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Article

Study of Polyurethane Microplastics Removal from Water Using Smart Instalation

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Abstract

Microplastics, plastic particles smaller than 5 mm in size, have become a contaminant of priority concern in the environment. Microplastic pollution is a significant environmental challenge, highlighting the need for improved water treatment methods. This study investigates the removal of two fractions of polyurethane microplastics ranging in size from smaller than 100 μm, D1, and in range 200 μm - 500 μm, D2, from aqueous synthetic solutions having a concentration of 0.2 g/L, around 175 NTU. In the first stage of the study, tests were performed to identify the optimal doses of efficient reactive agents for microplastic removal, using the classical method: the Jar test. At this stage, attention was directed towards analyzing the variation of turbidity and their removal efficiency in the presence of classical coagulants such as aluminum sulfate, SA, ferrous sulfate, SF, aluminum polychloride, PA; aloe vera flocculant; and activated carbon, CA of the Norit GAC 830 W type. The classical coagulants such as aluminum sulphate, ferrous sulphate have a good efficiency on microplastic removal, which can provide a residual turbidity in range of 6-10 NTU after a retention time of 50 - 60 minutes. In the second stage of the study, the efficiency of smart decantation-filtration system, DFS, was determined. The efficiency of decanter was studied using **Response Surface Methodology (RSM)** for identification of the mathematical models necessary to evaluate the effects of key process variables: Flow rate (A), Microplastic size (B), and Aluminum sulphate concentration (C) on microplastic removal efficiency. The sedimentation can raise the optimal value of 98.98% at the outlet of the decanter. Microplastics in D1 and D2 sized synthetic solutions can be removed from contaminated water by decantation and filtration, the efficiency is around maximum permissible limit, MPL, values of 1 NTU.

Keywords: microplastics; polyurethane foam; turbidity; removal efficiency; Jar test; sedimentation; coagulation; filtration

1. Introduction

Globally, the demand and manufacturing of plastics has shown significant growth. Plastics have become an integral part of today's civilization, due to their convenience, accessibility and functional properties. Their use is driven by several variables such as population development, increasing urbanization, and advances in technology [1].

However, the intense demand and accelerated production rate of plastics has created significant challenges for environmental sustainability and inadequate waste management. Moreover, plastic pollution have raised concerns about their impact on ecosystems and human health [1].

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The production of plastics started around 1950, as a result of its high properties, but the industry has been using them for about a hundred years. In these decades, global plastic production has increased from two million tons in 1950 to 370 million tons in 2019, and it is growing. Each year, an estimated 4 to 12 million tons of plastics enter the oceans, and by 2050 there may be more plastics than fish in the sea [2].

Plastics can be described as „synthetic" (non-renewable) or „bio" (renewable) plastics. Synthetic plastics are extracted from crude oil, natural gas or coal. Biological plastics are made from renewable products - materials such as starch, carbohydrates, fats and vegetable oils [3].

The most common plastics include polypropylene (PP), polyethylene (PE), low-density polyethylene (LDPE), high-density polyethylene (HDPE), polyethylene terephthalate (PET), polystyrene (PS), polyurethane (PU) and polyvinyl chloride (PVC) [4]. Polyethylene is one of the most environmentally persistent plastics and its accumulation has created a serious pollution concern [5].

Around 76% of the total plastics production is treated as waste, of which 12% is incinerated, 79% is landfilled or released into the environment and only 9% is recycled [2].

It is estimated that between 5,3 and 14 million tons of plastic waste are dumped along the coasts every year, 10% of which ends up in the hydrosphere and accumulates over time [4]. Plastics are exposed to a gradual decomposition process that changes the chemical and physical structures of polymers, which results in the formation of small plastic fragments [6,7]. These molecules have the following sizes: megaplastic (>50 cm), macroplastic (5-50 cm), mesoplastic (0.5-5 cm) and microplastic (<5 mm) [8].

The term "microplastics" was first used in 2004, in a study of their presence in the marine environment, both in beach sediments and inside marine organisms [9], with non-biodegradable characteristics that make them long-durable in the environment [10,11].

Microplastic particles are classified by size, specific gravity, chemical composition and shape in two groups primary and secondary [12]. Primary microplastics are manufactured for industrial purposes or personal applications and include plastic granules used in the cosmetics industry, personal care products such as facial cleansers, toothpaste; and secondary results from the degradation of plastics in environment, by mechanical, chemical or biological action [12,13]. By their shape, microplastics can be classified (by their shape) as microbeads, granules, fibers, films, fragments and filaments [14]. Color is an important factor affecting aquatic organisms. Microplastics present in seawater samples and sediments are colorless or white, multicolored or black microplastics are less common. Aquatic animals may confuse their natural food with microplastics because of the similarity of colors [15].

Microplastics are present in a variety of environments around the world, including natural water, food, drinking water, wastewater, seawater, agriculture land and soil. Microplastics have been found in bottled water and groundwater sources [16,17].

The life cycle of microplastics starts with the release of primary or secondary microplastics into terrestrial and aquatic ecosystems, continues with their transportation in water systems, the final destination of microplastics [10].

Atmospheric currents, rainfall, surface runoff and ocean circulation transport microplastics from the soil into water. Lighter and smaller microplastics can be carried by wind in the air, and can reach more distant regions such as glaciated areas and high mountains [4].

The concentration of microplastics, as well as their distribution in sediments and water, are influenced by location, wind intensity and water flow [18]. After entering the sea, plastics are picked up by ocean currents and transported to the open sea [19].

After they accumulate in various parts of the environment, microplastics have the two main destinations: they either fragment and sediment, or are ingested by aquatic organisms. The final destination of microplastics is the body of a human or an environmental area [18]. Many scientific communities such as biology, soil chemistry, microbial ecology, environmental toxicology have studied the impact of microplastic pollution [20,21]. The numerous presence of microplastics in aquatic environments has generated intense concern about their harmful effects on marine life. These

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particles are detected in the entire water column, from surface waters to the deep sea and also in the atmosphere and soil, thus causing a substantial risk to the aquatic life [12]. Around 70% of marine plastic waste is deposited in sediments, 15% is found in coastal areas and the rest floats on the surface of seawater [22]. Microplastics act as carriers of dangerous chemicals including heavy metals, organic pollutants and plasticizers. Ingestion of microplastics by marine organisms disturbs their physiological functions and can cause mechanical damage in the digestive tract, which results in inflammation [23]. Microplastics obstruct the growth and expansion of plants, affect soil characteristics and have negative effects on the growth of the plants [24]. In the air, microplastics affect both aquatic and terrestrial ecosystems. Wind can carry microplastics that can become attached to dust or soil particles, and be returned to the environment [25].

The environmental fate of microplastics is a concern for human health [26]. Ingestion, inhalation and dermal contact are the most common routes of exposure to microplastics, of which ingestion is considered the most important [27]. Microplastics that enter the body may interact with histologic structures, affect cell structure and function, and can be transported via the bloodstream to vital organs, potentially including heart. Microplastics can induce adverse reactions, including possible negative effects on the cardiovascular system [26]. Microplastics get into the human body through the digestive system, the respiratory system and skin contact. The pathways into the digestive system are numerous and include food, air and drinking water. Seafood are the most common source of food that contain high levels of microplastics [28,29]. These cause alterations in the intestinal microbiota and induce a disequilibrium between healthy and harmful microorganisms, having a negative impact on the gastrointestinal tract and the entire body [30]. The respiratory system is affected by the production of oxidative stress in the respiratory airways and the lungs, which causes cough, sneezing and difficulty breathing due to inflammation, as well as dizziness due to low oxygen concentrations in the blood [30].

Additives used to improve the quality of plastics, such as bisphenol A, are released and absorbed into the body, and are responsible for endocrine and reproductive system disruption. [30]. Microplastics also accelerate hemolysis and the formation of a molecule that stimulates inflammation. These negative consequences vary depending on the level of exposure to these particles, and the susceptibility of the human body [31]. As can be seen, the negative effects of microplastics on the environment and humans are multiple and not all implications are yet known.

Their recovery from the environment is very difficult due to their small size, low specific gravity and compositional variety. The microplastics removal tehnologies from natural water and wastewater can be used physical, chemical and biological methods. The physical methods consist in fitration by membrans (microfiltration, ultrafiltration, and reverse osmosis) [32,33], or filtration by different media like quartos sand, zeolites, activated carbon and biochar, etc. [34–36], sedimentation associated with coagulation flocculation [37], magnetic separations [38], etc.

For example, chemical methods can be (for example) oxidation process. Advanced oxidation processes and UV/H_2O_2 [39,40]. Other chemical processes are very well represented by coagulation – flocculation followed by sedimentation, [41–43], photocatalysis [44]. Biological methos consists in biodegradation using mainly bacteria and fungi [45,46]. An effective method for removing microplastics is the flocculation coagulation process, which can remove between 40.5–54.5% and even 77.83% of MPs in water treatment plants, DWTP. [47]

The main coagulants used can be iron salts (ferrous sulfate, ferric chloride, etc.) and aluminum (aluminum sulfate, aluminum chloride, poly-aluminum chloride, PAC), and flocculants such as polyacrylamide (PAM), lysozyme monomers, aloe verra, etc. [48,49]. For optimum doses, reduction percentages can reach 72% for turbidity, and 91% for suspended matter. Aloe Vera contains phytochemical groups such as tannins, saponins, flavonoids, anthracene derivatives, quinone derivatives and proteins. Therefore, Aloe Vera gel contains chemical compounds that can agglomerate fine particles in suspension, (and) promoting the formation of large, sedimentable floccules. The use of Aloe Vera is a possible alternative to chemical flocculants for treating water contaminated with microplastics [49].

 doi:10.20944/preprints202605.0209.v1

The study of the removal efficiency of microplastics by common coagulants in treatment plants is justified, because a high efficiency would reduce the costs of their removal. The behavior of microplastics in water is like colloidal compounds. They form dielectric layers, the diffuse electric double layer, described by the model Derjaguin–Landau–Verwey–Overbeek (DLVO), the electrical charge on the surface depending on the salt content, pH, and the chemical composition of the microplastic (additives, pigments, etc.). Usually, under normal conditions, the microplastics in water are negatively charged [50].

The present study focused on the removal of two fractions of polyurethane microplastics with sizes between 50 μm and 500 μm from aqueous solutions at a concentration of 0.2 g/L, around 175 NTU. In the first stage of the study, tests were performed to identify the optimal doses of efficient reactive agents for microplastic removal, using the classical method, the Jar test. At this stage, attention was directed towards analyzing the variation of turbidity and their removal efficiency in the presence of classical coagulants such as: aluminum sulfate, SA, ferrous sulfate, SF, aluminum polychloride, PAC, an aloe vera flocculant and a classical adsorbent, activated carbon, CA, of the Norit GAC 830 W type.

In the second phase of the study, the efficiency of a multi-compartment sedimentation - filtration installation specifically designed for the removal of microplastics was determined. The efficiency for the removal of microplastics of the above-mentioned sizes from synthetic solutions was tested with simple microplastics only, but also with the reactives in the optimal doses identified according to the Jar test.

2. Materials and Methods

2.1. Preparation of Materials

2.1.1. Microplastics Characterization and Work Condition

The microplastic samples used in the study were obtained from polyurethane foam by piling and grinding. This study focused on removing polyurethane from water because it is one of the most used plastic materials and at the same time one of the most dangerous, being the fifth out of 55 polymers evaluated according to the Hazard Classification of Monomers. [51]. Polyurethane is obtained by chemical reaction between polyols (soft part) and diisocyanates (hard part), two lichids.[52]

Polyurethanes are materials with special properties, variable density, opencell structure, good air permeability, good dimensional stability, flexibility, elasticity, good adhesion and others. and at the same time have the ability to form composites with nanomaterials, such as carbon nanotubes, graphene derivatives, clay, and silica, for improving qualities for modifying properties etc. [51]

They can be used in various industrial and agricultural applications because they can be found in the form of Low-density flexible foam used in construction, the car manufacturing industry for upholstery and car seats; landscaping with synthetic vegetal layers, construction for roofs and walls. They can also be in the form of low density elastomers used in footwear industry for smart soles and other components; They can also be used as flexible plastic materials for tapes, belts, protective suits, clothing and accessories, packaging, etc. Composite materials with polyurethanes can form high-density materials, hard polyurethanes that are used as components of electronic instruments, for the car manufacturing industry or molded parts with various applications in all fields (furniture, agriculture, electronics and household appliances, etc.)[53].

Fine particles of various sizes were produced. Separation into particle size fractions was performed using a sieve system with grid mesh sizes of 2 mm, 1 mm, 500 μm, 200 μm, 100 and 50 μm.

 doi:10.20944/preprints202605.0209.v1

2.1.2. Chemical Reagents

The coagulants used in this study: aluminum sulfate, ferrous sulfate and aluminum polychloride, compounds commonly used as coagulants in water treatment processes, were delivered by the company Sigma Aldrich. The coagulant doses used were in range 0 - 60 mg/L.

Aloe Vera gel, AV, was used to increase the efficiency of the sedimentation process. In literature studies, it has been shown that, even in low dosages, AV can remove highly loaded water from suspended solids, and thus turbidity. The Aloe Vera gel used in the study was taken from an Aloe Vera plant in the research laboratory. Aloe Vera was cut, the gel was removed, ground, filtered and dosed into synthetic solutions containing microplastics. The doses of Aloe Vera used were 1-3 g/L.

Norit GAC 830 W type activated carbon GAC 830 W was used as an adsorbent, which is recognized for its high efficiency in physical adsorption processes, due to his large specific surface area and high porosity [54,55]. The dose of activated carbon was 0.5 g/L. The technical specifications of Norit GAC 830 W activated carbon are summarized in Table 1.

Table 1. Technical specifications of Norit GAC 830 W activated carbon, [54].

No. crt.	Specification	Active Carbon tip Norit GAC 830 W
1	Particle size >2,36 mm	Maximum 15% in mass unit
2	Particle size <0.6 mm	Maximum 5% in mass unit
3	Moisture	Maximum 5%
4	Iodine number	957 (mg/g)
5	Methylene blue adsorption	20 g / 100 g
6	Ash content	12%
7	Total surface area, BET analyses	1100 m^2/g
8	Apparent density	500 kg/m^3

2.1.3. The Decantation - Filtration Installation

To study the removal efficiency of polyurethane microplastics, a smart installation made up by decantation and filtration units was used, as shown in Figure 1. This installation was specifically designed for the removal of microplastics. It operates continuously and consists of 4 components: a raw water tank, 1, a multicompartment decanter (enclosures 2, a quartz sand filter, 3, and a treated water tank, 4.

Synthetic solutions with microplastics, as well as microplastics with SA, were introduced into the raw water tank and dosed at variable flow rates, between 1.0 and 4.0 L/h. The synthetic solutions were passed through the decanter compartments. The input and the output from the decanter were analyzed for residual turbidity in the supernatant. The water was finally introduced into the quartz sand filter. Turbidity was also determined in the water sample from the treated water tank.

2.1.4. FTIR Spectrum of Microplastics

Fourier transform infrared spectroscopy (FT-IR) using a Nicolet IS50FT-IR spectrometer (Nicolet, Massachusetts, USA), equipped with a DTGS detector that provides information with high sensitivity in the range of 4000 cm^{-1} and 100 cm^{-1}, at a resolution of 4 cm^{-1}, were used. The IR spectrum is shown in Figure 2.

2.1.5. The Jar Test

The Jar VELP Scientifica F105A0117 FP4 test was used to determine the optimal doses of reagents. The Jar Test contains four stirring rods and four glasses of one liter. The stirring rate and time can be adjusted using the buttons located on the front panel.

 doi:10.20944/preprints202605.0209.v1

Figure 1. Microplastics decantation and filtration installation.

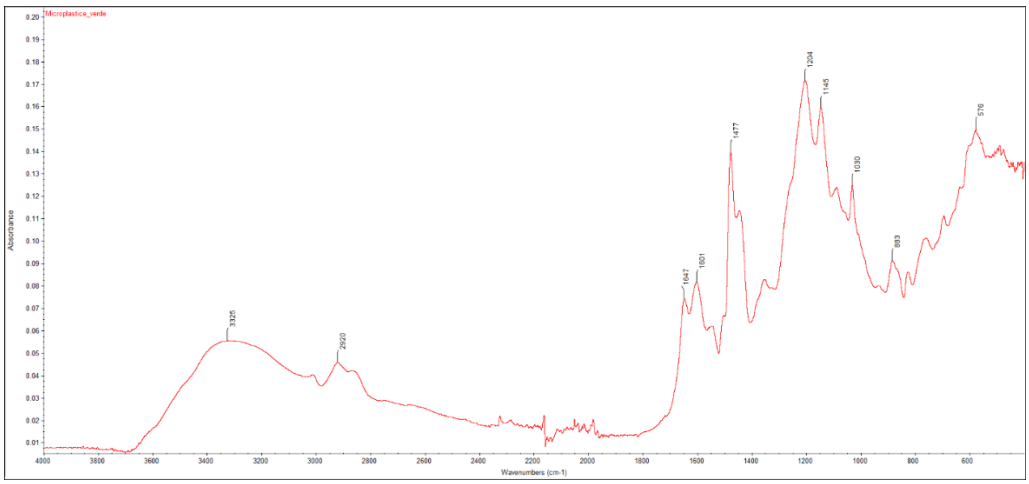

Figure 2. FTIR spectrum of microplastics used in the research.

Turbidity was determined using a HACH 2100P 150 turbidimeter. Synthetic solutions were prepared with a concentration of 0.2 g microplastics/L. The numerical values of the initial turbidity (numerical values) ranged from 165 - 180 NTU.

The following coagulants were used: aluminum sulfate $Al_2(SO_4)_3$, iron sulfate $FeSO_4$, aluminum polychloride - PAC, with concentration values ranging between 0 - 60 mg/L, Aloe Verra, AV, with doses between 1-3 g/L, activated carbon 0.5 g/L. The samples were stirred using the Jar test for 10 min / 200 rpm, and 5 min / 100 rpm. After reading the initial turbidity, the samples were allowed to settle. The retention time was 60 min, during which the turbidity in the supernatant was measured at different time intervals. Graphical representations of the variation of turbidity removal efficiency with time are shown in Figures 3–5.

 doi:10.20944/preprints202605.0209.v1

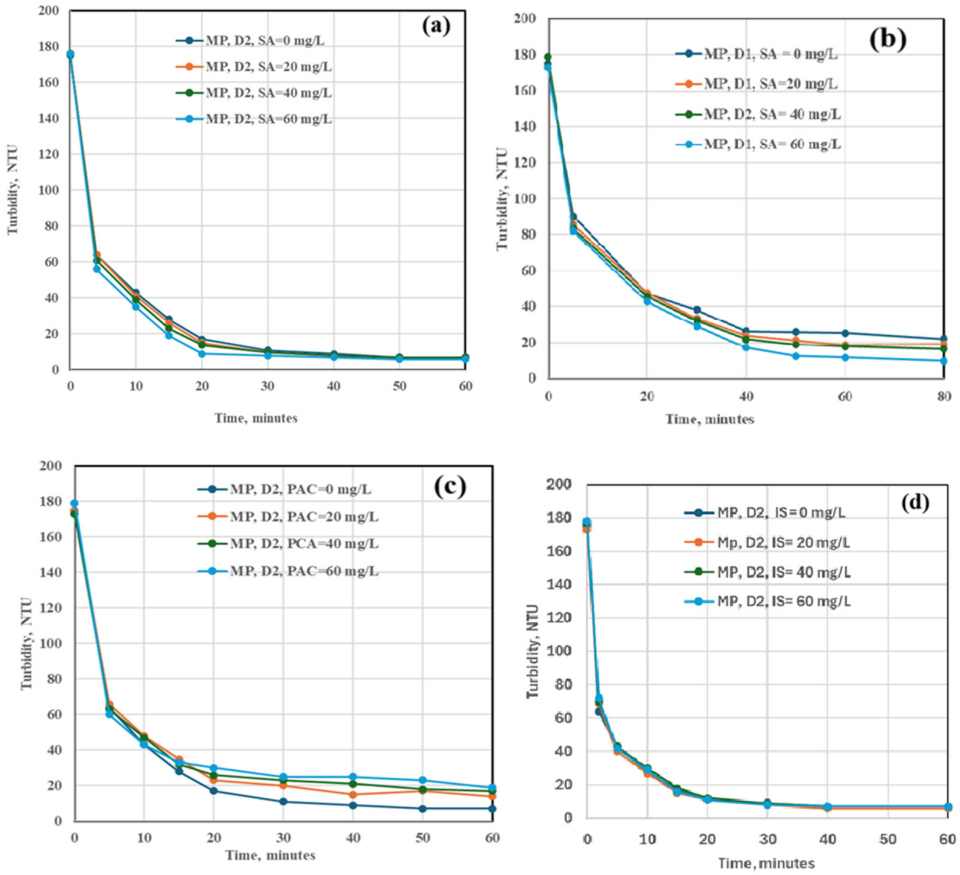

Figure 3. Turbidity variation of microplastics of various sizes in the presence of different coagulants: (a). Sulfate of aluminum, SA, 200 μm <D<500 μm, D2, (b). Sulfate of aluminum, D<100 μm, D1, (c). Polychloride of aluminum, PAC, D2, (d). Iron sulfate, IS, D2,.

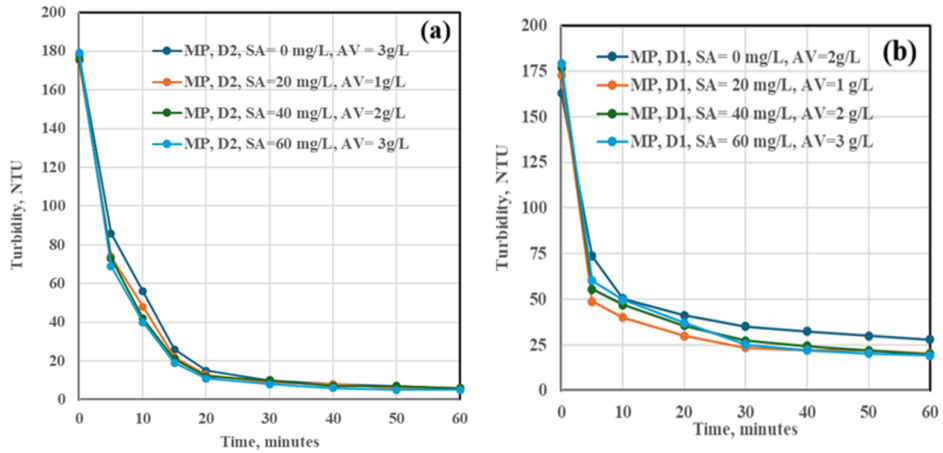

Figure 4. Turbidity variation of microplastics of various sizes in the presence of aluminum sulfate and aloe vera: a. 200 μm<D<500 μm, D2, and b. D<100 μm, D1.

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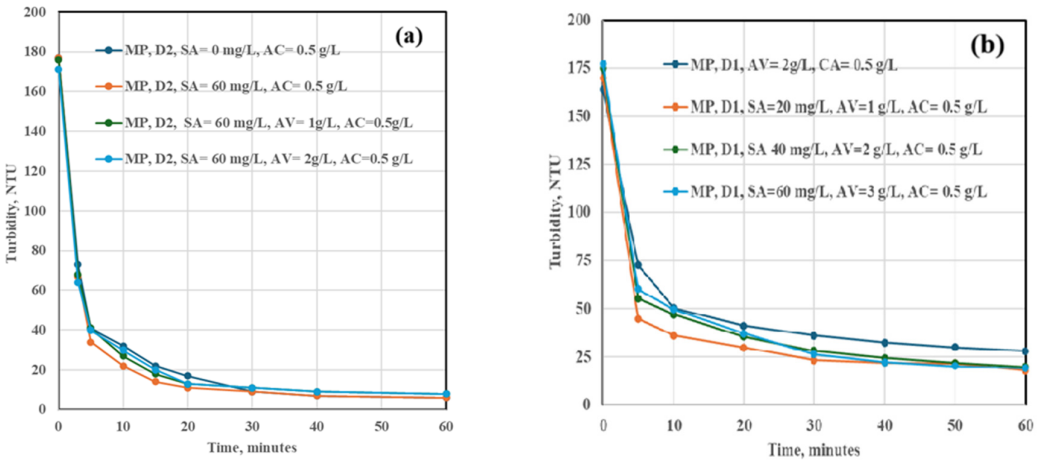

Figure 5. Turbidity variation of microplastics of various sizes in the presence of aluminum sulfate, aloe vera and activated carbon: a. 200 μm <D<500 μm, D2, b. D<100 μm, D1.

2.1.6. Design of Experiments Based on the Box-Behnken Approach

Response Surface Methodology (RSM) is a commonly used statistical approach designed to fine-tune experimental setups and assess how various factors impact a specific outcome. This technique facilitates the construction of predictive models that incorporate both the separate, and the combined influences of independent variables [56,57]. In this research, the Coagulation-Floculation process was optimized using the Box-Behnken Design (BBD) integrated within the Response Surface Methodology (RSM) framework, implemented through Design Expert 13 software. This approach is especially efficient in reducing the number of required experiments, while enhancing the precision of the predictive model [58, 59]. A three-level factorial design (−1, 0,+1) was used to evaluate the effects of key process variables: Flow rate (A), Microplastic size (B), and Aluminium sulphate concentration (C). The coded levels and corresponding experimental values for each factor are presented in Table 2.

Table 2. Variables and levels considered for microplastic removal.

Variable	Name	Title 3		
		-1	0	1
A	Flow rate (l/h)	1	2.5	4
B	Microplastic size (μm)	50	200	350
C	Aluminium sulphate concentration(mg/L)	0	30	60

Mathematical modeling aims to establish a functional relationship, denoted as

$$Y = f(x_1, x_2, x_3, \ldots, x_n) \quad (1)$$

where Y denotes the dependent outcome, and x_1 to x_n represent the independent variables or contributing factors. This model is classified as deterministic, meaning the result is strictly determined by the inputs, without accounting for randomness or measurement uncertainty. Often, the response is estimated using a second-degree polynomial equation, offering a versatile yet organized method for representing nonlinear interactions among variables.

$$Y\ (\%) = \beta_0 + \Sigma\ \beta_i x_i + \Sigma\ \beta_{ij} x_{ij} + \Sigma\ \beta_{ii}\ x_i^2 + \varepsilon \quad (2)$$

Y (%) represents the estimated response and serves as the key variable under investigation. It is measured with defined accuracy. The term β_0 refers to the intercept or mean value of the response, while β_i, β_{ij}, and β_{ii} denote the model's coefficients, which are initially unknown and must be derived

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from experimental observations. The variables x_i and x_j are the independent factors influencing the response, and ε accounts for the residual error in the model [56, 57].

3. Results and Discussions

3.1. FTIR Microplastics Characterization

One of the most effective methods for chemical characterization is Fourier Transform Infrared Spectroscopy (FTIR). The IR spectrum is characterized by the adsorption peaks that are specific to the vibrations of the constituent atoms of the material. The IR spectrum of the unknown material from which the microplastics are made was compared with the IR spectra of some known plastic materials [60,61]. The FTIR analysis is shown in Figure 2.

From the analysis of Figure 2 we can observe the characteristic peaks. The characteristic vibrations of the peaks were highlighted by Villegas-Camacho, O. et al. [61] as follows: 3325 cm^{-1} is specific to the vibration of the urethane group (-NH-COO-) in the heavy segment (diisocyanate); 2920 cm^{-1} is attributed to the aromatic C-N stretching; 1647 cm^{-1} is attributed to the stretching of the C= O bond of the ester group; 1601 and 1477 cm^{-1} indicate the presence of the C= C stretching vibration of the benzene rings; 1204 cm^{-1} and 1030 cm^{-1} are evidenced the C-O bond of the ester group; 883 cm^{-1} is characteristic of the C- H bending vibration of the substituted benzene ring; 576 cm^{-1} is specific for substituted C - H bonds. All these signals confirm the polyurethane properties of the plastic.

3.2. The Jar Test - Determining the Optimal Doses of Reagents

3.2.1. Identification of the Optimal Doses of Coagulants

Figures 3 a-d present the turbidity variations of microplastics with the dimensions of 200 μm < D < 500 μm and D < 100 μm, in the presence of different coagulants such as aluminum sulfate, aluminum polychloride, ferrous sulfate of various concentrations.

From the analysis of Figure 3, it can be seen that, at large sizes of microplastic particles, D2, aluminum sulfate has an important influence on the coagulation and sedimentation of the particles. The Jar test for the control sample on microplastics with size D2, and at an initial turbidity of 176 NTU, shows that, after 20 minutes, the turbidity reaches 90.2%, after 30 minutes - 93.7%, and after 60 minutes - 96%.

Analysing Figure 3a, it can be observed that the optimal dose required to ensure an efficient settling of microplastic particles with size 200 μm < D < 500 μm is of 60 mg/L $Al_2(SO_4)_3$ sulfate of aluminum, SA, ensuring a residual turbidity of 96.6% at a retention time of 50 minutes, 94.9% after 20 minutes, and 95.5% after 30 minutes. This dose was also used for the tests performed on the smart decantation - filtration installation. This behavior can be explained by the fact that large particles of type D2 settle as sediment/deposit after a longer retention time, even in the absence of coagulants.

As the size of the microplastics decreases, at D1, D < 100 μm, the residual turbidity grew to 47 NTU, 73% of the initial, after 20 minutes, 25.2 NTU, 85.6% after 60 minutes, for the system in which no coagulant was used. In the case of using SA, it was found that the optimal dose is of 60 mg/L, and this ensures an efficiency of 93.1% after 60 minutes, from an initial concentration of 173 NTU, as shown in Figure 3b. SA was used in doses similar to those used in the study and for the removal of microplastics from PS, PE and PVC. Their removal efficiency ranged between 84 and 100%, [62,63]. No similar studies were identified for the removal of microplastics from PU.

The efficiency of aluminum polychloride, PAC, is lower than in SA, according to Figure 3c, the optimum dose required to ensure efficient settling of microplastic particles with size D2 is 40 mg/L, presenting a residual turbidity of 14 NTU at a retention time of 60 minutes. The optimum dose of ferrous sulphate required to ensure effective settling of microplastic particles of size D2 is of 20 mg/L, as shown in Figure 3d, which gives a residual turbidity of 6 NTU at a retention time of 60 minutes, 96.6%, similar with 60 mg/L SA. Studies under similar conditions were performed with PAC for PET. Removal efficiency ranged between 33-100%. [62,63].

 doi:10.20944/preprints202605.0209.v1

3.2.2. Identifying the Optimal Dose of Aloe Vera

This stage is ment to identify the influence of some flocculants, in order to reduce the turbidity of contaminated water, by enhancing the aggregation of suspended particles and facilitating solid-liquid separation processes, in order to optimize the removal of polyurethane microplastics.

In addition to the coagulant aluminum sulfate $Al_2(SO_4)_3$ at a concentration of 60 mg/L, Aloe Vera gel, a natural flocculant, was added to the solution in various quantities: between 1 g/L to 3 g/L, to optimize the microplastic removal process, in order to determine the optimal dose to ensure a significant reduction in turbidity.

From the analysis of Figure 4a, it can be noted that the optimal dose necessary to remove microplastic particles with size D2 is of 60 mg/L $Al_2(SO_4)_3$ aluminum sulfate combined with Aloe Vera gel with a concentration equal to 3 g/L, ensuring a residual turbidity of 5 NTU at a retention time of 60 minutes with an efficiencyof 97.2%. Therefore, the combination of SA (classical coagulant) and AV gel (natural flocculant) is highly effective in reducing turbidity for D2 sizes. The optimal dose necessary to remove microplastic particles with D < 100 μm, D1, is of 60 mg/L aluminum sulfate $Al_2(SO_4)_3$ combined with Aloe Vera gel having a concentration equal to 3 g/L, as shown in Figure 4b. It can be seen that the turbidity efficiency without coagulant for D1 at 60 minutes is 85.6% (25.2 NTU residual turbidity), and, in the presence of SA with a concentration of 60 mg/L, it reaches 93.1% (10 NTU residual turbidity). In the presence of SA 60 mg/L and 3 g AV.The residual turbidity was 19.3 NTU, 89.3%. Aloe vera does not improve the particle removal capacity with D1. The best performance was obtained for SA 60 mg/L.

3.2.3. Identifying the Efficiency of Activated Carbon

The efficiency of granular activated carbon on the removal of microplastics from synthetic solutions was tested. Solutions were prepared in which were added: activated carbon c = 0.5 g/L, aluminum sulfate $Al_2(SO_4)_3$ of concentration 60 mg/L, as well as Aloe Vera gel of various concentrations.

According to Figure 5, the optimal dosages required to give effective settling of microplastic particles of size 200 μm < D < 500 μm for the activated carbon system, 0.5 g/L, and SA at 60 mg/L concentration provide a residual turbidity of 6 NTU at a retention time of 60 minutes. The combination of aluminum sulphate (classical coagulant) and activated carbon (adsorbent) was effective in reducing turbidity caused by microplastics, whereas the combination of Aloe Vera gel and activated carbon is not an effective solution.

The use of natural coagulants such as Aloe Vera with aluminum sulfate represents a combination that allows obtaining the best microplastic removal efficiency for D2, of 97.2% For size D1, the efficiency is lower than in the case of using only a dose of 60 mg/L of SA. Studies have been carried out on the efficiency of microplastic removal in the presence of biopolymer based coagulants, which include starch, cellulose, gum, seeds, tannins, and cactus. The removal efficiency of microplastics was around 95% for chitosan-tannic acid, 98% for protein amyloid fibrils, and greater than 90% for starc [64].

The use of only activated carbon in a concentration of 0.5 g/L for microplastics with size D2 determines an efficiency of 96.6%, comparable to that obtained in the case of using SA with a concentration of 60 mg/L The introduction into the system, along with SA 60 mg/L, CA 0.5 g/L and Aloe Vera 1 g/L for D2, determines a decrease in efficiency, see Figure 5a. This may be determined by a possible competition between microplastics and aloe vera for activated carbon adsorption centers. By adsorption of AV, there is inactivation of active centers and loading with negative charges similar to microplastics.. As the amount of AV increases, the microplastic removal efficiency decreases from 97.2% in the SA 60 mg/L and AV 2 mg/L system to 95.3% for the SA 60 mg/L, AV 2 mg/L and CA 0.5 g/L system for D2. The introduction of SA, CA or AV into the system causes a rather small increase in the removal efficiency of microplastics with size D2, ranging between 0.6-1.2%. This suggests that they do not represent a problem in terms of sedimentation, and the residual turbidity between 6 and 10 NTU does not represent a problem, because it can be removed by filtration. For size D1, the

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removal efficiency is lower than for those with size D2, the best efficiency is obtained in the presence of SA with a concentration of 60 mg/L, of 93.1%. The introduction of AV with a concentration of 3g/L and SA 60 mg/L decreases the efficiency to 91.06%. By introducing only AV and CA into the system, the removal efficiency is even lower than in the case of the system without coagulants, reaching 83%. This is explained by the fact that, instead of the particles unification, additional repulsion fields are created, AV and microplastics compete for the activated carbon. Structures are formed that repel each other and are more difficult to be removed from the system. The introduction of activated carbon in combination with aluminum SA and aloe vera is not an inspired solution. The use of aluminum sulfate is a viable, advantageous solution, (advantageous) because this coagulant is usually used in treatment plants and it is proven that it is also effective for the removal of microplastics. For sizes smaller than 100 μm of polyurethane microplastics, the residual turbidity between 20 and 40 NTU can cause water quality problems. These can be found in drinking water in the residual turbidity that comes out of the filter at values lower than 0.5 NTU, or at values higher than 0.5 NTU, which requires the use of additional removal methods.

Activated carbon is biological carbon that is subjected to chemical or physical processes using heat and other chemical agents. Recently, a significant number of published articles have focused on the use of activated carbon as an adsorbent to remove microplastics from wastewater. Activated carbon captures microplastics by physical adsorption mechanisms, due to his specific surface and porous structure, which provides numerous active sites for their attachment. The adsorption process is promoted by hydrophobic interactions and van der Waals forces between the microplastics and the activated carbon surface [55].

3.2.4. Analysis of Variance and Residuals

The microplastic removal efficiency, Y, was optimized using Response Surface Methodology (RSM), specifically through a Box-Behnken experimental design. A second-order polynomial model was developed, showing a high level of agreement between the adjusted and predicted R^2 values. The model's reliability was statistically validated through the analysis of variance (ANOVA), as detailed in Table 2 [65,66]. Mathematical modeling involved estimating the regression coefficients based on data from 17 experimental runs. These coefficients were calculated using Design Expert version 12, which generated the coded form of the quadratic model presented in the following Equation.

$$Y = 93.97 - 1.95\,A + 7.00\,B + 1.24\,C + 1.57\,AB - 0.2700\,AC - 1.11\,BC + 1.36\,A^2 - 3.90\,B^2 + 0.6625\,C^2 \quad (3)$$

To assess the suitability and predictive power of the quadratic model for microplastic removal, an analysis of variance (ANOVA) was conducted. The results, presented in Table 3, demonstrate that the model is statistically robust, with a highly significant p-value (< 0.0001) and an F-statistic of 76.52, confirming its capacity to account for variations in removal efficiency. According to the data in Table X, several factors were found to significantly influence the process ($p < 0.05$), including the flow rate (A), microplastic size (B), aluminum sulphate concentration (C), the interaction between flow rate and microplastic size (AB), the interaction between microplastic size and aluminum sulphate concentration (BC), as well as the quadratic terms of flow rate (A^2) and microplastic size (B^2). In contrast, the interaction between flow rate and aluminum sulphate concentration (AC), along with the quadratic term of aluminum sulphate concentration (C^2), did not exhibit a statistically significant effect (p-values > 0.05).

The very close fit between the predicted outcomes and the experimental data, along with a high determination coefficient (R^2), confirms the reliability of the model's predictive capacity. The ANOVA results further reinforce the appropriateness of the quadratic model in characterizing the coagulation–flocculation mechanism, highlighting the significant influence of flow rate, microplastic particle size, and aluminum sulphate concentration. An Adequate Precision value of 27.8049 - well above the recommended minimum of 4 - demonstrates a strong signal-to-noise ratio, validating the model's effectiveness in exploring the design space. Additionally, the coefficient of variation (C.V. = 0.9243%) falls comfortably within the acceptable threshold (<10%), indicating consistent precision

 doi:10.20944/preprints202605.0209.v1

and reproducibility across experimental trials. The high coefficient of determination ($R^2 = 0.9899$), and the adjusted R^2 value (0.9770) demonstrate that the proposed model is well-suited for accurately representing the experimental conditions. These statistical indicators confirm the model's capacity to reflect the observed data with strong predictive reliability.

Table 3. ANOVA for postulated model.

Source	Sum of Squares	df	Mean Square	F-Value	*p*-Value	
Model	520.80	9	57.87	76.52	< 0.0001	Significant
A-Flow rate	30.42	1	30.42	40.23	0,0004	Significant
B-Microplastic size	392.28	1	392.28	518.72	< 0.0001	Significant
C-Aluminium sulfate concentration	12.35	1	12.35	16.33	0,0049	Significant
AB	9.86	1	9.86	13.04	0.0086	Significant
AC	0.2916	1	0.2916	0.3856	0.5543	
BC	4.88	1	4.88	6.46	0.0386	Significant
A^2	7.76	1	7.76	10.26	0.0150	Significant
B^2	63.96	1	63.96	84.58	< 0.0001	Significant
C^2	1.85	1	1.85	2.44	0,1620	
Residual	5.29	7	0.7562			
Lack of Fit	2.60	3	0.8680			
Pure Error	2.69	4	0.6724			
Cor Total	526.10	16				
Std. Dev.	0.8696			R^2		0.9899
Mean	93.09			Adj. R^2		0.9770
CV (%)	0.9342			Pred. R^2		0.9128
				Adeq precision		27.8049

As illustrated in Figure 6, the residuals exhibit a normal distribution pattern, aligning closely along the reference line. This distribution suggests that the model errors are randomly and symmetrically dispersed, thereby supporting the assumption of normality and reinforcing the statistical validity of the regression analysis.

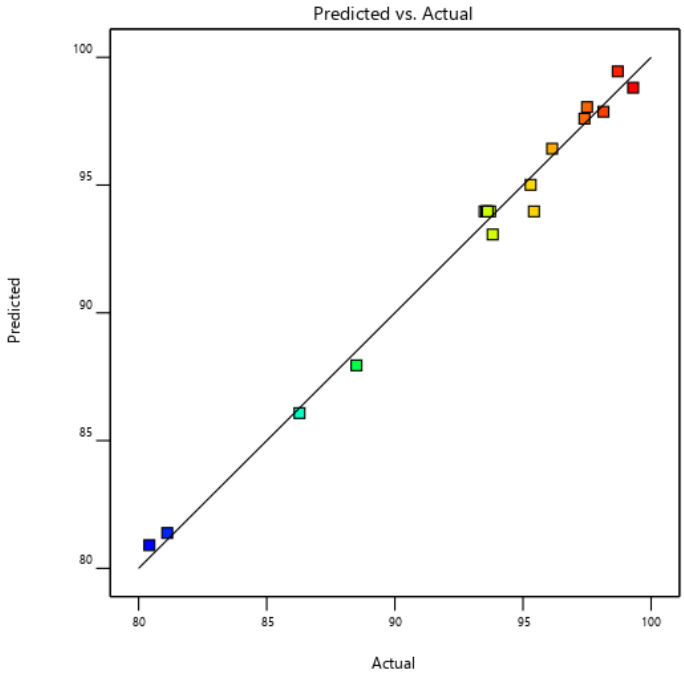

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Figure 6. Comparison of the experimental and BBD-RSM-predicted microplastic removal efficiency (%) response.

3.2.5. Graphical Representation of Response Surfaces

Three-dimensional response surfaces and two-dimensional contour diagrams offer meaningful visual interpretations of how process parameters interact to influence removal efficiency. These graphical representations depict the variation in microplastic removal efficiency (R %) when two variables are simultaneously adjusted, while the third remains fixed at its central value. The contour plots (Figure 7), generated using Design Expert version 12, were instrumental in examining the combined effects of the three selected factors on the response variable Y (%), providing a clearer understanding of their synergistic behavior. The established model equation enables the prediction of response values corresponding to specific levels of the input factors.

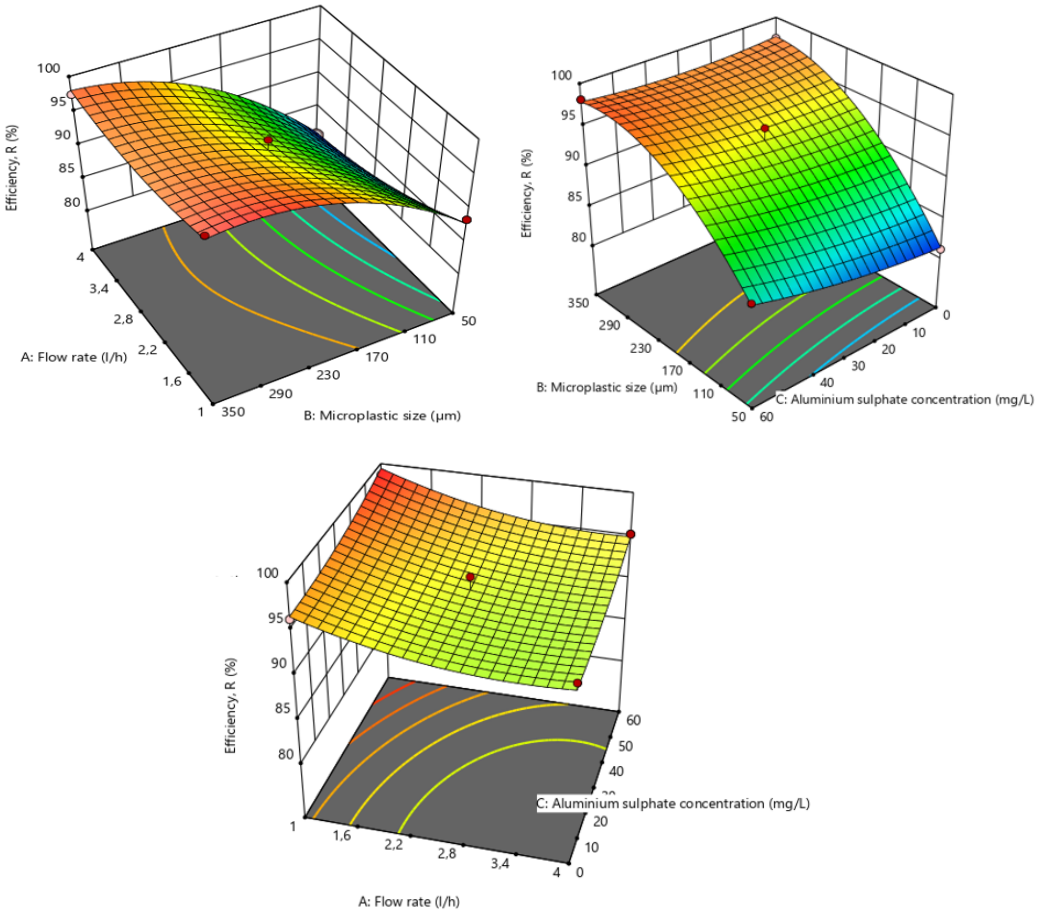

Figure 7. 3D response surface plots for microplastic removal efficiency as a function of: (a) Flow rate and microplastic size, (b) microplastic size and aluminum sulphate concentration (c) aluminum sulphate concentration and Flow rate.

Three-dimensional response plots in Figure 7 (a-c) give a close depiction of how the efficiency of microplastic removal is a function of the interaction among important operating parameters. In Figure 7a, the combined influence of Flow rate (l/h) and microplastic size (µm) indicates that high levels of both variables result in enhanced removal performance. Figure 7b illustrates the interactive effect of microplastic size (µm) and aluminum sulphate concentration (mg/L) with higher aggregation, and removal efficiency noted for higher particle sizes and higher dosages of coagulant. Figure 7c displays the combined effect of aluminum sulphate concentration and flow rate on overall

 doi:10.20944/preprints202605.0209.v1

process efficiency. The graph illustrates that efficiency improves under conditions of high coagulant dosage, and relatively low to moderate flow rates. The red to green gradation shift over the surface signifies improving performance that intensifies as increasing amounts of chemicals are added, and maximum efficiency observed where coagulant concentration is high. The findings of these results show that gentle operating conditions, coupled with adequate chemical input, facilitate improved aggregation of microplastics, (and) subsequently leading to improved removal performance.

3.2.6. Optimization Using Desirability Function

The desirability function is a well-established technique within response surface methodology (RSM) that enables the simultaneous optimization of multiple response variables. In the context of Coagulation-Flocculation processes, this method converts each response into a dimensionless value ranging from 0 (indicating complete undesirability) to 1 (indicating full desirability). Each response is assigned a desirability score based on how effectively it aligns with the predefined optimization goal typically aimed at enhancing microplastic removal efficiency.

Table 4 shows the optimization criterion, constraints, and optimum solution for microplastic removal. Based on the desirability function approach, the optimal conditions were determined as Flow rate of 1.108 l/h Microplastic size of 221.272 μm, and Aluminum sulphate concentration of 51.932 mg/L. These conditions correspond to a desirability value of 1, which implies that the optimization objectives were fully achieved.

Table 4. Criteria constraints and optimal solutions.

Name	Goal	Lower Limit	Upper Limit	Solution	Desirability
A-Flow rate	is in range	1	4	1.108	1
B-Microplastic size	is in range	50	350	221.272	1
C-Aluminium sulphate concentration	is in range	0	60	51.932	1
Efficiency (%)	maximize	80.42	99. 3	98.985	1

According to the numerical results obtained, the installation consisting of a multi-chamber decanter and a quartz filter is effective in removing microplastics with sizes between 50 μm and 350 μm, from a synthetic solution with a concentration around 175 NTU, and the flow rate in range 1- 4 l/h. The flow rate of the raw water supply is a very important parameter influencing the efficiency of the plant. As expected, an increase in plant feed flow rate causes a decrease in turbidity removal efficiency. The turbidity at the outlet of the decanter and at the outlet of the plant was compared with the values imposed by the current legislation Directive 2020/2184/EC and Ordonance 7 of 2023 which impose a maximum permissible limit, MPL, of turbidity of 1 NTU.

When using synthetic solutions containing only microplastics with aluminum sulfate in doze of 60 mg/L reactants, it was found that the total efficiency of the system ranged between 80.42 and 99.3%. The residual turbidity for the water sample with microplastic particles at the outlet of the decantor has values in range of 5 - 20 NTU, after a retention time of 60 minute, and after the water is passed through the filter, the rezidual turbidity has values between 1 - 3 NTU.

The turbidity at the exit of the decanter is comparable to the turbidity values at the exit of the decanters used at industrial level. Turbidity at the exit of the filter has values higher than the MPL, which requires either a resizing of the filter which turns out to be undersized, or an increase of the retention time in the decanter, or the use of coagulants, flocculants more efficient.

3. Conclusions

The use of activated carbon is effective in microplastics removal if used alone or in combination with aluminum sulfate.

The use of Aloe Vera gives increased microplastic removal efficiency, while the combination of Aloe Vera and activated carbon is not recommended.

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Microplastics in D1 and D2 sized synthetic solutions can be removed from contaminated water by decantation and filtration, and the efficiency is around MPL values (maximum permissible limit), of 1 NTU.

The use of classical coagulants and flocculants as aluminum sulphate, ferrous sulphate has a good efficiency on microplastic removal, which can provide a residual turbidity of 5 – 20 NTU after a retention time of 50 - 60 minutes at the outlet of the decanter. The decanter is efficient both for synthetic solutions containing only microplastics, and for synthetic solutions with various reagents. The filtration operation is not sufficiently well sized to ensure maximum efficiency.

In conclusion, it is necessary either to identify more efficient flocculating coagulants to further increase the efficiency of the decanter, or to resize the filter in order to reach the values required by the legislation in force.

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