Revista ARACÊ
ISSN: 2358-2472

REMOVAL OF POLYETHYLENE MICROPLASTICS BY CHEMICAL COAGULATION: CONTRIBUTIONS TO ENVIRONMENTAL POLLUTION CONTROL

REMOÇÃO DE MICROPLÁSTICOS DE POLIETILENO POR COAGULAÇÃO QUÍMICA: CONTRIBUIÇÕES PARA O CONTROLE DA POLUIÇÃO AMBIENTAL

ELIMINACIÓN DE MICROPLÁSTICOS DE POLIETILENO MEDIANTE COAGULACIÓN QUÍMICA: APORTACIONES AL CONTROL DE LA CONTAMINACIÓN AMBIENTAL

https://doi.org/10.56238/arev7n7-055

Date of submission: 06/03/2025 **Date of publication:** 07/03/2025

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ABSTRACT
This study evaluated the coagulation process using Aluminum Sulfate ($Al_2(SO_4)_3$) for the removal of pristine and aged low-density polyethylene (LDPE) microplastics (MPs). The methodological procedure consisted of an experimental assay applying coagulation in specialized equipment (Jar Test) for MP removal, with turbidity analysis as a control parameter for coagulation/flocculation efficiency. Subsequently, the treated samples underwent laser granulometry to assess the Mean Diameter (Mz) parameter, evaluating the coagulant's effect on aggregate formation. The key findings demonstrated optimal removal efficiencies for pristine and aged MPs (30, 90, and 120 days), with maximum removal rates of 96.47%, 97.85%, 99.07%, and 77.28%, respectively. This technique proved highly effective in MP removal, particularly for MPs degraded up to 90 days.

Keywords: Coagulation; Microplastics; Removal; Efficiency; Aluminum sulfate.

RESUMO
Este estudo avaliou o processo de coagulação usando sulfato de alumínio ($Al_2(SO_4)_3$) para a remoção de microplásticos (MPs) de polietileno de baixa densidade (LDPE), pristinos e envelhecidos. O procedimento metodológico consistiu em um ensaio experimental que aplicou a coagulação em equipamento especializado (Jar Test) para a remoção de MPs, com análise de turbidez como parâmetro de controle da eficiência da coagulação/floculação. Posteriormente, as amostras tratadas foram submetidas à granulometria a laser para avaliar o parâmetro de diâmetro médio (Mz), avaliando o efeito do coagulante na formação de agregados. Os principais resultados demonstraram eficiências de remoção ideais para MPs pristinos e envelhecidos (30, 90 e 120 dias), com taxas de remoção máximas de 96,47%, 97,85%, 99,07% e 77,28%, respectivamente. Essa

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ISSN: 2358-2472

técnica se mostrou altamente eficaz na remoção de MPs, especialmente para MPs degradados por até 90 dias.

Palavras-chave: Coagulação; Microplásticos; Remoção; Eficiência; Sulfato de alumínio.

RESUMEN

En este estudio se evaluó el proceso de coagulación con sulfato de aluminio ($Al_2(SO_4)_3$) para la eliminación de microplásticos (MP) de polietileno de baja densidad (LDPE) prístinos y envejecidos. El procedimiento metodológico consistió en un ensayo experimental aplicando coagulación en equipo especializado (Jar Test) para la eliminación de MP, con análisis de turbidez como parámetro de control de la eficiencia de coagulación/floculación. Posteriormente, las muestras tratadas se sometieron a granulometría láser para evaluar el parámetro Diámetro Medio (Mz), evaluando el efecto del coagulante en la formación de agregados. Los resultados clave demostraron una eficiencia de eliminación óptima para MP prístinos y envejecidos (30, 90 y 120 días), con tasas de eliminación máximas del 96,47%, 97,85%, 99,07% y 77,28%, respectivamente. Esta técnica demostró ser muy eficaz en la eliminación de MP, en particular para las MP degradadas hasta 90 días.

Palabras clave: Coagulación; Microplásticos; Eliminación; Eficacia; Sulfato de aluminio.

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INTRODUCTION

One of the most pressing global challenges today is the contamination of water bodies, particularly in developing countries, where inadequate sanitation infrastructure exacerbates the problem. Solid waste, predominantly originating from urban centers, contributes significantly to this issue, with an estimated 25 million tons discharged into oceans annually. Plastic debris is a major concern, with Brazil alone generating 11.3 million tons of plastic waste in 2020, ranking as the fourth-largest global producer (Montenegro M; Teles D B; Vianna M, 2020). Worldwide plastic production reached 367 million tons in 2021 and is projected to rise to 550 million tons by 2030 (Geyer; Jambeck; Law, 2017).

Plastics from urban areas, when improperly disposed of, intensify marine litter pollution globally (Silva et al., 2021). Primary microplastics (MPs)—such as resin pellets or granules (<5 mm) used in manufacturing or found in cosmetics, pharmaceuticals, and industrial products (Napper et al., 2015) enter the environment via wastewater and industrial effluents (Picó Y; Barceló D, 2019). Secondary MPs, defined as synthetic polymer particles below 5 mm, result from the fragmentation of macroplastics by microbial activity, particularly in marine environments (Elkhatib; Oyanedel-Craver; Carissimi, 2021).

The ecological impact of plastic debris is severe, as MPs adsorb toxic chemicals (e.g., heavy metals, organic pollutants, and pathogenic bacteria), disrupting marine trophic chains and posing risks to human health. These particles can infiltrate organs, tissues, and even cellular structures, inducing toxic or lethal effects (Rafiee et al., 2018). Recent *in vitro* and *in vivo* studies have identified MPs in brain tissues shortly after ingestion, triggering microglial phagocytosis and apoptosis (Kwon et al., 2022).

MPs are ubiquitous, contaminating water bodies, soils, effluents, drinking water, and aquatic organisms (Massuga et al., 2022; Shen et al., 2022). The European Union recognizes MPs as a global crisis due to their persistence, ecological disruption, and mismanagement by administrative authorities (Silva et al., 2021). Conventional wastewater treatment methods often fail to remove MPs effectively due to their high surface area, persistence, and low density (Elkhatib; Oyanedel-Craver; Carissimi, 2021; Shen et al., 2022). Coagulation, however, is a promising technique for suspended and colloidal particle removal (Murphy F et al., 2016).

Plastics are classified by type, shape, and color. Types include fragments, pellets, filaments, films, foams, and granules. Pellets may be cylindrical, disc-shaped, or spherical, while fragments vary from rounded to angular with irregular edges (Hidalgo-Ruz et al., 2012). The most common thermoplastic resins—PET, PP, PE, PVC, and PS—are used globally for packaging and products (Andrady, 2011).

Plastic debris is categorized by size: macroplastics (>2 cm), mesoplastics (2 cm–5 mm), MPs (<5 mm–1 µm), and nanoplastics (<100 nm). The latter, comparable to engineered nanomaterials, threaten base trophic levels like nanoplankton (Wagner et al., 2014).

Photooxidation and abrasion drive plastic fragmentation, altering chemical bonds and facilitating MP formation (Montagner et al., 2021). Ingestion of plastic debris across trophic levels causes morphological, biochemical, and behavioral disruptions in organisms (Hartman G, 2015).

Low-density polyethylene (LDPE) and its variants (LLDPE, HDPE, etc.) are widely used due to their low cost and versatility (Coutinho; Mello; Santa Maria, 2003). Polymer degradation under UV radiation (UVA/B/C) accelerates MP formation. UV transilluminators emit electromagnetic radiation through ionized gas excitation, enabling controlled aging studies.

This study evaluates the coagulation process using aluminum sulfate ($Al_2(SO_4)_3$) for removing pristine (time-zero) and aged (30, 90, and 120 days) MPs, analyzing their granulometric distribution.

METHODS

Coagulation assays were conducted using a Jar Test apparatus, with turbidity as the key parameter for flocculation efficiency. Treated samples underwent laser granulometry to assess particle size distribution (Mz). All experiments were performed at the Federal University of Goiás, in the Sanitation and Geotechnics Laboratories of the School of Civil and Environmental Engineering (EECA).

The experimental setup included a bench-scale jar test apparatus, a turbidimeter, a pH meter, and a laser diffraction particle size analyzer for granulometric characterization. Laboratory glassware comprised 1000 mL low-form beakers, graduated pipettes, and other standard items. Reagents and solutions used in the coagulation assays included a 1% (w/v) $Al_2(SO_4)_3$ solution, hydrochloric acid (HCl) at a concentration of 1 mol L^{-1}, deionized water, and distilled water. The microplastics (MPs) employed in the study consisted of polyethylene (PE) particles with a nominal size of 0.6 mm, both in pristine condition and

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artificially aged. The aging process was conducted in a dark chamber using UVC irradiation provided by a transilluminator operating at a wavelength of 254 nm.

Treatability assay

Several coagulation conditions were evaluated for the removal of polyethylene (PE) microplastics (MPs), considering a fixed particle size of 0.6 mm and an initial concentration of 400 mg L^{-1}. Both pristine MPs and those subjected to artificial aging for 30, 90, and 120 days were tested. Aging was performed by UVC irradiation at a wavelength of 254 nm in a dark chamber for 15 days (360 hours), following the procedures described by de Oliveira (De Oliveira et al., 2023a) and (Guan et al., 2022). The coagulation experiments were conducted using $Al_2(SO_4)_3$ at concentrations of 2.50, 4.50, and 6.00 mg L^{-1} as the primary independent variable.

Rapid mixing was performed at 400 rpm for 1 minute, followed by slow mixing at 100 rpm for 15 minutes. Subsequently, a sedimentation period of 30 minutes was allowed. Microplastic samples were collected from a depth of 3 cm below the water surface, corresponding to a calculated sedimentation velocity of 0.1 cm min^{-1}, as reported by de De Oliveira et al. (2023b).

The effectiveness of the coagulation process was assessed by measuring turbidity before and after coagulant addition to determine the MP removal efficiency. Additionally, the granulometric distribution of the PE MPs was characterized according to particle size following the Brazilian standard NBR 7181.

Granulometric analysis was performed to determine the weight percentage of different particle size fractions in both untreated and coagulated samples. The particle size analyzer operates on laser diffraction principles, equipped with three red lasers (780 nm) and capable of measuring particles ranging from 0.02 µm to 2.8 mm. Particle size distribution curves were generated using both Mie and Fraunhofer diffraction models, providing high-resolution detection over a wide particle size range. The instrument employs a detector array illuminated by neon and helium light sources, where the laser wavelength determines the particle size measurement range.

RESULTS AND DISCUSSION

For the calculation and determination of MP removal efficiency, initial and final turbidity values were used, along with pH measurements, under predefined concentrations

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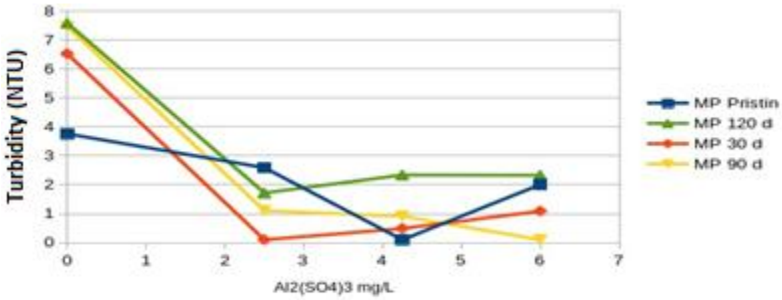
of $Al_2(SO_4)_3$, following the methodology described by (De Oliveira et al., 2023a). All experimental assays were conducted in up to five replicates to ensure reproducibility.

To characterize the formation of chemical flocs, the ionic strength—or total ion concentration of the solution—was considered, along with pH. Higher coagulant dosages and lower pH values were associated with increased MP removal efficiencies, particularly for MPs smaller than 0.5 mm.

Factorial matrix analysis of the bibliographic reference data indicated that lower final pH values, combined with predefined salt-based coagulant concentrations, resulted in higher MP removal efficiencies in the experiments conducted at the pilot-scale facility.

The present study confirmed the effectiveness of MP removal through acid-mediated salt hydrolysis using aluminum sulfate at increased dosages and low pH conditions during MP–coagulant interaction. During the rapid mixing phase, aluminum cations (Al^{3+}) and sulfate anions (SO_4^{2-}) dissociated, accompanied by water ionization. This molecular dissociation promoted charge destabilization in the suspension, enhancing MP aggregation through particle collision and floc formation, followed by sedimentation, thereby facilitating the removal of the generated flocs. Final turbidity measurements were used to determine the optimal coagulation efficiencies for MP removal using aluminum sulfate at the tested concentrations (mg L^{-1}), as further discussed and presented in Figures 1, 2, and 3.

Figure 1: Turbidity Parameters in the Treatability Test of MPs



In Figure 1, pristine MPs with an aluminum sulfate dosage of 4.25 mg·L^{-1}, MPs aged 30 days with the same coagulant at 2.25 mg·L^{-1}, and MPs aged 90 days at a dosage of 6 mg·L^{-1} showed better performance and reached a final turbidity (NTU) of 0.10. Based on these empirical data, the removal efficiency was calculated to construct Figure 2.

Figure 2: Treatability Efficiency of MPs

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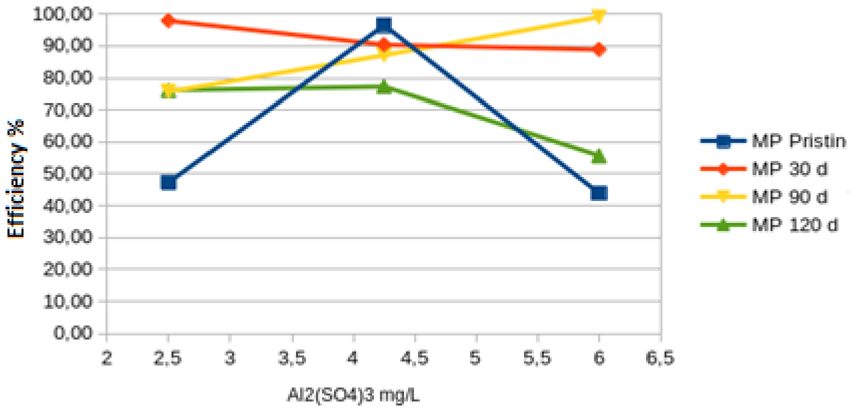
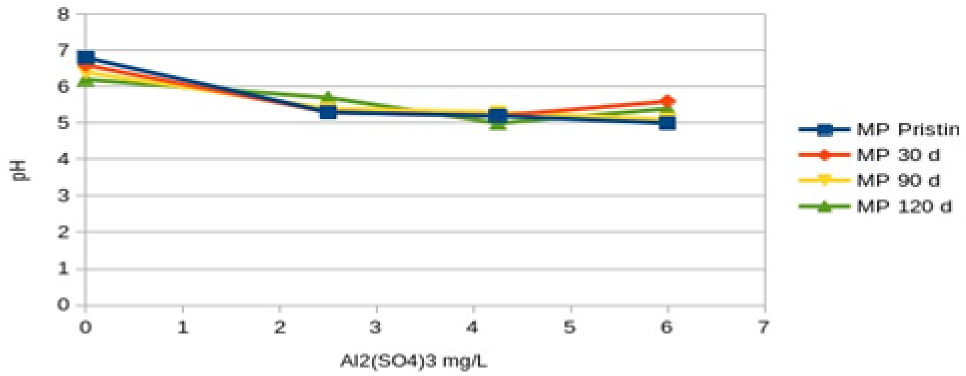


In this context, analyzing the percentage removal efficiency based on the coagulant dosage applied, the best result was obtained for MPs aged 90 days at 6 mg·L⁻¹, with a removal efficiency of 99.07%, followed by MPs aged 30 days at 2.5 mg·L⁻¹ with 97.88%, and pristine MPs at 4.25 mg·L⁻¹ with 96.47% efficiency. MPs aged 120 days showed comparatively moderate efficiency of 77.28% at a coagulant dosage of 4.25 mg·L⁻¹.

Figure 3: pH Parameter in the Treatability Test of MPs



In the previous figure, as reported in the study by (De Oliveira et al., 2023a) on the influence of pH on MP treatment efficiency, these tests also showed improved treatability under acidic pH conditions.

Therefore, the treatability tests presented in the graphs above demonstrated higher efficiencies for MPs aged up to 90 days. In contrast, the tests conducted with MPs aged 120 days showed a significant decrease in treatment efficiency. Consequently, the data indicate that the longer the exposure time of the MPs, the lower their removal efficiency through the coagulation process.

In addition to the treatability tests mentioned above, the effect of the coagulant on MP particle size was evaluated using laser diffraction granulometry, considering the Mean

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Diameter (Mz) parameter at a fixed aluminum sulfate concentration of 4.25 mg·L^{-1} for the aged MPs from the third test. A comparison of MP particle sizes is presented below in Table 1.

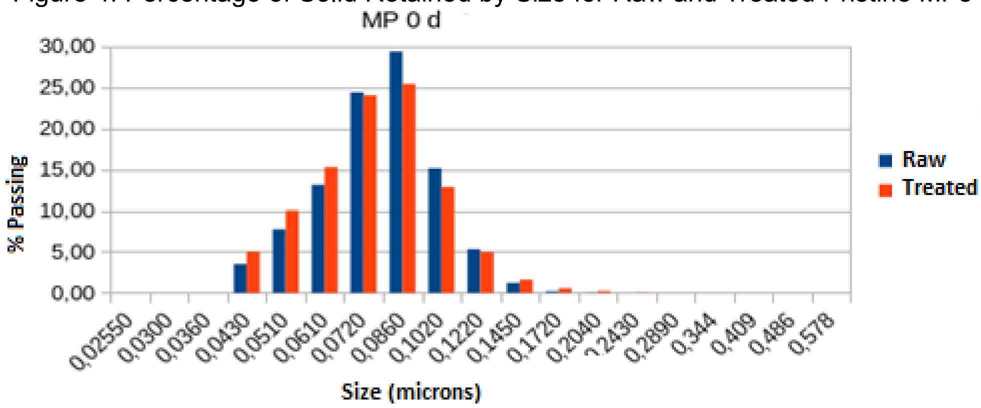
Table 1: Comparison of MP Sizes Based on Mean Diameter (Mz)

TEMPO DE ENVELHECIMENTO (DIAS)	Mz BRUTO (µm)	Mz TRATADO (µm)	TRATADO/ BRUTO	PORCENTAGEM
0	0,0877	0,0924	1,05	5%
30	0,0897	0,0870	0,97	-3%
90	0,0900	0,2260	2,51	151%
120	0,0812	0,0924	1,14	14%

It was observed that the most pronounced floc formation occurred in MPs aged for 90 days, where the Mz increased by 151%, thereby explaining their easier removal and the high removal efficiency achieved. However, it is noteworthy that in the experiment with MPs aged for 30 days, a reduction in particle size was observed—a behavior not seen in the other MP samples. This result may be attributed to the instability of the 30-day aged MPs when treated with $Al_2(SO_4)_3$, as evidenced during the granulometric analysis. In this analysis, sample agitation is induced by pumping within the mixing chamber of the laser diffraction analyzer to ensure homogenization. For the other aging periods of 0 and 120 days, no significant increase in Mz was observed.

Figures 4, 5, and 6 below present the effect of treatment on MP particle size, detailing the sections in micrometers in the determination of particle size distribution. This analysis is based on the standard laser diffraction technique, which involves exposing the particles to a monochromatic light beam, as performed in each respective test.

Figure 4: Percentage of Solid Retained by Size for Raw and Treated Pristine MPs



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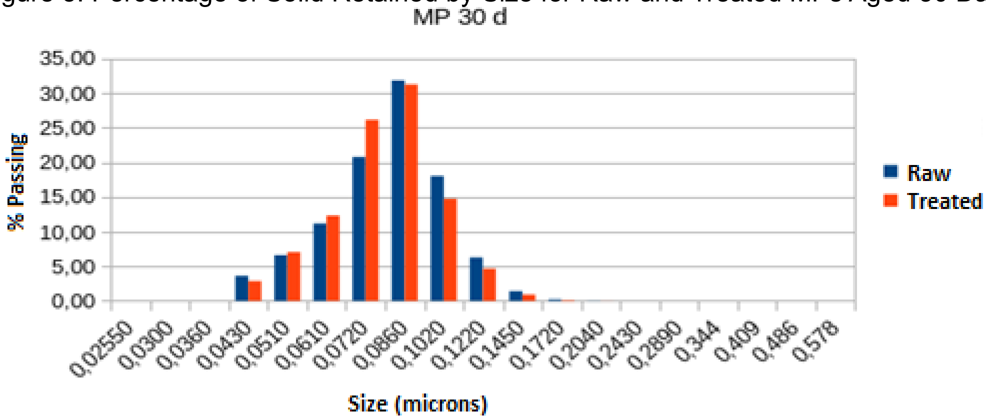
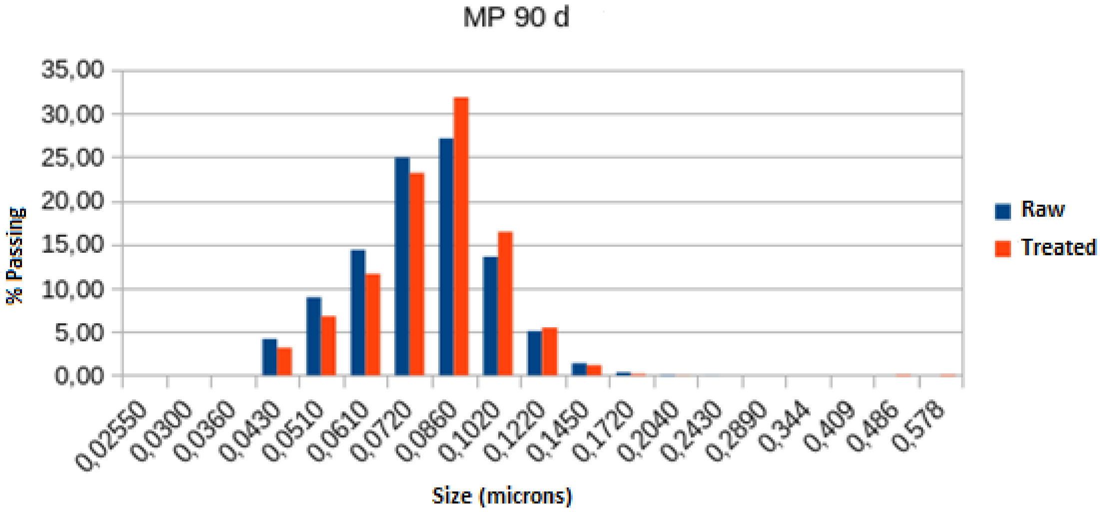
In Figure 4, which represents the pristine MPs, a slight increase was observed in the proportion of particles larger than 0.1450 µm. For the size range between 0.0720 µm and 0.0860 µm, a decrease in the number of MPs within this interval was noted, indicating that particles in this range underwent fragmentation. This fragmentation resulted in an increased quantity of particles smaller than 0.0720 µm.

Figure 5: Percentage of Solid Retained by Size for Raw and Treated MPs Aged 30 Days



In Figure 5, representing MPs aged for 30 days, a reduction in the number of particles larger than 0.086 µm was observed, indicating that these particles underwent fragmentation, which in turn increased the quantity of particles below 0.0860 µm. A 3% decrease in the mean particle size was recorded.

Figure 6: Percentage of Solid Retained by Size for Raw and Treated MPs Aged 90 Days



In Figure 6, representing MPs aged for 90 days, an increase in the mean particle size (Mz) was observed based on the obtained results. For particle sizes above 0.0860 µm,

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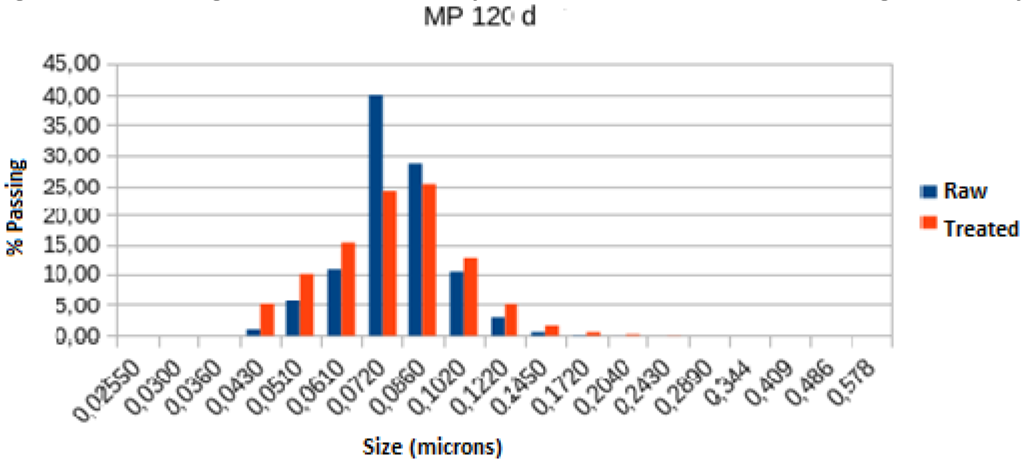
a higher percentage was found in the treated sample compared to the raw sample. This increase is attributed to the coagulation of a significant portion of the particles smaller than 0.0860 µm.

Figure 7: Percentage of Solid Retained by Size for Raw and Treated MPs Aged 120 Days



In Figure 7, representing MPs aged for 120 days, a slight increase was observed in the proportion of particles larger than 0.1020 µm. For the range between 0.0720 µm and 0.0860 µm, a decrease in the number of MPs was noted, indicating that these particles underwent fragmentation, which resulted in a significant increase in the proportion of particles smaller than 0.0720 µm. This behavior demonstrates resistance to the action of the coagulant, consequently hindering their removal.

4 CONCLUSIONS

The coagulation process for the removal of polyethylene (PE) microplastics (MPs) proved to be effective using $Al_2(SO_4)_3$ at dosages ranging from 2.5 to 6.00 $mg \cdot L^{-1}$ for pristine MPs and MPs aged for 30 and 90 days, achieving removal efficiencies between 90.37% and 99.07%. The highest efficiency was observed in MPs aged 90 days, as confirmed by laser diffraction granulometry, which indicated a 151% increase in the mean particle diameter (Mz), thereby confirming the improved treatability of MPs with $Al_2(SO_4)_3$ and the high removal efficiency.

However, the coagulation process using $Al_2(SO_4)_3$ at concentrations up to 6.00 $mg \cdot L^{-1}$ was not effective for MPs aged 120 days, indicating that the more deteriorated the MPs are, the more difficult their removal becomes through coagulation.

Further experimental studies are recommended involving coagulation of polystyrene, polypropylene, polyethylene terephthalate, polyvinyl chloride, and other plastic components in both pristine and aged conditions. These studies should test aluminum sulfate at variable dosages as well as alternative coagulants, such as ferrofluid, polyaluminum chloride (PAC), anionic/cationic polyacrylamide, and tannin-based coagulants.

ACKNOWLEDGEMENTS

The authors want to acknowledge the Fundação de Amparo à Pesquisa do Estado de Goiás for the financial support to this research.

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