, and polymer composition interact with operational variables and with effluent constituents—including surfactants, solvents, dyes, recalcitrant organic matter, and high concentrations of suspended solids—ultimately determining the actual performance of treatment systems (BARRETO, 2024; PAN et al., 2023; WANG et al., 2020).

Therefore, discussing the efficiency and limitations of each technology individually is essential to guide design decisions and industrial applications, as technical selection must account for both particle behaviour and the specific parameters of the matrix to be treated (PEREIRA; LOPES, 2021).

6.1 Critical Comparison of Technologies

A critical evaluation of the technologies discussed in the previous sections reveals significant variability in removal efficiencies, operational complexity, and applicability to industrial effluents containing microplastics. The comparative analysis presented in Table 4 synthesizes reported performance ranges, main advantages, and inherent limitations of each treatment approach, providing a structured basis for assessing their technical feasibility in industrial scenarios.

Table 4 – Comparison of microplastic removal efficiencies.

Technology	Overall Efficiency	Advantages	Limitations	Reference
Coagulation–Flocculation	40–60%	Low cost, easy application	Low efficiency for particles < 50 µm	Cristóvão et al. (2014); Pan et al. (2023)
Sedimentation / Flotation	Moderate	Effective for dense or hydrophobic particles	Limited performance in effluents with surfactants	Talvitie et al. (2017)
Granular Filtration	30–85%	Simple and cost-effective	Low efficiency for smaller particles	Hidayaturrahman & Lee (2019)
Granular Activated Carbon (GAC)	70–98%	High efficiency and final polishing	Filter media saturation	Barreto (2024)
Membranes (MF/UF/NF/RO)	~100%	Near-complete removal	High fouling and cost	Talvitie et al. (2017); Pan et al. (2023)

Technology	Overall Efficiency	Advantages	Limitations	Reference
AOPs (Advanced Oxidation Processes)	Low (direct)	Reduces organic load	Fragments MPs, requiring post-treatment	Resmini (2023)
Adsorption with Biochar	60–80%	Sustainable and low-cost	Efficiency depends on modification	Pan et al. (2023)
Hybrid Processes	High	More robust and efficient	Higher operational complexity	Talvitie et al. (2017); Pan et al. (2023); Barreto (2024)

Source: Author’s own work, based on the cited authors, 2025.

Coagulation–flocculation remains one of the most widely applied technologies in effluent treatment due to its operational simplicity, low cost, and broad availability of chemical reagents. In the context of microplastic removal, this process promotes charge neutralization and particle aggregation, increasing effective particle size and facilitating subsequent separation stages. Studies indicate satisfactory performance with larger particles, particularly those exceeding 50 µm, which justifies its frequent use as a pretreatment step. However, coagulation–flocculation efficiency decreases significantly for smaller particles—especially those below 50 µm—thereby limiting its effectiveness as a standalone technology for fine microplastics (CRISTÓVÃO et al., 2014; BARRETO, 2024).

Sedimentation and flotation processes are widely used as complementary stages and are suitable for removing particles of higher density or those with hydrophobic properties. Flotation has demonstrated effectiveness for low-density microplastics associated with flocs or air bubbles. Nevertheless, both processes are sensitive to the presence of surfactants and detergents commonly found in industrial effluents, which may impair stable floc formation and reduce separation efficiency.

Granular filtration stands out as an economical, operationally simple alternative, employed either as an intermediate stage or as a preliminary polishing step. Its efficiency in removing microplastics varies substantially with filter media granulometry, filtration velocity, and influent solids load. Although it performs well for larger particles, its capacity to retain fine microplastics is limited when compared to more advanced technologies.

Granular activated carbon (GAC) filtration has been widely reported as one of the most effective technologies for polishing final effluent, combining physical retention with surface adsorption. GAC demonstrates high efficiency in removing microplastics associated with organic matter and micropollutants, particularly within intermediate size ranges. Its main limitation lies in the need to periodically regenerate or replace the carbon bed and properly manage saturated material, which increases operational costs (BARRETO, 2024).

Membrane processes—including microfiltration, ultrafiltration, nanofiltration, and reverse osmosis—are among the most efficient technologies available, capable of achieving near-total retention of microplastics and, in some cases, nanoplastics. Despite this high performance, their application is constrained by elevated capital and operational costs, as well as recurrent fouling issues associated with the accumulation of polymers, organic matter, and biofilms on membrane surfaces.

Advanced oxidation processes (AOPs), such as ozonation and Fenton-based systems, are not suitable for direct microplastic removal when applied in isolation. Their primary contribution lies in pre-treatment, promoting degradation of organic matter, biofilm removal, and surface modification of plastic particles, which may enhance subsequent physical separation stages. However, the literature warns of the risk of fragmentation of macro- and microplastics when these processes are used alone, potentially increasing the number of smaller particles in the treated effluent.

Adsorption using biochar and other adsorbents is a sustainable, relatively low-cost approach, particularly when agro-industrial residues are used as raw materials. Process efficiency strongly depends on the physical and chemical modification of the adsorbent material, with improved performance reported for functionalized or doped biochar that interacts more effectively with hydrophobic particles and associated micropollutants.

Finally, hybrid systems combining two or more complementary technologies have proven to be the most robust and efficient solutions for complex industrial effluents. Process integration enables compensation for individual limitations, reduces adverse effects such as fouling, and enhances operational stability, frequently resulting in high overall removal efficiencies. Conversely, such systems require greater operational complexity, higher capital investment, and specialized technical expertise—factors that must be carefully considered during the design and decision-making phases (TAE MAGAZINE, 2019; BARRETO, 2024).

7. INDUSTRIAL APPLICABILITY AND PROPOSED TREATMENT STRATEGY

7.1 Integration of Analytical Results into Industrial Decision-Making

The characterization of microplastics in industrial effluents constitutes a strategic tool for decision-making, as it enables understanding of particle behaviour within treatment systems and their interaction with different removal technologies (PAN et al., 2023; BARRETO, 2024).

In the present study, due to the low particle concentration detected in the analyzed samples, it was not possible to identify the polymer using spectroscopic techniques. Therefore, the technical discussions and proposed solutions were grounded in specialized scientific literature, considering the polymers most frequently associated with the evaluated industrial sector and the physicochemical parameters characterized in the effluent.

Although direct identification of the polymer type was not achieved, integrating experimentally obtained data with evidence reported in the literature provides a consistent basis for technical decision-making aligned with emerging contaminant control principles and increasingly stringent regulatory requirements (PAN et al., 2023; EUROPEAN UNION, 2024).

7.2 Criteria for Selecting Microplastic Removal Technologies

The selection of technologies for microplastic removal must consider characteristics such as particle density, morphology, size distribution, and interaction with dissolved organic matter, as extensively discussed by Pan et al. (2023) and Barreto (2024).

Low-density polymers, such as polyethylene (PE) and polypropylene (PP)—frequently predominant in effluents from the plastics industry—tend to remain suspended or float, thereby exhibiting higher removal efficiency through processes such as dissolved air flotation, granular filtration, and adsorption onto granular activated carbon (PAN et al., 2023).

Microplastics associated with pigments or organic additives demonstrate greater affinity for adsorptive surfaces, favouring the use of granular activated carbon or modified biochar as complementary polishing stages (RESMINI, 2023; OLIVEIRA; BARBOSA; MENDONÇA, 2023).

For smaller particles, particularly those below 50 µm, technologies based on physical barriers—such as microfiltration and ultrafiltration—are recommended when greater retention control or water reuse is required (WANG et al., 2020; PAN et al., 2023).

7.3 Proposed Treatment Strategy Suitable for Industrial Scale C-Pack

The gravimetric analysis performed after oxidative digestion, density separation, and filtration indicated a retained mass of 0.0188 g on the filter, corresponding to the analyzed sample volume of 1 L. Based on this value, the concentration of retained particulate material was estimated according to Equation 1:

$$C = \frac{\Delta m}{V} \quad (1)$$

where:

C = estimated concentration ($mg \cdot L^{-1}$);
Δm = mass retained on the filter (mg);
V = sample volume (L).

Substituting the obtained values:

$$C = 0.0188\, g/L$$

Converting to milligrams:

$$C = 18.8\, mg/L$$

Considering the monthly volume currently generated by the industrial unit (approximately 500 $L \cdot month^{-1}$), the estimated monthly load of retained particulate material is calculated as:

$$\boldsymbol{Mmonthly = 18.8\, mg/L \times 500\, L}$$

$$\boldsymbol{Mmonthly = 9400\, mg \approx 9.4\, g/month}$$

Thus, the potential generation is estimated at approximately 9.4 g of particulate material per month, representing a relatively low load when compared to larger-scale industrial systems.

In light of these results, the adoption of a decentralized and simplified treatment strategy compatible with the operational scale of C-Pack is recommended, prioritizing compact solutions, ease of integration into the production routine, and reduced operational costs.

Initially, a storage and equalization tank with a capacity between 600 and 800 L is proposed, allowing batch treatment operation. This configuration is particularly suitable for low monthly volumes, avoiding the need for complex continuous systems while enabling improved operational control.

Subsequently, an initial physical filtration step—such as fine screening or cartridge filtration—is recommended to remove larger fragments and visible particles, thereby reducing the solids load and preventing clogging of downstream units.

Considering the predominance of low-density polymers, such as polyethylene (PE) and polypropylene (PP), in plastics industry effluents, a simplified gravitational separation or flotation stage is suggested. This step would favour the removal of hydrophobic particles that tend to remain suspended or float.

As a final polishing stage, granular activated carbon (GAC) filtration may be considered as a complementary treatment step. This technology is particularly suited for low flow rates and operates through a combination of physical retention and surface adsorption mechanisms. However, its effectiveness is predominantly associated with the removal of dissolved organic compounds rather than particulate contaminants. In this context, GAC presents limited efficiency for microplastic removal, especially for particles in the micrometric range, and should therefore be applied as a support unit for the removal of organic matter, additives, and associated compounds rather than as a primary barrier for microplastics.

The estimated monthly load below 10 g reinforces that, under the current operational scenario, highly complex technologies—such as high-pressure membrane systems—may not be strictly necessary. A sequential arrangement comprising physical retention, gravitational separation, and filtration processes is technically sufficient, economically feasible, and aligned with the productive scale of the industrial unit. In this configuration, adsorption processes such as GAC may be incorporated as a polishing step focused on dissolved micropollutants, contributing to overall treatment performance without being relied upon for particulate removal.

7.4 Operational and Regulatory Implications

The industrial implementation of the strategies proposed in this study directly contributes to compliance with European environmental guidelines aimed at controlling emerging contaminants, including microplastics. Although specific legal limits for microplastics in industrial effluents have not yet been established, regulatory trends indicate a progressive strengthening of precautionary principles and ecosystem protection criteria, particularly concerning aquatic environments (EUR-LEX, 2014; EUROPEAN UNION, 2024).

Furthermore, incorporating analytical data into industrial environmental management systems enables continuous monitoring of treatment efficiency, identification of critical points in microplastic generation, and the implementation of preventive actions within production processes. Such measures help reduce operational costs and mitigate long-term environmental risks (BARRETO, 2024).

8. FINAL CONSIDERATIONS

The present study enabled a comprehensive analysis of the occurrence, characterization, and removal of microplastics in industrial effluents, integrating both literature review and experimental evaluation. The results reinforce that microplastics constitute a persistent and complex class of emerging contaminants, characterized by high morphological variability, resistance to degradation, and strong interaction with other pollutants present in industrial matrices.

The experimental findings indicated a retained mass of 0.0188 $g \cdot L^{-1}$, corresponding to an estimated concentration of 18.8 $mg \cdot L^{-1}$, highlighting the potential contribution of industrial activities—particularly polymer processing—to the release of particulate material into aquatic systems. However, it is important to emphasize that gravimetric analysis does not allow differentiation between polymeric and non-polymeric particles, which may lead to overestimation of actual microplastic concentrations in complex effluents.

Regarding treatment technologies, the analysis of available methods demonstrated that conventional physicochemical processes, such as coagulation–flocculation and filtration, present moderate efficiency for larger particles but are limited in the removal of fine microplastics, especially those below 20 µm. Advanced technologies, particularly membrane filtration processes, show higher removal efficiencies, although associated with higher operational costs and maintenance requirements.

In this context, although granular activated carbon (GAC) is widely recognized for its effectiveness in the removal of dissolved micropollutants, its applicability for microplastic removal is limited. The process is more efficient for contaminants present at nanogram-level concentrations and in dissolved form, whereas microplastics are particulate contaminants often present at higher concentrations. Consequently, the role of GAC in microplastic removal should be understood primarily as a complementary polishing step, rather than a standalone solution.

The results and literature analysis indicate that the most effective approach for microplastic removal in industrial effluents lies in the implementation of integrated treatment systems, combining coagulation–flocculation, physical filtration, and advanced separation processes. Such combinations enhance overall removal efficiency and allow the treatment system to address both particulate and dissolved contaminants.

From an industrial and environmental perspective, even relatively low-volume effluent discharges may represent significant cumulative loads of particulate material over time, reinforcing the importance of implementing adequate treatment strategies. Furthermore, the absence of specific regulatory limits for microplastics in both Brazilian and European frameworks highlights the need for advancements in environmental legislation and monitoring protocols.

Finally, this study contributes to the understanding of microplastic behavior in industrial effluents and emphasizes the need for methodological standardization and the application of complementary analytical techniques, such as FTIR, Raman spectroscopy, or Py-GC/MS, to improve the accuracy of identification and quantification. Future research should focus on the optimization of integrated treatment systems and the evaluation of emerging technologies capable of effectively removing fine and nanometric plastic particles.

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