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Coagulation of Wastewater Containing Polyethylene Terephthalate (PET) Microplastics by Using Ferric Chloride, Aluminum Sulfate and Aluminum Chlorohydrate: A Comparative Study

Lilya Irsianti Fadlilah[1], Arseto Yekti Bagastyo[2]

[1,2] Department of Environmental Engineering, Faculty of Civil, Planning, and Geo-Engineering, Institut Teknologi Sepuluh Nopember, Kampus ITS Sukolilo, Surabaya, 60111, Indonesia

ABSTRACT: This study investigated the efficacy of different coagulants, namely ferric chloride and aluminum sulfate at a concentration of 1.95 g/L, and aluminum chlorohydrate at a concentration of 2.1 g/L, in removing microplastics from plastic recycling facility wastewater under different pH conditions. The results showed that the presence of microplastics in wastewater was 76% in the form of fragments, followed by films and fibers in smaller amounts. The size of these microplastics varies, with a dominant size range of 251-500 µm. At pH 6, ferric chloride and aluminum sulfate were able to remove 75% and 90% of the microplastic abundance, respectively, by adding a dose of 1.95 g/L. However, the highest removal efficiency was obtained by adding a dose of 2.1 g/L Aluminium chlorohydrate at pH 8. These findings underscore the importance of selecting suitable coagulants and optimizing treatment operational conditions based on the type and size distribution of microplastics present in the wastewater system.

KEYWORDS: aluminum chlorohydrate, aluminum sulfate, coagulation-flocculation, ferric chloride, microplastics

I. INTRODUCTION

Research has indicated that microplastics, defined as plastic particles ranging from 1 µm to 5 mm in size, are increasingly prevalent across various ecosystems worldwide (Brown et al., 2023). Studies suggest that microplastics can travel through aquatic systems from urban areas to rivers and oceans, and are also transported via atmospheric systems, carrying them from land to sea. This makes the oceans a primary route for microplastic transport globally (Su et al., 2022). Recent research underscores the necessity of global cooperation to reduce plastic waste at all stages of its lifecycle to combat the growing environmental plastic pollution through mechanical recycling (Borrelle et al., 2020;Lau et al., 2020). This mechanical process is also a key factor in the degradation of plastic into smaller sizes, known as microplastics (MP).

Plastic recycling facilities offer waste management practices where plastics are sorted by type, shredded, and converted into granules, which are then processed into pellets for reuse. This process involves mechanical friction, abrasion, or similar methods that can increase the concentration of microplastics in the wash water used and discarded during recycling (Altieri et al., 2021).While plastic recycling facilities are expected to reduce the spread of microplastics in the environment, they inadvertently contribute to microplastic pollution because the wastewater still contains microplastics. Concentrations of 5.97×106 to 1.12×108 m-3 microplastics have been detected in the wastewater generated from plastic recycling facilities (Brown et al., 2023). There has been limited research on the release of microplastic pollution from recycling facility wastewater. In addition, there is insufficient knowledge about how these facilities contribute to environmental plastic pollution (Brown et al., 2023).

The removal of microplastics using the coagulation-flocculation method has been conducted by Lu et al. (2021), who studied the effluent of a wastewater treatment plant in China. The type of microplastic targeted for removal was polyethylene terephthalate (PET) using coagulation with alum as the coagulant. The alum used was 0.1 mmol/L at pH 7, which was able to remove 100% of the PET microplastics. In addition to wastewater, microplastics have also been detected in the effluent of drinking water treatment plants (DWTP), as investigated (Prokopova et al., 2021). The type of microplastic present in the samples was polyvinyl chloride (PVC). The removal method used was coagulation, with $Fe_2(SO_4)_3 \cdot 9\ H_2O$ as the coagulant. The coagulant was used at a concentration of 40–60 mg/L with a pH of 5-8, which was able to remove 80% of the microplastics.

II. MATERIALS AND METHOD

A. Coagulation-flocculation experiment

The wastewater samples used in this study were taken from the influent of wastewater treatment plant in the plastic recycling facility, located in West Java. The coagulants used and tested in this study were Ferric chloride ($FeCl_3$) and aluminum sulfate ($AL_2(SO_4)_3$) at 39% (w/v), aluminum chlorohydrate (ACH) at 21-22% (w/v). Anionic polyacrylamide (PAM) was used as a flocculant. Sodium hydroxide (NaOH) at 40% and sulfuric acid (H_2SO_4) at 40% were used to adjust the working pH. The coagulation-flocculation experiment was performed using a jar test with a working volume of 1000 mL (1L) in the beaker glass. The experiments were carried out by varying the dosage and working pH, where the dose variations used were 5 mL (1.95 g/L), 10 mL (3.9 g/L), and 15 mL (5.85 g/L) for aluminum sulfate and ferric chloride coagulants. As for the type of aluminum chlorohydrate coagulant, the dose variations used are 5 mL (1.05 g/L), 10 mL (2.1 g/L), and 15 mL (3.1 g/L) while the pH variations were at a working pH of 6, 7, and 8. The jar tests were conducted at 251 rpm of rapid mixing, followed by 39 rpm of slow mixing, before allowing the samples for 30 minutes of the sedimentation process. Then, the samples were undergone the wet peroxide oxidation (WPO) process.

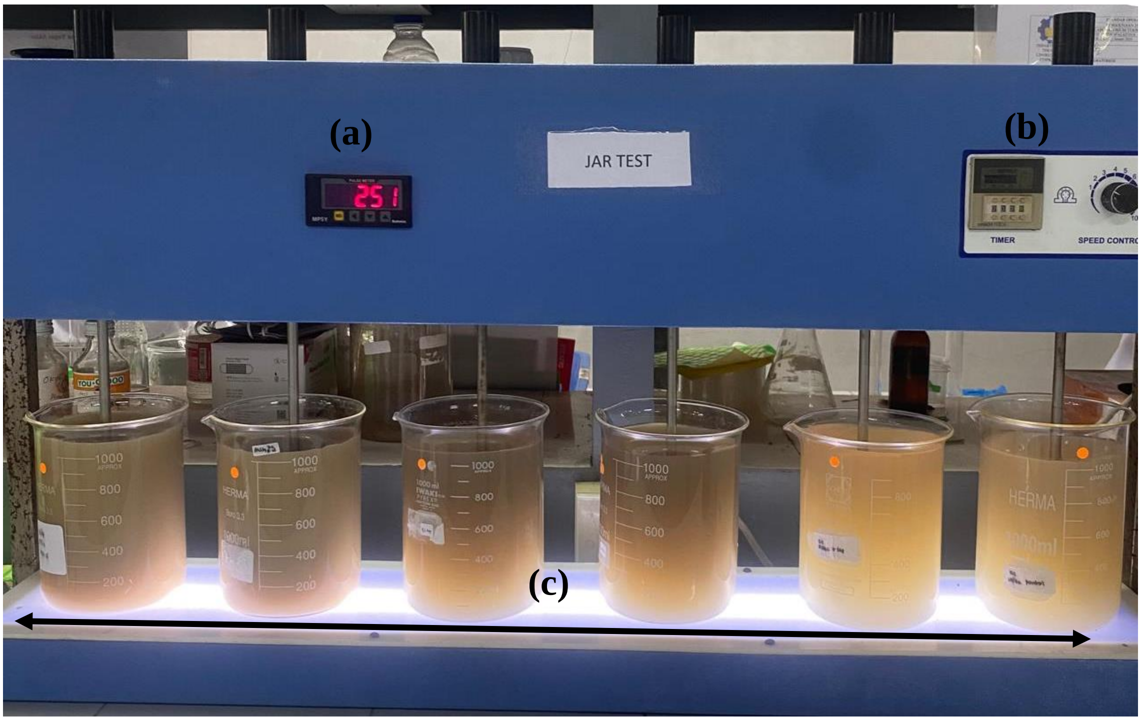


Figure 1: Jar test setup. (a) mixing speed, (b) time, (c) 1 L sample in beaker glass

B. Sample preparation for microplastic analysis

The sample extraction in this experiment uses a modified method from the National Oceanic and Atmospheric Administration (NOAA). First, samples from the coagulation-flocculation sedimentation process were separated between the supernatant and the sediment. The sediment sample was heated at 150 °C for 24 hours and oxidized using the Wet Peroxide Oxidation (WPO) method by dissolving it in 250 mL of distilled water. The WPO method was carried out by adding ± 30 mL of 30% H_2O_2 to remove the organic content in the sample, depending on the organic concentrations. After that, the sample was allowed to stand for ± 5 minutes and heated at 75 °C for 30 minutes. Following this, the density separation was carried out by adding 6 g NaCl per 20 mL, and then stirred using a hot stirrer at 300 rpm with a temperature of 75 °C for ± 1 hour. The addition of NaCl is intended to increase the density of the aqueous solution to 1.15 g/mL (Masura Julie et al., 2015), so that microplastic particles can float more easily during separation.

The sample was then filtered using hydrophobic PTFE membrane filter with a pore size of 0.22 µm and a diameter of 47 mm. The membrane filter was then kept overnight before being analyzed by using a microscope (Olympus Corporation Model SZ2-ILST with a magnification of 6.7x–35x and an OptiLab camera).

C. *Microplastic abundance*

The abundance of microplastics dispersed in the samole can be calculated using the formula (Masura Julie et al., 2015). Analysis of microplastic abundance in water and sediment were determined by equations (1) and (2).

Analysis of microplastic abundance in water:

$$K = n/V \qquad (1)$$

Microplastic abundance, denoted as K, is measured in particles per liter (Particles L-1). This calculation is based on the number of particles observed, denoted as n, and the volume of water filtered, denoted as V. By determining the number of microplastic particles in each volume of water, researchers can measure the concentration of microplastics in that sample.

Analysis of microplastic abundance in sediment:

$$K = n/V \qquad (2)$$

K is the abundance of microplastics measured in kilograms of particles (Particles kg^{-1}). The K value is calculated by dividing the number of observed microplastic particles (n) by the dry sediment weight (m) in kilograms. In this formula, n is the number of particles found in the observation, while m is the weight of the dry sediment used as a sample. K thus gives an idea of how many microplastics are present in one kilogram of dry sediment.

III. RESULTS AND DISCUSSION

A. *Coagulants*

1) ***Ferric chloride:*** In water treatment, one of the important stages is coagulation. In this stage, colloid particles floating in the water are collected so that it can settle and be removed. Coagulation, a crucial stage in water treatment, gathers colloidal particles for eventual settling and removal. Ferric chloride ($FeCl_3$) is a common coagulant used in this process. When ferric chloride ($FeCl_3$) is dissolved in water, it dissociates into ferric ions (Fe^{3+}) and chloride ions (Cl-). The iron ions then undergo hydrolysis, reacting with water to form ferric hydroxide ($Fe(OH)_3$) and release hydrogen ions (H^+), which can lower the pH of the solution (Khan et al., 2023). This formation of iron hydroxide is very important in the water treatment process as it acts as a coagulant, helping to aggregate colloidal particles and enhance their flocculation, thereby easing the removal process from the water.

Coagulants such as ferric chloride can successfully remove microplastics from water. This coagulant ability is influenced by several components, including pH and the dose of coagulant used (Iswanto et al., 2009). In the results found, it can be seen that at pH 6 with a measurement of 5 mL (1.95 g/L), the removal rate reached 75%, indicating a high removal ability under acidic conditions. However, when the pH was increased to 7 and 8, the removal ability decreased to 46.02% and 19.3% for the same dose, as shown in **Figure 1**

Using a coagulant dose of 10 mL (3.9 g/L) at pH 6 caused the removal to drop to 65.7%. However, at pH 7 the effectiveness increased to 73.33%. This indicates that increasing the coagulant dose seems to widen the operating pH range, but this does not seem too significant considering that the coagulant works well over a wide pH range (Iswanto et al., 2009). Using a coagulant dose of 15 mL (5.85 g/L), the removal at pH 6 dropped to 62.2%, while at pH 7 and 8, the effectiveness was stable at around 58.33% and 51.19%. This may indicate that there is an optimum limit for ferric chloride dosage, as increasing the dosage does not necessarily increase the effectiveness of microplastic removal (Khan et al., 2023).

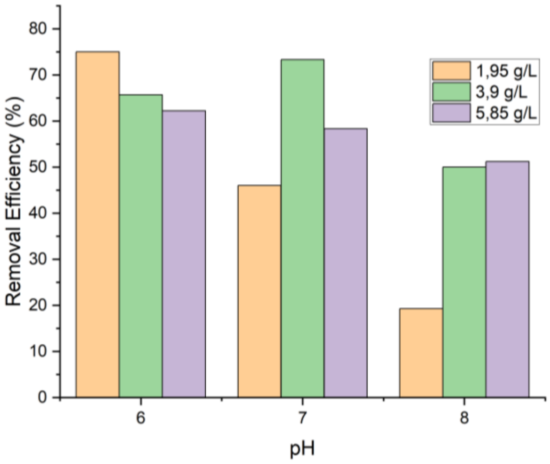


Figure 1: Effect of dose and pH on microplastic removal with $FeCl_3$ coagulant

Based on a study by Rajala et al. (2020), the usage of ferric chloride in removing microplastics can be optimized by choosing the right coagulant and pH. Other research results also state that variables such as the density of certain particles in water can affect coagulation productivity (Zhang et al., 2021). It can be concluded that ferric chloride has potential as a coagulant for microplastic removal, but the removal ability is highly dependent on operational conditions such as pH and dosage. Other observations are needed to optimize these conditions to achieve higher removal efficiency.

2) ***Aluminum chlorohydrate*** **:** Aluminum Chlorohydrate (ACH) is a pre-hydrolyzed aluminum-based coagulant and contains positively charged polymeric aluminum species, including $AlO_4Al_{12}(OH)_{24}(H_2O)_{12}^{7+}$ and $[Al_{30}O_8(OH)_{56}(H_2O)_{24}]^{18+}$, known as Al_{13} and Al_{30} teams. Unlike the monomeric Al hydroxide from alum, the cationic type produced from ACH is more stable over a wide range of pHs (Lapointe et al., 2021). ACH acts as a coagulant by destabilizing colloidal particles in water through charge neutralization and coagulation mechanisms. When ACH is added to water, it forms positively charged aluminum hydroxide (Al_2O_3) flocs. These flocs attract negatively charged particles in the water, such as suspended solids and organic matter, causing them to stick together. When these flakes get bigger, they settle in the water, so turbidity and other impurities can be removed (Omar & Aziz, 2021).

In water treatment, a coagulation-flocculation strategy that utilizes aluminum chlorohydrate (ACH) as a coagulant has potential in the removal of microplastics. Research conducted at pH 6, 7, and 8 and doses of 5 mL (1.05 g/L), 10 mL (2.1 g/L), and 15 mL (3.1 g/L) provided information on the efficiency of ACH under several conditions. **Figure *2*** displays the removal results for ACH coagulant.

At a lower dose of 5 mL (1.05 g/L), the removal ability reached its highest point at pH 7 with a value of 44.17%. This result was influenced by both pH and the dose of coagulant added. This is in accordance with other studies showing that alum-based coagulants are less effective at high pH (Lapointe et al., 2021). In addition, if the coagulant dose is too low, the particles in the water will not be sufficiently destabilized to allow effective flocculation. If the dosage is too much, the excess coagulant can instead destabilize the particles, thus disrupting the process (Filipkowska et al., 2019).

Increasing the ACH dosage to 10 mL (2.1 g/L) further increased its effectiveness, especially at pH 8 (96.43%), which is in agreement with the finding that polynuclear aluminum coagulants such as ACH can achieve high removal rates at alkaline pH levels (Farraj et al., 2024). At the highest tested measurement of 15 mL (3.1 g/L), there was a decrease in removal at pH 8 (16%), which may be due to an overdose of coagulant that promoted restabilization of the colloid. This result has been observed in other observations where excessive coagulant dosage has led to incomplete removal efficiency (Khan et al., 2023).

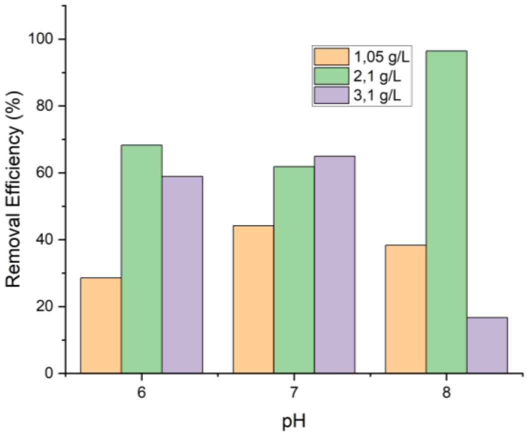


Figure 2 Effect of dose and pH on microplastic removal with ACH coagulant

3) ***Aluminum sulfate*** **:** Aluminum sulfate has been investigated as a material to remove microplastics from water by coagulation-flocculation. The results show that aluminum sulfate is quite effective under certain conditions (Khan et al., 2023). When aluminum is dissolved in water, it undergoes hydrolysis, forming aluminum hydroxide and releasing hydrogen ions (Khan et al., 2023). The aluminum hydroxide formed can then interact with other ions in the water, leading to the formation of various aluminum species, which can affect the pH and overall chemistry of the water. This hydrolysis is very important in the water treatment process, as it contributes to the coagulation and flocculation of suspended particles, including microplastics.

Aluminum sulfate has been investigated as a material to remove microplastics from water by coagulation-flocculation. The results show that aluminum sulfate is effective under specific conditions (Khan et al., 2023). This study found that aluminum sulfate is effective in removing microplastics at pH 6 with a dose of 5 mL (1.95 g/L), which is 90%. This is in line with the observations of Farraj et al. (2024) higher removal efficiency at lower pH levels when using alum-based coagulants. However, the higher the pH of the water (neutral to alkaline, pH 7 and 8), the ability of aluminum sulfate to remove microplastics decreased. These results are in agreement with previous studies showing that microplastic removal was significantly reduced at higher pH values when alum was used as a coagulant (Lapointe et al., 2021).

At doses of 10 mL(3.9 g/L) and 15 mL(5.85 g/L), the effectivity of aluminum sulfate did not show an obvious pattern at different pH. High doses of aluminum sulfate do not always result in a significant increase in effectiveness due to different factors, including colloid saturation point, interaction between colloids, floc formation, pH effects, and microplastic characteristics (Shahi et al., 2020; Rajala et al., 2020). At a dose of 10 mL, its efficiency at pH 7 dropped significantly (only 18.75%). This may be because the ideal process for alum does not apply at higher concentrations. This decrease was also observed in other studies where excessive coagulant doses led to a decrease in effective (Khan et al., 2023).

This study showed that at a dose of 15 mL, aluminum sulfate did not improve its ability to remove microplastics compared to lower doses. This may occur for several reasons, i.e. an overload of aluminum sulfate may cause the microplastics that are bound to re-release and return to the water, the overload of aluminum sulfate may not decompose properly, so it cannot bond and precipitate all microplastics (Lee & Jung, 2022). Removal of microplastics with ($Al_2(SO_4)_3$) coagulant is shown in the

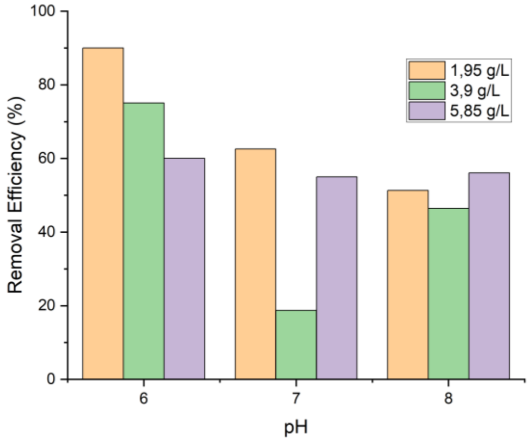


Figure 3. Effect of dose and pH on microplastic removal with $Al_2(SO_4)_3$ coagulant

Furthermore, microplastic removal by aluminum sulfate coagulant, seems to be affected by several factors, including solution pH, coagulant dosage, and the physical characteristics of microplastics. Optimizing these parameters is important to increase removal efficiency and reduce operational costs (Khan et al., 2023). Solution pH is critical as it determines the surface charge of the coagulant and the contaminants to be removed (Burhani et al., 2017).

The interaction of coagulants with microplastics is affected by pH. Low pH supports coagulation by increasing the positive charge of the coagulant, neutralizing microplastics, and enhancing removal (Khan et al., 2023). In opposition, a higher pH decreases efficiency as the neutralization capacity is reduced. It was observed that a higher pH was associated with lower removal efficiency. Although increasing the dose of aluminum sulfate can help, there is an optimal limit where further dosing does not increase effectiveness and could lead to restabilization of the colloid (Naceradska et al., 2019).

In conclusion, although aluminum sulfate proved to be an effective coagulant for microplastic removal under certain conditions, its performance is highly dependent on environmental factors such as pH and dosage. The challenge will be to fine-tune these parameters to achieve optimal results across different water tissue types and microplastic contaminants.

B. MP characteristics

1) ***Microplastic size distribution***: The abundance of microplastics by size is shown in **Figure 4**. Based on Figure 5, the sample mostly consists of microplastics between 251µm and 500µm in size. This observation is in line with the findings of Sun et al. (2019) that microplastics in wastewater are mostly ≤ 500 µm, with a percentage reaching 90%. The smallest microplastics ranged from 1001 µm to 5000 µm.

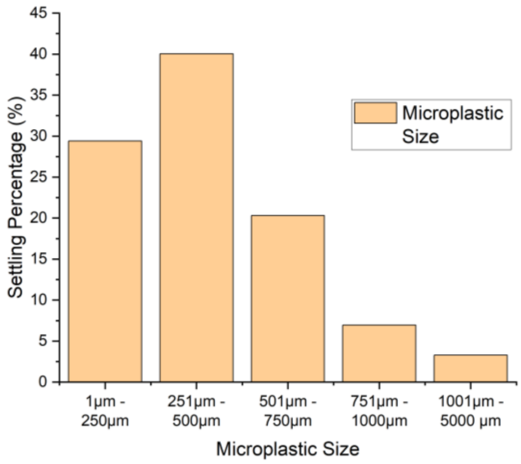
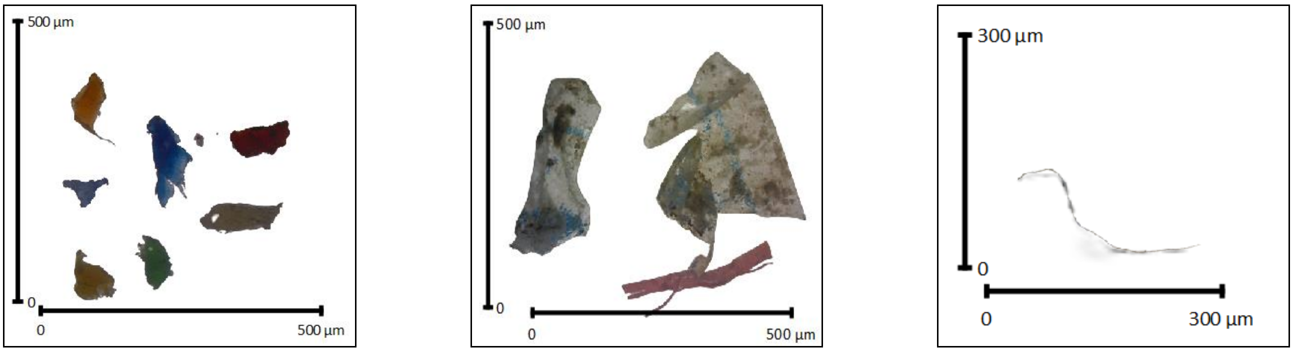


Figure 4 The classification of microplastic sizes

Size is one of the important factors that can influence the distribution of MPs in the water column (Schwarz et al., 2019). However, this size may be reduced step-by-step due to degradation mechanisms in the river, such as photodegradation, mechanical-physical processes, weathering, and biodegradation (Kooi et al., 2018). Therefore, the longer plastic waste is affected by these degradation mechanisms, the smaller the particles become (Firdaus et al., 2020). In addition, the specific amount of microplastics can have various impacts. One such impact is that Large Microplastic Particle (LMP) sizes ranging from 1 to 5 mm can pose risks to water treatment plants (WTPs) that use rivers as the main water source. For example, the efficiency of the filtration process to remove MPs from WTPs is determined by particle size (Marsden et al., 2019). Another impact is that smaller particles are more susceptible to ingestion by different aquatic organisms (Cole et al., 2013).

2) ***Classification of microplastic shapes***: The form of MP consists of 5 types, namely film, fragments, fibers, foam, and pellets (Lestari et al., 2020). The observation results show that microplastics are mostly in the form of fragments, characterized by a jagged and hard shape, usually coming from bottles and strong and thick plastic materials (Free et al., 2014). The shape of microplastics is mostly fragments, followed by films and fibers.

Fragmentscin the sediment accounted for 76%, followed by films at 21% and fibers at 1.3%. The various forms of microplastics observed are depicted in **Figure 5**



(a) (b) (c)

Figure 5 Stereo shapes of the MPs: (a) fragment, b) film, c) fiber

IV. CONCLUSION

Research indicates that the coagulation-flocculation method can effectively remove microplastics from water of various sizes. This process involves the formation of larger microplastic flocs that can be easily filtered or removed from the water. Three types of coagulants studied (ferric chloride, aluminum chlorohydrate, aluminum sulfate) exhibit different abilities in removing microplastics, depending on the water conditions being treated. The optimal use of each coagulant depends on parameters such as pH and coagulant dosage. Microplastics can vary in shape (such as fragments, fibers, films) and size depending on the type and material of plastics involved in the treatment process. Understanding these variations is crucial for designing effective water treatment strategies.

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