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SURAT KETERANGAN

Nomor: 118/UNUSA-LPPM/Adm-I/II/2024

Lembaga Penelitian dan Pengabdian Kepada Masyarakat (LPPM) Universitas Nahdlatul Ulama Surabaya menerangkan telah selesai melakukan pemeriksaan duplikasi dengan membandingkan artikel-artikel lain menggunakan perangkat lunak **Turnitin** pada tanggal 05 Februari 2024.

Judul	: *Identification and quantification of microplastics in seawater and sea salt collected from sea salt ponds*
Penulis	: Endah Budi Permana Putria, Achmad Syafiuddinb, Sofi Azilan Ainib, Iswahyudi Iswahyudic, Marchel Putra Garfansac
No. Pemeriksaan	: 2024.02.05.058

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NPP. 20071300

Identification and quantification of microplastics in seawater and sea salt collected from sea salt ponds

by Achmad Syafiuddin

Desalination and Water Treatment
www.deswater.com

doi: 10.5004/dwt.2023.29719

300 (2023) 130–135
July

Identification and quantification of microplastics in seawater and sea salt collected from sea salt ponds

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Received 3 September 2022; Accepted 10 June 2023

ABSTRACT

The existence of microplastics (MPs) in sea salt pond has the potential to pollute produced commercial salt. Therefore, identification and quantification of MPs in sea salt pond become necessary. This study was aimed to estimate the abundance of MPs in Osowilangun and Pademawu sea salt ponds producing commercial salt. Properties of the identified MPs in terms of shapes, color, size, and polymer type were characterized. This study found that the two study locations contained MPs with different levels, namely, a total of 38 particles were found in the Pademawu sea salt pond and 35 particles were found in the Osowilangun sea salt pond. Colors for the identified MPs were blue, black, red, and white depending on the sampling points and samples. In addition, size of MPs ranged from 0.2 to 0.8 mm and shape of MPs was dominated by fragment (90.32%) in Osowilangun sea salt pond and 86.98% in Pademawu sea salt pond. Findings of this study are highly important and significant for confirming the presence of MPs in sea salt ponds before producing for consumption.

Keywords: Microplastics; Seawater; Sea salt; Environmental pollution

1. Introduction

The issue of microplastics (MPs) in environment has received considerable critical attention [1]. Polymers used to manufacture MPs are polyethylene (PE), polypropylene (PP), polyethylene terephthalate (PET), and polyvinylchloride (PVC) [2]. More than 8,300 million metric tons of plastics had been produced for various uses because of their low production costs, versatility, lightweight, and durability properties [3]. Despite numerous benefits, the presence of MPs has the potential to threat the environments. It can pollute the marine and coastal environments with potentially fragmented <5 mm particles in size due to mechanical and photochemical processes that have potential to become a source of food for marine and coastal organisms as well as threaten their ecosystem. MPs were found in almost half (48%) of the crustaceans examined [4], in Mediterranean mussels (mean 0.55–3.20 items/g) [5], and globally, 94.4% of all oysters had MPs, with an average of 1.41 ± 0.33 per gram of soft tissue wet weight [6].

Previous studies prescribed that MPs were detected in marine and coastal environments, for instance sediments, beaches, seawater, saltwater pond, and polar regions. Since MPs were not effectively separated by conventional WWTPs

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[7], it is means that MPs can continue to flow to the ocean. Moreover, total MPs pollution in beach sediment Saint Martin Island in the Bay of Bengal was 317 particles/kg across 14 sampling sites [8]. In addition, 63% of MPs fiber pellets from seabird regurgitated were found in the Iberia peninsula, Northwest Spain, since MPs is one of non-digestible materials for seabird [9]. Another study showed that 83% of fiber particles were found in *Mytilus* spp., whereas usually *Mytilus* spp. was used as food source for human consumption [10]. These studies indicated that MPs strongly have high negative effect in marine and coastal environments. Thus, MPs in marine and coastal environments are interesting and important to be inspected.

Several studies have investigated the presence of MPs in various salt samples. For instance, 81.3% of the salt brands assessed (n = 16) and 55.6% of the total sample (n = 27) were contaminated with MPs in the 2022 Lebanese Market [7]. In addition, the presence of MPs in commercial salt was also found in Italy [11]. The significant amount of MPs reached 1653 ± 29 MPs/kg of sea salt. In total, 80.6% of the MPs were in the form of fibers, 18.9% in the form of fragments, and 2.7% in the form of balls [11]. Since MPs were found in various salts consumed by humans, it is very important to identify the presence of MPs in salt collected from salt ponds.

Several techniques have been applied for sampling of MPs regarding the inspection MPs in marine and coastal environments, such as using manta net, neuston net, bongo net, and plankton net [12]. Manta, neuston, and bongo nets are suitable for continuous flow in marine and coastal environment, whereas plankton net can be applied for non-continuous flow. Thus, plankton net is more appropriate to collect MPs sample in surface water, sea salt pond and sediments [13]. Plankton nets distinguish between particles that can and cannot pass through the net [14]. It allows to estimate the abundance of MPs in surface water, sea salt pond and sediments. Plankton net is the most technique for MPs sampling in sea salt pond [15]. The presence of MPs in sea salt ponds have potential to contaminate commercial salt. Previous reported the presence of PET and PP in commercial salts from different countries, such as Australia, France, Japan, Iran, Malaysia, New Zealand, Portugal, and South Africa [16]. It revealed that the possibility of MPs can be consumed by human along with the consumption of commercial salt. It is noted that MPs can enter the human body through ingestion of food and drink, and through inhalation. Consumption of food contaminated with MPs can potentially induce several problems related to immune systems, such as immunosuppression, immune activation, and abnormal inflammatory responses [17]. Moreover, it can also cause stress and induce reproductive toxicity and developmental disorders. Therefore, further research is needed to explore the potential impact of MPs to the environment and human health in more rigorous clinical studies.

MPs presence in sea salt pond used to produce commercial salt is required to be observed. It is one of preventive approach to understand and can minimize the abundance of MPs in sea salt ponds before consumption. Thus, the objective of this study was to estimate the abundance of MPs in Osowilangun and Pademawu salt ponds, Indonesia. The novelty of this study was the identification of MPs sea salt ponds which is missing in literature. Findings of this study are significant to (i) provide new information regarding the MPs present in sea salt and to (ii) encourage industry to apply effective technology to remove MPs from their commercial salt products to make sure safer sea salt for consumption.

2. Materials and method

2.1. Sampling location

Seawater and sea salt were sampled at two different salt ponds, which are at Osowilangun, Surabaya and at Pademawu, Pamekasan. Plankton net monyl screen mesh T165 with a pore size of 40 µm was used to filter the seawater samples. Strong acid solution, H_2O_2, was used to break down and release the organic matters from MPs samples. Distilled water was utilized to rinse the samples after placed in strong acid solution.

2.2. Sampling method

Seawater of 10 L were collected and then filtered using a plankton net mesh of 40 µm and 10 cm in diameter stainless-steel glass. In each study location, the samples were collected from 4 sample points, including seawater before entering the salt pond (BT), seawater at the pond (T1), seawater at pond evaporated for few days (T4), and 500 g of produced sea salt (salt).

2.3. MPs identification

Collected seawater samples were filtered by using the Plankton net mesh. The remaining materials on the filter were mixed with 30% H_2O_2 solution and kept for 48 h before drying at room temperature for 1 h. The solution of H_2O_2 was used to degrade any organic residue so that targeted MPs can be freely from organic residue before observing by using microscope. The microscope used in this study was Trinocular Digital Ways Dw-tc-y Black Edition, Kaisi Rotation LED Lamp K-D056 for microscope and 51 MP microscope camera connected to Samsung Smart TV 32. Next, the MPs samples analysed using Fourier-transform infrared spectroscopy (FTIR) Thermo Scientific Nicolet iS10 to characterize type of polymer.

3. Results and discussion

3.1. Presence of MPs

The presence of MPs in all samples collected from two locations, which are Osowilangun and Pademawu sea salt ponds is presented in Table 1. For Osowilangun, number of MPs found was 93 particles while at Pademawu salt pond was 126 particles. It is surprisingly that the number of MPs at salt for the two locations were consistently higher compared to BT, T1, and T4. It is noted that in Osowilangun, the MPs found was 35/250 g of salt while in Pademawu, the MPs found was 38 particle/250 g of salt. Findings of this study supports previous works who also observed the presence of MPs in sea salt produced by using different techniques, which are tunnel ponds, geomembrane, and traditional method [18]. The average contaminations of MPs in sea salt were 118.2 ± 43.7, 154.4 ± 79.8, and 273.5 ± 108.0 particles/kg of salt for tunnel ponds, geomembrane, and traditional method, respectively. Alternative study also reported that MPs were identified in refines sea salt, unrefined sea salts, and in rock salts with the number of particles of 1,400–1,900, 1,900–2,300, and 200–400 particles/kg of salt, respectively [19].

Table 1
Presence of MPs in Osowilangun and Pademawu

Sample	Shape	Number of MPs	Color	Size (mm)
		Osowilangun		
BT	Fiber (10)	10	Blue, black, red	0.4–2
T1	Fiber (28) Fragment (1)	29	Blue, black, red, white	0.2–3
T4	Fiber (15) Fragment (4)	19	Blue, black, red	0.4–3
Salt	Fiber (31) Fragment (4)	35	Blue, black, red, white	0.6–4
		Pademawu		
BT	Fiber (29) Fragment (5)	34	White, red, black, blue	0.2–3
T1	Fiber (32) Fragment (2)	34	White, red, black, blue	0.8–3
T4	Fiber (17) Fragment (3)	20	White, red, black, blue	0.2–3
Salt	Fiber (35) Fragment (4)	38	White, red, black, blue	0.2–3

3.2. Size and shape of MPs

The current study found that identified MPs have various sizes ranging from 0.2 to 4 mm for Osowilangun sea salt pond and from 0.2 to 3 mm for Pademawu sea salt pond. It is noted that smaller MPs have higher toxicity because it can easily permeate biological membranes, impacting on the functioning of blood cells [20]. Previous study showed that the presence of MPs in small size (<2 mm) was found in long Tuticorin Coastal salt pan stations, Gulf of Mannar, South India, with MPs of fragment, fibers, and sheet shapes [21]. The identification of MPs shapes obtained from Osowilangun and Pademawu sea salt ponds are provided in Fig. 1. Generally, there are two shapes of MPs, including fiber and fragment. In Osowilangun sea salt pond, 90% of fiber and 10% of fragment were found. Meanwhile, in Pademawu sea salt ponds, there were 89% of fiber and 11% of fragment.

Previous work reported that fiber shape of MPs was observed in subsurface seawater in the NE Pacific Ocean [22]. Fragment shape of MPs particles was also found in Indian sea salts [23]. It indicated that fiber and fragment shapes are easy to be found in coastal environment. Mostly, in coastal environment including sea salt ponds, fragments shape was generated by the breakdown of larger pieces of MPs. Fragmentation of plastics from larger size to smaller sizes, MPs, can be done via photodegradation, physical impacts, and other processes depending on environmental condition [24]. In addition, fibers MPs can also be derived from macro-plastics via photo oxidative process [25].

3.3. Polymer type investigation

Physical properties of polymeric materials depend on chemical constituents and configuration of the macromolecules. Many spectroscopic techniques are

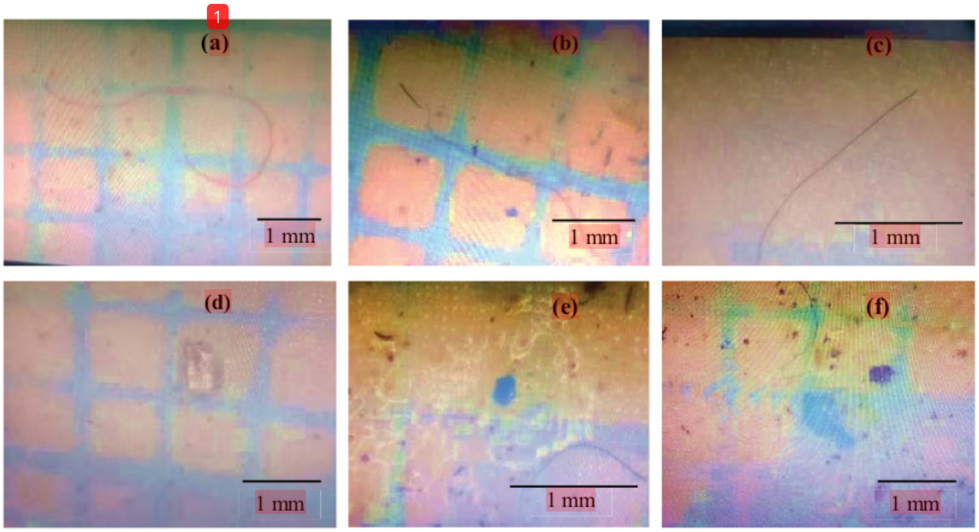

Fig. 1. MPs shapes of (a) fiber, red, 4 mm, (b) fiber, red, 2 mm, (c) fiber, red, 2 mm, (d) fragment, white, 1 mm, (e) fragment, blue, 1 mm, and (f) fragment blue, 1 mm.

available nowadays to examine these properties and FTIR-spectroscopy is one of techniques that is widely used due to its versatility in determining composition, hence, the polymer type can be determined by comparing their FTIR spectra with the library data. Figs. 2 and 3 show FTIR spectra for all analyzed MPs from two locations.

It is interesting to note that the FTIR spectra for MPs from sea salt at Pademawu (Fig. 2a) and salt from Osowilangun (Fig. 3b) have similar characteristics. For Pademawu, the vibrational bands located at 3,352; 2,915 and 1,692 cm^{-1} and for Osowilangun 3,336; 2,915 and 1,643 cm^{-1} are related to O–H groups, C–H stretching, and C=C stretching, respectively and theyare commonly identified for characteristics of cellophane as also reported by Rostami and Faraji [26]. Several peaks at 3,323; 1,713; 1,248; 1,089 and 1,016 cm^{-1} as shown in Fig. 2b are due to OH group (hydroxyl), stretching of C=O of carboxylic acid group, terephthalate Group ($OOCC_6H_4$–COO), methylene group, and vibrations of the ester C–O bond, respectively, and they are the typical characteristics of PET [27]. Strong peak intensities (Fig. 3a) at 2,920 and 2,851 cm^{-1} CH_2 are related to asymmetric stretching and CH_2 symmetric stretching, suggesting the typical of PE [28].

This study found that there were three types of polymer, which are PE, cellophane, and PET as shown in Figs. 2 and 3. For Pademawu samples, there were cellophane and PET found in seawater and sea salt, respectively, and the related FTIR spectra are shown in Fig. 2. As a comparison, for Osowilangun samples, there were PE and cellophane found in seawater and sea salt samples, respectively, and the related FTIR spectra are shown in Fig. 3.

It is beneficial to note that PE is one of the most commonly used plastics in the world because of its advantage such as toughness, almost nil water absorption, great chemical inertness, low coefficient of friction, simplicity of manufacturing, and low electrical conductivity. Numerous goods, including pipes, sheets, containers, and other items, use PE as raw material. In addition, PE has also been used as an electrical insulating material for wire and cable applications because of its great dielectric strength and extremely low electrical conductivity [29].

Cellophane is a cellulose derivative, specifically cellulose acetate (AC) [30]. Cellophane is one of the most important commercial cellulose derivatives that are widely used as photographic films, fibers, plastics, and membranes. Cellophane has outstanding mechanical properties, such as good tensile strength, high transparent ability, and high flexibility [31,32]. However, for decades, they have been used primarily as candy wrappers. They are also used as glossy transparent covers on tea boxes. Besides cellophane, PET is the most commonly used plastic polyester. PET is a transparent polymer that has good mechanical properties and good dimensional stability under variable loads [33]. In addition, PET has good gas barrier properties and good chemical resistance. PET is one of plastics that has excellent physical properties and be able to recycle [34]. It represents about 18% of all polymers produced worldwide. More than 60%

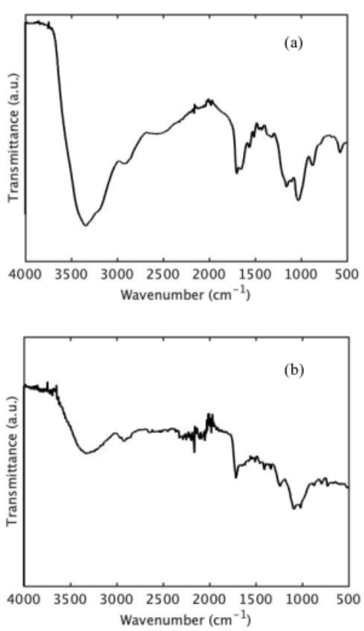

Fig. 2. Fourier-transform infrared spectra for MPs detected in (a) seawater at Pademawu (identified as cellophane) and (b) sea salt at Pademawu (identified as PET).

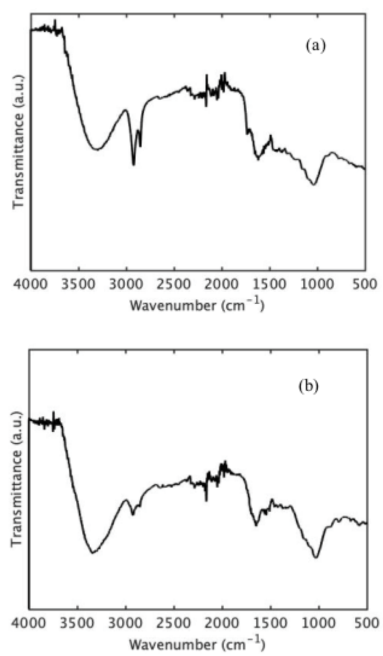

Fig. 3. Fourier-transform infrared spectra for MPs detected in (a) seawater at Osowilangun (identified as polyethylene) and (b) salt at Osowilangun (identified as cellophane).

of its production is for synthetic fibers and bottles, accounting for about 30% of global PET demand [35].

4. Conclusion

The aim of this study was to estimate the abundance of MPs in Osowilangun and Pademawu sea salt ponds producing commercial salt. This study found that MPs were found at two sea salt ponds with different levels depending on the sampling points and the colors for the identified MPs were blue, black, red, and white. This study also found that the size of MPs ranged from 0.2 to 0.8 mm and shape of MPs was dominated by fragment with 90.32% in Osowilangun sea salt pond and 86.98% in Pademawu sea salt pond. FTIR analysis clarified that the types of polymer were PE, cellophane, and PET. In general, this study has successfully identified the presence of MPs and findings of this study are significant particularly to improve community awareness about MPs present in sea salts. Future studies need to be conducted for designing a simple and reliable MPs removal technology to make sure the produced sea salts are safe to consume by community.

Funding

This study was funded by Universitas Nahdlatul Ulama Surabaya.

Acknowledgements

The authors are also grateful for all resources provided by the Universitas Nahdlatul Ulama Surabaya.

Conflicts of interest

The authors declare no conflict of interest.

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