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Preliminary Study of Reduction of Microplastics Contained in Salt Produced in Padang, Indonesia

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ABSTRACT

Salt produced from seawater evaporation contains harmful microplastics (MP). For this reason, a technology that can remove MP from seawater using coagulation and filtration techniques is needed. The purpose of this study is to utilize alum as a coagulant and sand as a filtration media to reduce MP pollution in seawater as a source of raw material for salt making. Seawater from Buo Bay, Padang City, Indonesia was taken as raw material for salt production. The MP abundance of salt made from seawater without alum and sand treatment was found to be 400 particles/kg. To reduce the abundance of MP in the salt, we varied the alum concentration (0.1; 0.3; and 0.5 g/L) and sand particle size (≥2, ≥1-<2, and <1 mm). From the results obtained, the optimal condition is an alum concentration of 0.5 g/L and sand particle size is <1 mm. The optimal condition of salt made from seawater in treatment H obtained MP abundance from 400 particles/kg to 30 particles/kg with an MP reduction efficiency of 92.5%. Visual analysis using optical trinocular microscopy found 4 forms of MP, namely: fragments (51.13%), fibers (28.95%), films (15.41%), and pellets (4.05%). Rewith the most dominant MP size found was >100-300 µm. The results of ATR-FTIR analysis identified the types of MP as Polyethylene (14.28%), Polyethylene Terephthalate (42.85%), Polypropylene (14.28%), and Polyamide (28.57%).

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INTRODUCTION

Plastic is a synthetic polymer from crude oil and natural gas (S. K. Kim et al., 2021; Peixoto et al., 2019). The hydrocarbon chains in plastic make plastic waste difficult to decompose in the environment. Plastic will be fragmented into smaller pieces through various processes (Iñiguez et al., 2017; S. K. Kim et al., 2021). Plastic fragments into pieces smaller than 5mm are called microplastic or MP (J. S. Kim et al., 2018; Lippiatt et al., 2013). MP can be categorized based on its shape viz: fragments (irregular shape), fibers (long and thin like yarn), films (transparent and flat), and pellets (perfectly round). MP can also be categorized based on the type of constituent polymer such as polyethylene (PE), polypropylene (PP), polyamide (PA), polyvinyl chloride (PVC), and others (Vidyasakar et al., 2021).

Salt is one of the marine products consumed by humans. The World Health Organization

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(WHO) has recommended a salt intake of 5 grams per day for adults, as salt is required for osmotic balance in human cells (Selvam et al., 2020). Based on its origin, salt is categorized into sea salt, lake salt, rock salt, river salt, and borehole salt (D. Yang et al., 2015). Sea salt is made by evaporating seawater and forming salt crystals. Research in various countries informs that sea salt has been contaminated with MP (Iñiguez et al., 2017; Karami et al., 2017; Sari et al., 2017). MP-contaminated salt will reach humans through the food chain and cause health problems in humans (Peixoto et al., 2019). D. Yang et al., (2015) examined the abundance of MP in 15 brands of commercial salt circulating in Chinese supermarkets, and MP was found in all samples analyzed with an abundance in sea salt of 550-681 particles/kg. Karami et al., (2017), also examined 17 salt brands from eight countries (Australia, France, Iran, Japan, Malaysia, New Zealand, Portugal, and South Africa) and found that they were contaminated with MP. Ha (2021), also examined MP contamination in 9 brands of sea salt from Vietnam, with an average MP abundance of 878±101 particles/kg for raw sea salt and 340±26 particles/kg for refined sea salt. India is the third largest salt producer after China and the United States (US), Eight brands of salt studied in India were contaminated with MP with particle concentrations ranging from 103±39 to 56±49 particles/kg (Seth et al., 2018). Iñiguez et al., (2017), examined the abundance and impact of MP in salt when consumed by humans but did not find a solution to overcome it. During the process of evaporation of MP-contaminated seawater into salt, it will reach the human body through the salt consumed (Peixoto et al., 2019; Ravikumar et al., 2023). Therefore, a technology that can reduce the abundance of MP from seawater for salt making is needed.

Plastic waste in the ocean will become small pieces and difficult to remove by conventional sedimentation (Talvitie et al., 2017). In addition, conventional sedimentation is costly to operate and does not match the selling price of salt (Edzwald et al., 2011). Other techniques commonly applied in solid-liquid separation are filtration, coagulation, and flocculation (Wei et al., 2018). In the coagulation process, colloids suspended in the liquid are destabilized by the coagulant and then collected and flocculated (B. Xu et al., 2011). The accumulated clots will be large, and the solid clots can be separated from the liquid phase by simple filtration or sedimentation. Coagulation techniques have advantages such as simple setup, and cost-effectiveness, and have also been widely used by the public (Mudunkotuwa et al., 2014).

Based on the above description, this study was conducted to (1) analyze the abundance of MP in salt made from Buo Bay seawater in Indonesia, before and after the use of coagulation and filtration techniques; and (2) obtain the optimal conditions of varying alum concentration and sand particle size as a simple method to obtain low MP salt raw materials.

MATERIAL & METHODS

Study site description. Buo Bay is located at 100°23'3.068" E; 1°3'53.341" S, one of the areas on the outskirts of Padang Beach which has the potential to become a marine tourism center because it has white sandy beaches and tobacco forests. Buo Bay is also the center of the mariculture industry, such as grouper and shrimp. Seawater that can be used as raw material for salt making has a salinity level of 30-40‰. In this study, Buo Bay seawater was chosen as raw material for salt making because the salinity level was 33‰ (Figure 1).

Seawater sampling. Seawater sampling and testing as raw material for salt production was conducted in June 2022. Water was pumped from the sampling location, and then filtered using a bag filter (100 μm). Seawater sampling was done by filling a 200 μm plankton net using a 5 L bucket. Filtered samples were transferred to sample bottles, and tagged with the date of collection (Viršek et al., 2016).

Salt Making Process. Salt farmers in West Sumatra add stimulant salt up to 100 grams/10 L of seawater to increase the salinity. The salt-making treatments in this study are summarized in Table 1. Commercial salt (n=5) circulating in Padang City and produced in December 2021 - March 2022 was also studied for MP abundance.

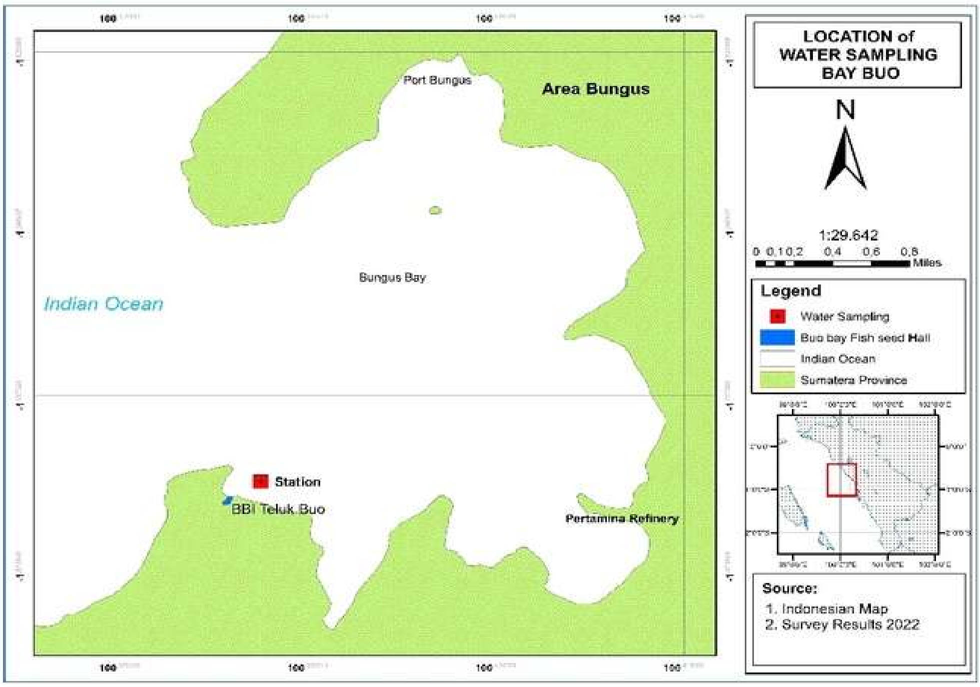

Fig. 1. Seawater intake station as raw material for making salt

Table 1. Treatments used to make salt

Treatment	Description
A	10 L seawater is heated to produce salt
B	Table salt (as stimulant salt)
C	Seawater 10 L + stimulant salt 100 grams + heated to produce salt.
D	Seawater 10 L + stimulant salt 100 grams + filtered (sand and fibers) + heated to produce salt.
E	Seawater 10 L + stimulant salt 100 grams + alum 1 gram as a coagulant, then allowed to stand for 2 hours; the supernatant is taken and heated to produce salt
F	Seawater 10 L + stimulant salt 100 grams + alum 1 gram as a coagulant, then allowed to stand for 2 hours, the supernatant is taken + filtered (sand and fibers) + heated to produce salt.
G	Seawater 10 L + stimulant salt 100 grams + alum 3 grams as a coagulant and then allowed to stand for 2 hours; the supernatant is taken + filtered (sand and fibers) + heated to produce salt.
H	Seawater 10 L + stimulant salt 100 grams + alum 5 grams as a coagulant, then allowed to stand for 2 hours; the supernatant was taken + filtered (sand and fibers) + heated to produce salt.

Sand filters are used to remove MP particles that pollute seawater with a simple design as shown in Figure 2. The sand particle sizes used as filters are ≥2mm, ≥1mm - <2mm, and <1mm. The screening unit is made of glass with a length of 15 cm, width of 15 cm, and height of 20 cm. The filtering stages are: (1) palm fiber is placed at the lowest position, (2) washed sea sand is placed on top, and (3) screening is carried out according to the treatment used.

Extraction of MP. To remove organic contaminants, 50 grams of salt sample was added to 100 mL of 30% H_2O_2 (Liebezeit et al., 2012). The mixture was stirred at 300 rpm at 60°C for 15 minutes, then allowed to cool. Next, 850 mL of distilled water was added and stirred again at 300 rpm for 15 minutes. The samples were left for 24 hours under closed conditions. The samples were then filtered with 0.45 µm filter paper using a vacuum pump (Millipore, 17 kPa). The filter paper was stored in a closed petri dish at room temperature for further identification (Iñiguez et al., 2017; Karami et al., 2017; Masura et al., 2015).

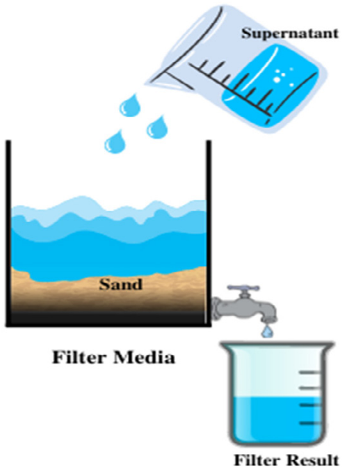

Fig. 2. Sand filter design

Identification of MP. Microplastic identification was conducted visually using a microscope (trinocular optics) connected to a camera. Visuals were observed directly through the monitor screen with 40 times magnification on the microscope. Visual identification of microplastics is grouped into four forms, namely films, fibers, fragments, and pellets (Hidalgo-Ruz et al., 2012). Salt samples from various treatments were identified, and the most dominant form was taken as a representative sample for polymer analysis. ATR-FTIR is the most popular and frequently used technique for functional group analysis in identifying polymer types MP. The observed functional groups were then analyzed to determine the chemical formula and molecular structure of the tested plastic monomers (Deswati et al., 2023; J. L. Xu et al., 2019).

Contamination Prevention. Preventive and quality control measures are implemented to prevent contamination of the entire MP analysis procedure. The solutions used (30% H_2O_2, distilled water, distilled water) were filtered with 0.45 µm filter paper to avoid MP contamination. This procedure aims to ensure that the microplastic data obtained is accurate. Distilled water was used to clean all equipment, while cotton gloves were chosen to avoid cross-contamination (Suteja et al., 2021). Salt samples that had been extracted and placed on filter paper were stored in closed Petri dishes to avoid contamination by airborne MP. During sampling, plankton nets were set on the side of the vessel to reduce contamination. All equipment used for laboratory purposes was sterilized and made of non-plastic materials (Falahudin et al., 2020).

RESULTS AND DISCUSSION

Salt Made from Seawater

Microplastic Abundance in Different Salt-Making Treatments. The seawater used for salt making taken from Buo Bay, Padang City, Indonesia has been polluted by MP, evidenced by the discovery of MP as much as 510 particles/kg. Buo Bay is the center of the mariculture industry and many fishermen's activities in the use of nets cause the sea to be polluted by MP. In addition, plastic waste from community activities that is dumped into the sea from other locations can also move far from its source because its low density will be carried by ocean currents and waves (Karami et al., 2017). In salt making, seawater from Buo Bay is boiled to produce salt and minimize contamination from MP (Table 1). In treatment A, the MP abundance was 400 particles/kg, the results obtained decreased by 21.56% from the previous findings (Figure. 3a). This is assumed to be the effect of heat applied during the process of making seawater into salt for five hours which increases the brittleness of MP and the formation of a large number of carbonyl groups making it more susceptible to damage (Lin et al., 2022; Mao et al., 2020).

Table 2. The abundance and the removal efficiency of MP in various treatments

Treatment	Abundance of MP (particle kg^{-1})	The removal efficiency of MP (%)
A	400	-
B	240	-
C	138	65.5
D	99	75.25
E	99	75.25
F	79	80.25
G	69	82.75
H	30	92.5

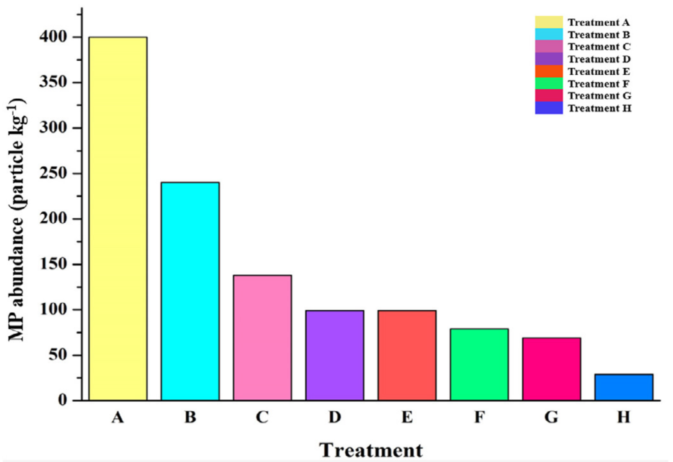

Fig. 3a. MP abundance (particles/kg) with different treatments

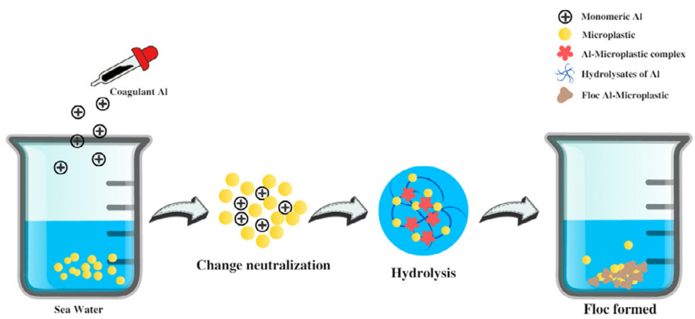

Fig. 3b. Illustration of the coagulation-flocculation mechanism of Al coagulant with MP

MP abundance in treatment B was 240 particles/kg. Treatment C was a combination of treatments A and B, resulting in an MP abundance of 138 particles/kg. To improve salt quality, treatment D was conducted with sand filtration, and MP abundance was reduced to 99 particles/kg. The use of sand as a filter media can reduce MP by trapping MP between sand grains or sticking to the surface of sand grains, as evidenced by the MP reduction efficiency of 80.58%.

The experimental results of each treatment on MP reduction and MP reduction efficiency can be seen in Table 2.

Treatment E (coagulant) obtained a reduction of MP from 400 particles/kg to 99, and the efficiency of reduction is: (400 -99)/400 x 100% = 75.25%, in the same way for other treatments can be seen in Table 2. The number of MP particles remaining in the salt is 30 particles/kg (can be seen in Treatment H).

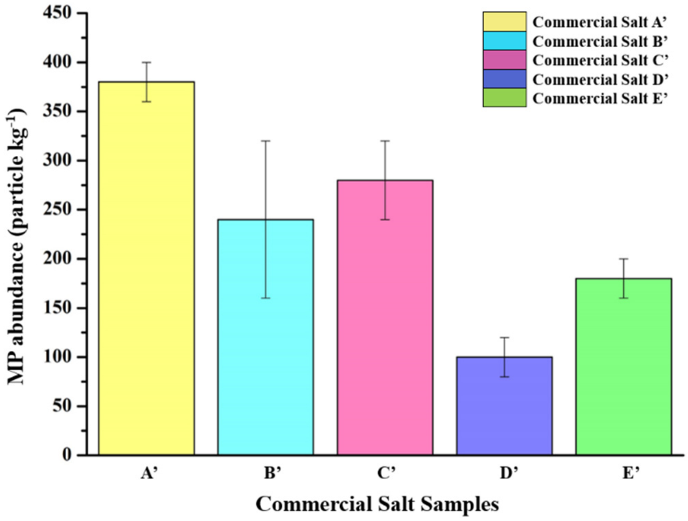

Fig. 4a. MP abundance in commercial salt

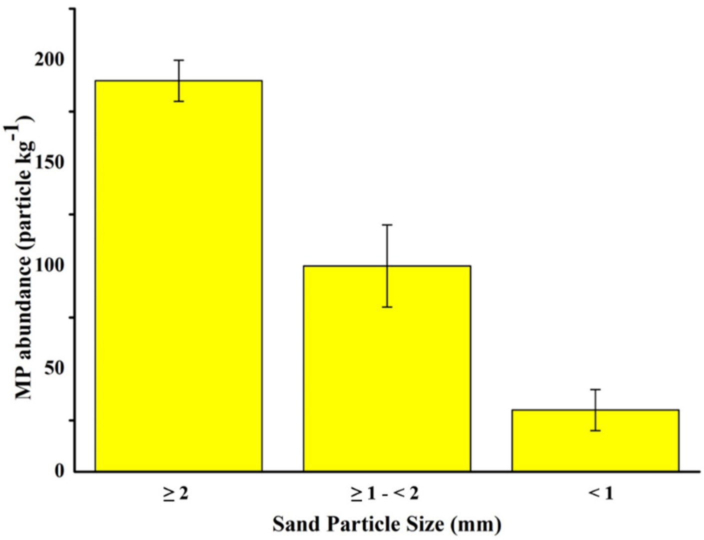

Fig. 4b. The abundance of MP with optimal treatment conditions of 0.5 g/L alum at various sizes of sand particles.

Then treatment E was carried out by adding alum as a coagulant, the abundance of MP obtained was the same as treatment D, namely 99 particles/kg and the efficiency of MP reduction was 80.58%. When the coagulant is not present in the water, MP will remain stable due to the electrostatic repulsion between MP. After the coagulant is added, the surface charge of MP will be neutralized so that the electrostatic repulsion is reduced. The functional groups of MP will be negatively charged when interacting with the positively charged Al from the alum coagulant so that the positively charged Al metal coagulant hydrolysate can be efficiently adsorbed on the MP surface which will neutralize the charge on the MP surface and reduce the electrostatic repulsion that makes MP unstable and will form flocs (Figure 4) (Shahi et al., 2020; Q. Xu et al., 2021; C. Zhao et al., 2021; M. Zhao et al., 2022). However, according to Lee et al., (2022), Al-based coagulants are not effective in removing most microplastics in seawater with the charge neutralization process performed only by Al.

Table 3. One-way ANOVA test abundance of MP

No	Treatment	Normality test Sig.	Average Abundance MP	Homogeneity test (based on average)	ANOVA test
1.	Sea salt	0.148	195.2	0.225	0.124
2.	Variation of alum concentration	0.363	59		
3.	Commercial salt	0.931	223.3		
4.	Variation in sand particle size	0.862	106.6		
		Sig. p>0.05		Sig. p>0.05	Sig. p>0.05

Therefore, treatments F, G, and H were conducted by varying the concentration of alum, then filtered with sand and palm fiber. MP removal increased with increasing coagulant concentration (Figure. 3a, treatments F; G; H). MP can interact with amorphous Al formed through hydrolysis of the coagulant by reducing electrostatic repulsion. When one end of the coagulant chain adsorbs MP particles, the other end also adsorbs other MP particles, thus forming an "MP-coagulant-MP" flocculant. The longer the coagulant chain, the better at reducing MP, and the larger the floc produced, the more MP is captured (Figure. 3b) (Xue et al., 2021; Zhang et al., 2021). At lower alum concentrations (treatment F) insufficient floc size was formed and no inter-particle bridges were formed, resulting in no settling through sweep flocculation which caused the MP abundance not to decrease. Increasing alum concentration facilitates floc growth, and interaction of MP with coagulant also occurs, thus reducing the surface charge of MP. As a result, the frequency of collision will be more frequent and more flocs will be formed (Zaman et al., 2021). Giving too high a dose of alum will result in flocs not forming because the MP particles will absorb too much positive charge from the coagulant and the MP potential will change to a positive charge so that it will stabilize again (Shahi et al., 2020; Tang et al., 2022; R. Yang et al., 2016).

In addition, the decrease in MP abundance was also affected by sand filtration. The negatively charged nature of the sand crystal structure can attract positively charged particles of colloidal matter (such as carbonate crystals and iron and aluminum hydroxide flocculants) and cations of iron, manganese, aluminum, and other metals (Huisman, 1974). MP will be absorbed into the sand surface due to the surface area possessed by sand. After adding alum 0.5 g/L and then filtering with sand (treatment H), the MP abundance was found to be 30 particles/kg. The efficiency of MP reduction from seawater (510 particles/kg) then made into salt with treatment H (30 particles/kg) was found to be 94.11%.

These results prove that the combined treatment of adding alum coagulant with sand filtration can reduce the abundance of MP in seawater which will be used as raw material for salt making.

Microplastic abundance in commercial salt

In Indonesia, salt production still uses sunlight-assisted evaporation techniques. In this study, the MP abundance of five brands of salt circulating in Padang City (salt A'; B'; C'; D'; E') without alum and sand treatment was studied. Salt A' had the highest abundance of 380 particles/kg, followed by salt C', B, 'E,' and D' with abundances of 248, 240, 180, and 100 particles/kg, respectively (Figure 4a). The difference in abundance of each salt brand is thought to be due to the source of seawater used as raw material for salt making and differences in salt-making procedures. As in sample D', the process uses refinery technology, which is a process with stages of dissolving, precipitating impurities, and filtering heavy metals and then crystallizing samples using modern high-tech machines to produce salt that is suitable for consumption so that the lowest MP abundance is obtained among the five salt brands tested as many as 100 particles/kg.

MP abundance in the treatment of sand particle size variations as filter media

The sand particle size was varied to find the most effective size in reducing MP abundance in salt, and three sand particle sizes were used in this study (≥2; ≥1-<2; and <1 mm). To see the efficiency of MP abundance reduction with sand particle size variation, an alum concentration of 0.5 g/L was applied before sand screening as it was the optimal condition from the obtained alum concentration variation. This treatment was tested on commercial salt A' because it has the highest MP abundance. MP abundance in commercial salt A' treated with 0.5 g/L alum and sand filtration decreased compared to untreated commercial salt A' (380 particles/kg). It is suspected that MP is trapped between the pores of the sand particles so that it can prevent MP from leaving the filtration media (Fajar et al., 2022).

The use of sand as MP filter media has many advantages. In addition to the low cost of obtaining sand as filter media, this sand filter can also be used repeatedly. When the top surface of the sand filter is dirty (darker in color), the sand can be replaced with a new one so that it can be used optimally again.

Sand particle sizes ≥ 2mm had an MP abundance of 190 particles/kg with a reduction efficiency of 20.83%. This is because large sand particles have large pores and the sample flow rate is fast so that MP is not trapped in the sand pores (Song et al., 2020). In sand particle sizes ≥ 1 - < 2 mm, the abundance of MP is 100 particles/kg so the efficiency of MP reduction increases to 58.33% when compared to sand particles measuring ≥ 2m. The highest MP abundance reduction efficiency was obtained by filtering sand particle size <1 mm as much as 30 particles/kg because the smaller the pore size of sand, the more MP was trapped in it so the MP reduction efficiency was obtained at 87.15% (Figure 4b).

A one-way ANOVA test was conducted to see if the treatments had the same mean if the significance value was greater than 0.05 (Table 3). In this study, a significance value of 0.124 was obtained and indicated that the average abundance of MP in each treatment was significantly the same.

MP Identification Based on Shape

Based on its shape, MP is divided into fragments, fibers, films, or pellets. The difference in particle shape is influenced by the length of time MP is in the marine environment, MP in the ocean is easily decomposed by waves and light, and wind and sand friction can also degrade plastic into small particles (Lin et al., 2022). Some forms of MP found in this study can be seen in Figure 5a.

Overall, in each MP treatment, the fragment form was found more and the least pellet form was found (Figure 5b). The fragment, fiber, and film forms indicate that the MPs found in this study are secondary MPs formed through photolysis, thermo-oxidation, thermodegradation, and biodegradation (S. Zhao et al., 2015). MP pollution in the form of fragments (52.98%) is assumed to come from plastic waste used by people around the coast. MP in the form of fiber was found as much as 23.95%, derived from textile waste. MP in the form of film comes from fishing ropes or fishing nets, and MP in the form of pellets comes from the waste of cosmetic products (Tanaka et al., 2016; Tsang et al., 2017).

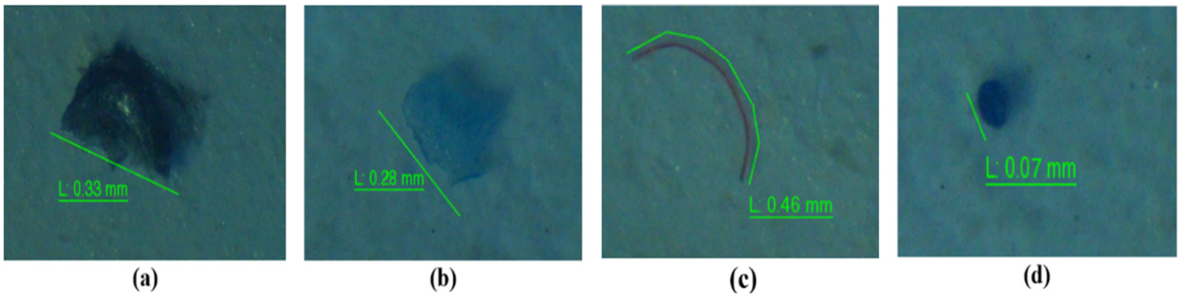

Fig. 5a. Forms of MP found in salt samples (a) fragments, (b) films, (c) fibers, (d) pellets

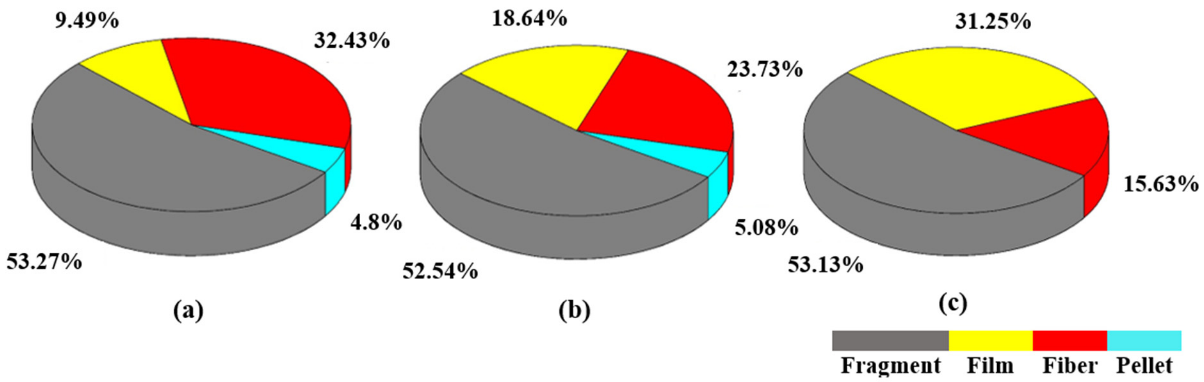

Fig. 5b. MP abundance percentage diagram based on the shape of (a) salt made from seawater, (b) commercial salt, and (c) treatment of variations in sand size.

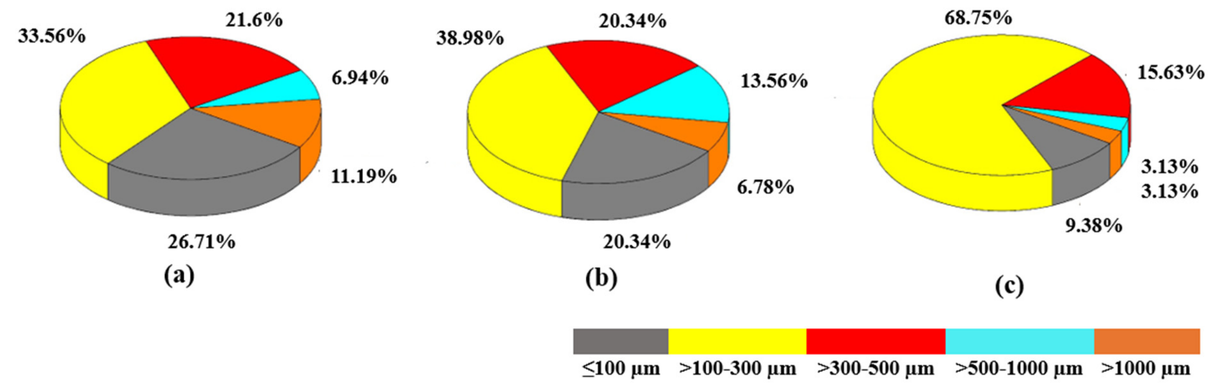

Fig. 6. Percentage of MP abundance diagram based on the size of (a) salt made from seawater, (b) commercial salt, and (c) treatment of variations in sand size.

MP identification by size

MP has a very dynamic nature because its size and shape will continue to change when it is in an environment that is influenced by various factors making it difficult to understand. Based on the size, MPs found in this study were classified into five size categories namely; ≤100; >100-300; >300-500; >500-1000; and >1000 µm. MPs with sizes >100-300 µm were the most dominant in this study (Figure 6). Hydrodynamic forces cause larger MPs to be less likely to collide (Xue et al., 2021). Larger MP particles require larger floc sizes to be removed through settling and will be retained by the filter media (Xue et al., 2021). Therefore, in this study, there were few microplastic particles >1000 µm in size because they had gone through the coagulation and filtration process. The small size causes MP in salt samples cannot be seen with the naked eye, so these particles will easily enter the human body through contaminated food products.

Identification of MP polymer types with ATR-FTIR

To see the role of Al^{3+} as a coagulant in the reduction of MP in salt, the FTIR spectra of treatment A and treatment H on salt prepared from seawater were compared (Figure 7a). The type of polymer present in treatments A and H was indicated to be polyethylene terephthalate (PET) with five peaks characteristic of PET polymer viz: 1715 (C=O), 1245 aromatic ether (C-O), 1100 aliphatic ether (C-O), 870 and 730 cm^{-1} aromatic (C-H) (Ioakeimidis et al., 2016).

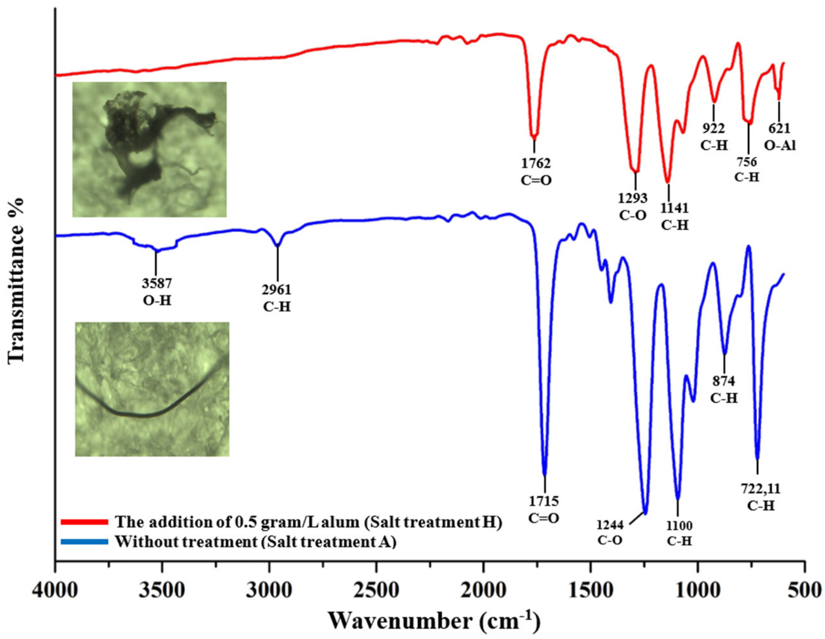

Fig. 7a. FTIR comparison of PET MP before and after treatment with 0.5 g/L alum

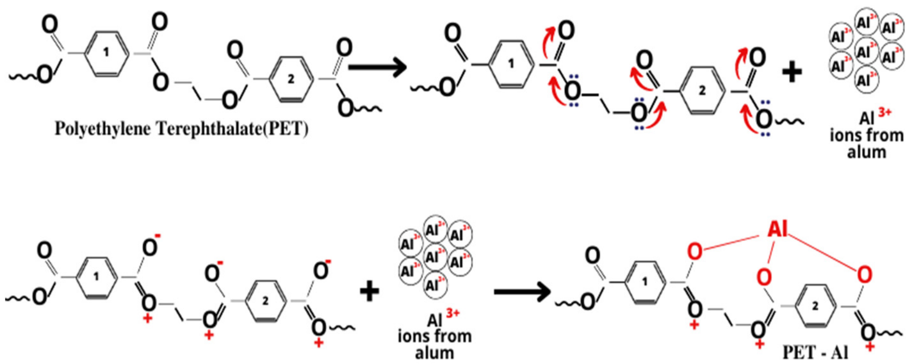

Fig. 7b. Prediction of the reaction mechanism of PET with Al^{3+} ions

In the FTIR spectrum of treatment H, there was a shift, disappearance, and appearance of new peaks when compared to the FTIR spectrum of treatment A. The peak shifts were induced by the formation of new bonds between Al and PET (Lu et al., 2021). The new peak at wave number 621 cm^{-1} in treatment H proves that Al^{+} is coordinated with oxygen-containing functional groups on the MP surface. The new oxygen-containing functional groups can effectively react with Al^{3+} ions through electrostatic interaction. Electrostatic adsorption occurs between compounds with opposite charges, and the interaction mechanism is shown in Figure 7b (Yu et al., 2021). In this study, the abundance of PET polymer was found to be the highest at 42.85% compared to PA (28.57%), PE (14.28%), and PP (14.28). The high density of PET polymer (1.38 g/cm^3) when compared to PE (0.94 g/cm^3) and PP (0.90 g/cm^3) polymers causes PET to precipitate on the salt product during the capitalization process. In addition, PET is a polymer that is widely used as a material for making bottles, and plastic bags, and in the textile industry is also widely used so that it will be found in the form of fibers in the marine environment (Iñiguez et al., 2017). In other studies, PET polymers have also been found in salt samples from various countries studied (Iñiguez et al., 2017; Karami et al., 2017; D. Yang et al., 2015).

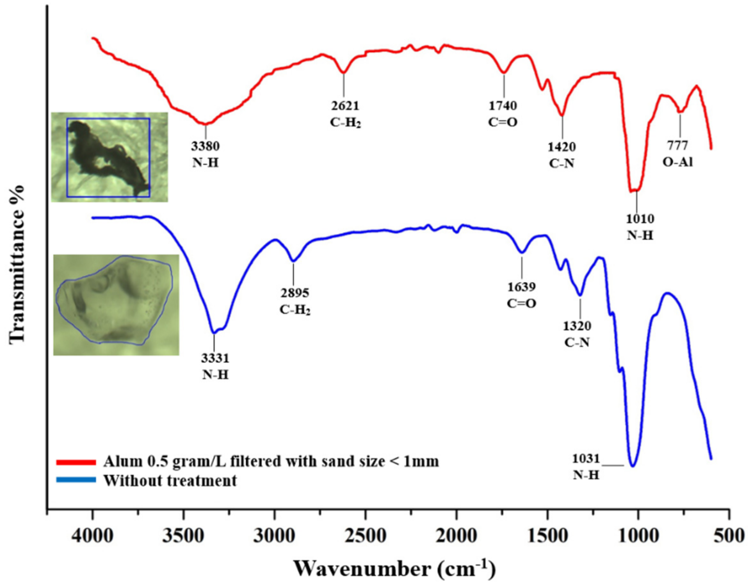

Fig. 8a. FTIR comparison of untreated salt MP (commercial salt A') with salt treated with 0.5 g/L alum and filtered with sand < 1mm in size.

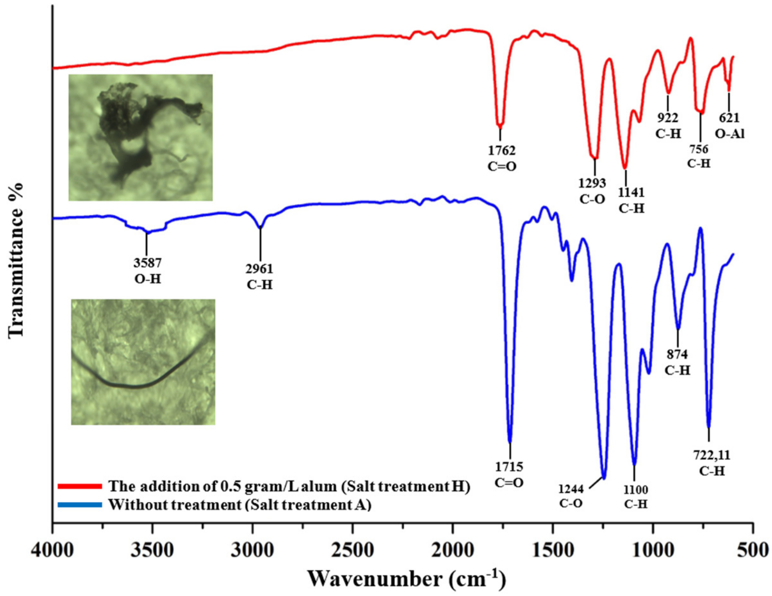

Fig. 8b. FTIR of MP on commercial salts B' (polyethylene) and C' (polypropylene)

Identification of polymer type of MP before and after filtration treatment with sand. The FTIR spectrum of commercial salt A' without treatment was compared with the spectrum of commercial salt A' treated with 0.5 g/L alum addition and <1mm sand filtration (optimal conditions) (Figure 8a). The type of polymer found was polyamide with characteristic peaks found at 3331 for N-H bonds, 2895 for $C\text{-}H_2$ bonds, 1639 for C=O bonds representing carbonyl amide groups, 1320 for C-N bonds, and at 1031 peak for N-H bonds (Cheval et al., 2012). The existence of peak shifts and the emergence of new peaks are related to the interaction of Al^+ ions with OH from amides, and Al^+ ions will attach to amides to form Al-O bonds at 777 cm^{-1} (Ng et al., 2012).

Identification of MP polymer types in commercial salts. Different polymers were found in commercial salts B' and C' (Figure 8b). Salt B' was indicated to be a polyethylene-type polymer and salt C' was indicated to be a polypropylene-type polymer with characteristic peaks appearing (Jung et al., 2018).

CONCLUSION

It can be concluded that the optimal conditions obtained from this research are: (1) alum concentration of 0.5 g/L and sand particle size <1 mm. (2) The utilization of alum and sand can reduce MP in salt from 400 particles/kg to 30 particles/kg with an MP reduction efficiency of 92.5%, (3) The most dominant form of MP found fragments, while the most dominant MP size found is >100 - 300 µm, and (4) The types of MP polymers found are PET, PE, PP, and PA.

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CONFLICT OF INTEREST

The authors declare that there is not any conflict of interest regarding the publication of this manuscript. In addition, the ethical issues, including plagiarism, informed consent, misconduct, data fabrication and/ or falsification, double publication and/or submission, and redundancy have been completely observed by the authors.

LIFE SCIENCE REPORTING

No life science threat was practiced in this research.

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