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Microplastics in Salt of Tuticorin, Southeast Coast of India

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Abstract

Microplastics (< 5 mm) are considered to be global environmental pollutants. This study investigates the occurrence, physical properties, polymer composition and surface morphology, and element composition of MPs present in food-grade salts produced from seawater and bore-well water in Tuticorin, Tamil Nadu, Southeast coast of India. Fourteen different brands of sea salts and bore-well salts were collected from the salt manufacturing units. The mean abundance of microplastics was 35 ± 15 to 72 ± 40 items/kg in sea salt and 2 ± 1 to 29 ± 11 items/kg in bore-well salt. Four types of polymers viz. polyethylene (51.6%), polypropylene (25%), polyester (21.8%), and polyamide (1.6%) were found in salt. Polyethylene fibers of size ranging from 100 to 500 µm were observed commonly. Being manufactured from seawater, sea salt had the highest quantities of different microplastic particles. The study reveals that people consume approximately 216 particles of MPs per year via sea salt and 48 items per year via bore-well salt if the average person has a daily salt intake of 5 g. The surface morphology of MPs as exhibited in the SEM-EDAX images obtained in the study revealed the different weathering features of MPs, such as pits, cracks, and particles adhering to the surface. The presence of the elements Fe, As, and Ni on the surfaces as identified by energy-dispersive x-ray spectroscopy indicates that these elements exist in the environment as contaminants and have become associated with the MPs. The trace metals adsorbed onto MPs increase the risks of human exposure and may cause some adverse effects in humans.

The occurrence and extent of plastic pollution has increased over the past several decades (Lavender 2017). Plastic items take an exceedingly long time, hundreds and even thousands of years, to degrade in the environment especially in the marine environment (Gallo et al. 2018; Barnes et al. 2009). Microplastic (MP) particles with a diameter of less than 5 mm (Arthur et al. 2009) are derived from the successive breakdown of larger plastic pieces through the action of UV, and via mechanical and biological degradation processes (Booth et al. 2018; Gewert et al. 2015). They originate both from marine and land-based sources, including spillages during handling and transfer and losses during transportation (Turner and Holmes 2011). MPs constitute an increasingly important class of emerging contaminants (Sedlak 2017). The widespread occurrence and accumulation of MPs have been reported from various marine environments (Liu et al. 2019; Sharma and Chatterjee 2017; Anderson et al. 2016; Desforges et al. 2014; Andrady 2011), freshwater bodies (Eerkes-Medrano et al. 2015), and terrestrial ecosystems (Anderson et al. 2017). Because of their large surface area, MPs adsorb various other pollutants (Zhang et al. 2018; Kwon et al. 2017; Wu et al. 2016; Brennecke et al. 2016; Bakir et al. 2012; Barnes et al. 2009; Rios et al. 2007; Mato et al. 2001) and thus act as carriers of such pollutants. They produce a wide range of effects detrimental to the environment and adverse to the economy (Everaert et al. 2018; Schymanski et al. 2018; Burns and Boxall 2018; Syberg et al. 2015). They have negative impact on our safety, health, and culture (Catarino et al. 2018; Smith et al. 2018; Iñiguez et al. 2017; EFSA Panel on Contaminants in the Food Chain 2016). The oceans offer several resources for human consumption. Sea resources, such as salt and seafood, are some of the major sources of food for man. MPs in the sea will undoubtedly contaminate the sea resources. MPs have been detected in a wide range of marine organisms, including bivalves, mussels, lug worms, crabs, shrimps, and fishes (Tanaka and Hideshige 2016; Li et al. 2015; Van

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Cauwenberghe et al. 2015; Watts et al. 2014). Furthermore, the presence of MPs in coastal waters (Naidu et al. 2018), marine sediments (Reddy et al. 2006), beaches (Sathish et al. 2019; Jayasiri et al. 2013), and invertebrates (Kumar et al. 2018) along the Indian coasts has already been reported. MPs can even be transferred from sea resources to humans through the food chain, which potentially increases human health risks (UNEP 2016).

Sea salt (sodium chloride, NaCl) is our main source of sodium (Brown et al. 2009). A few recent studies from China, India, Spain, and Turkey have found that sea salt contains MPs (Seth and Shriwastav 2018; Gündoğdu 2018; Maria et al. 2017; Yang et al. 2015). For salt production, India is ranked third in the world, after China and the United States (GOI 2017). In India, Tuticorin comes next to Gujarat in salt production (Ananthalaxmi and Jeyakumari 2016). On the basis of origin salts are categorized as sea salt, lake salt, rock salt, river, and bore-well salt (Yang et al. 2015). In Tuticorin, seawater and bore-well water are used for salt production. Typically, salt is produced by the evaporation of water. On evaporation, water leaves behind the salt to crystallize. The contaminants present in the water are inevitably left behind in the salt. Even though information on microplastic pollution in sea products is limited, not many works have been undertaken to study the quality of salts produced in Tamil Nadu. Particularly, there are no data on the level of microplastic contamination of sea salts produced in Tuticorin. With this background, this study proposes to identify and characterize microplastic particles present in the food grade salts. It also aims to investigate the surface morphology using a scanning electron microscope (SEM) and to analyse the elemental composition using an energy dispersive x-ray spectrometer (SEM-EDAX) unit. This study offers an insight into the level of MPs (and associated elements) in commercial food grade salts. It brings out the extent of exposure that human beings are subjected to and suggests that current MP contamination represents an exposure risk via the food chain.

Materials and Methods

Collection of Salt Samples

In Tuticorin, salt is produced in salt pans using seawater and bore-well water. Seven different brands of fine food-grade sea salts (S) and seven other brands of bore-well salts (B) were procured during the period between March and August 2018. Sea salts (S) were obtained from Veppalodai (S1), Vaipar (S2), Thirespuram (S3), Roche Park (S4), Sippikulam (S5), Melmanthai (S6), and Periyasamipuram (S7). Bore-well salts (B) were acquired from Arasady (B1), Sivandakulam (B2), Mullakad (B3), Karapad (B4), Lavingipuram (B5), Mappilaiyurani (B6), and Davispuram (B7). The samples were collected on site by the researchers in three different replicate packages of each brand (Fig. 1). At the collection site, the sample was immediately packed in glass sample bottle with label (bearing the particulars of nature of the sample, collection place, sampling date, and time) and transported to the laboratory under hygienic condition.

Microplastics Analysis

The analysis of MPs in the salt samples was done according to the method of Yang et al. (2015) with a slight modification. Approximately 250 g of each type of salt was mixed with 100 ml of 30% H_2O_2 to digest the organic matter, and the mixture was kept in the incubator at 65 °C for 24 h. Digestion of the sample with 30% H_2O_2 at 70 °C does not degrade the polymers PP, LDPE, HDPE, PS, PET, PA-6,6, PC, and PMMA (Sujathan et al. 2017). The samples were then kept at room temperature for a further period of 24 h. One liter of filtered distilled water was added to the samples to dissolve the salt. This solution was centrifuged at 1900 rpm for 1 h to remove sand particles, if any. The supernatant solutions of salt were immediately transferred onto a 0.8-µm cellulose nitrate filter paper through a Millipore Filtration Unit. The material deposited at the bottom was transferred onto Petri dishes. The filters and the contents of Petri dishes were dried at room temperature and inspected under a 40x Motic stereomicroscope. The MPs were visually determined based on the homogeneous color, brightness, and absence of cellular structures. Hot Needle Test (De Witte et al. 2014) also was performed for the primary confirmation of microfibers. The suspected microplastic particles on the filter paper were selected for verification using Fourier Transform Infrared Spectrometer (Thermo Nicolet model iS5) equipped with Deuterated Triglycine Sulfate detector and attenuated total reflection (ATR) attachment to identify the type of polymer. The spectrum range was from 700 to 4000 cm^{-1}. The abundance of MPs was calculated based on the microscopic and FTIR-ATR observation. The spectra were compared with spectral library database (Thermo Scientific) to identify the chemical composition of the particle. Matches with a threshold > 80% were accepted. FTIR spectroscopy also was used to assess the weathering pattern of the polyethylene and polypropylene MPs using a Carbonyl index (Barbeş et al. 2014)

$$\text{Carbonyl index (CI)} = A_{1715}/A_{2870}$$

where A_{1715} is absorbance of carbonyl group (–CO–); A_{2870} is absorbance of methylene group (CH2–), symmetric stretching vibration.

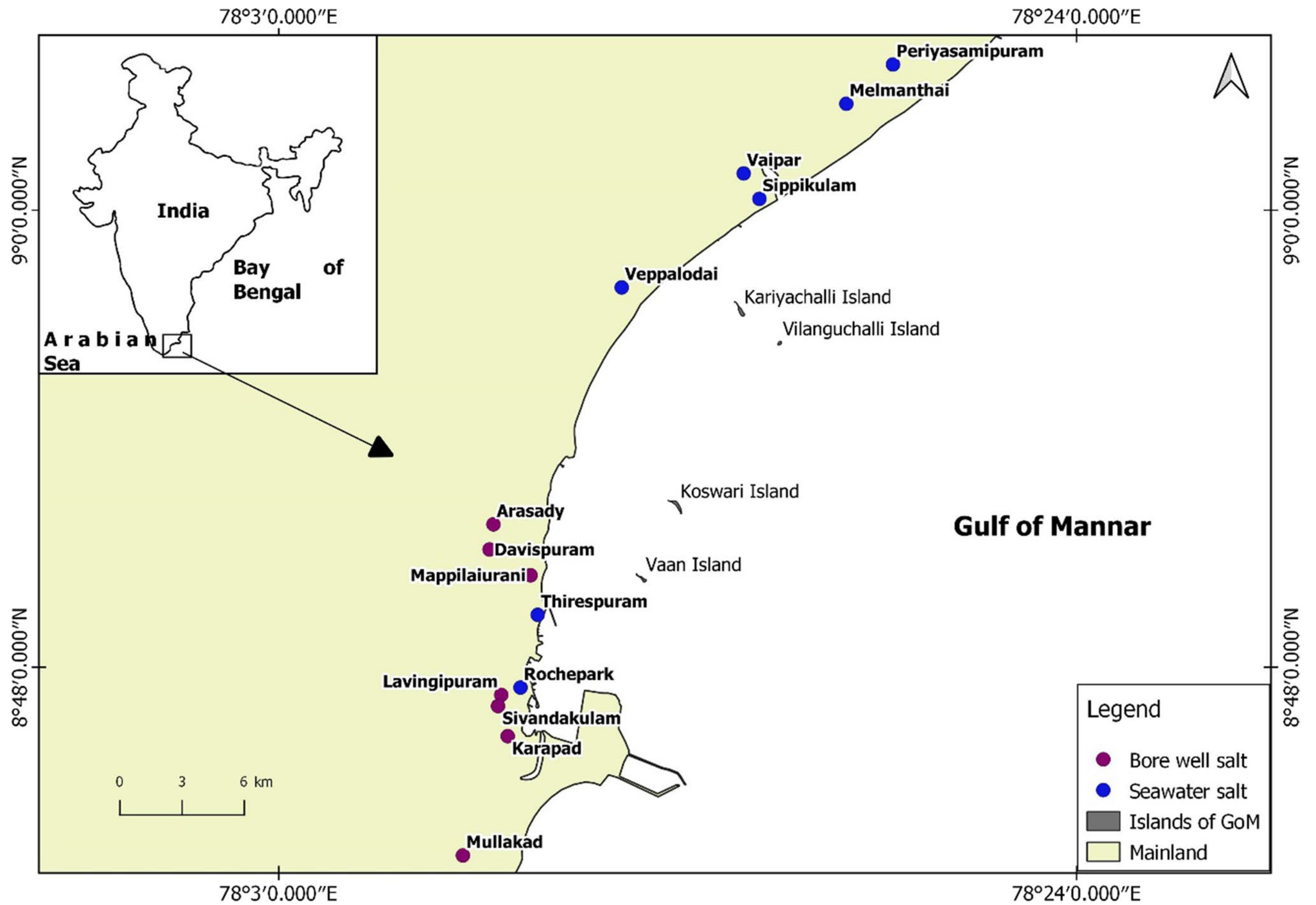

Fig. 1 Location of sampling of sites

Quality Assurance and Quality Control

During each step of the analysis, precaution was taken to avoid background contamination. The lab windows were closed throughout the experiment. For mitigating cross-contamination risks, all glass wares were cleaned with ultrapure water before usage, and the samples were covered with lid. The research personnel wore cotton coats to reduce the airborn contamination, if any. Blank experiments also were performed following the same procedure without sample to assess air contamination. Then, the value was subtracted from the value of the field samples to remove the error due to air contamination.

Surface Morphology

Scanning Electron Microscope (JEOL model JSM 6390) was used to study the microplastic morphology. In this procedure, the samples are spread on a double-sided adhesive tape, coated with a thin film of evaporated gold, and the images are taken with an optimized acceleration voltage of 10 kV and the detector working distance of about 2 mm. An energy-dispersive x-ray spectroscopy (EDAX Oxford Instrument) revealed the elements adhering to the microplastic particle.

Statistical Analysis

In order to understand the variations in microplastic distribution between seawater salts and bore-well salts, one-way ANOVA was performed under a level of statistical significance with a p value < 0.05. The MP abundance is presented as the mean number of MPs per kg of salt. Analyses of variance were carried out using the SPSS 20.0 software package (SPSS, Chicago, IL).

Results and Discussion

Abundance, Shape, Size, and Color of Microplastics

The mean abundance of MPs (items/kg) in different types of salts is shown in Fig. 2a. The microplastic particle content is found to be 35 ± 15 to 72 ± 40 items/kg in sea salt

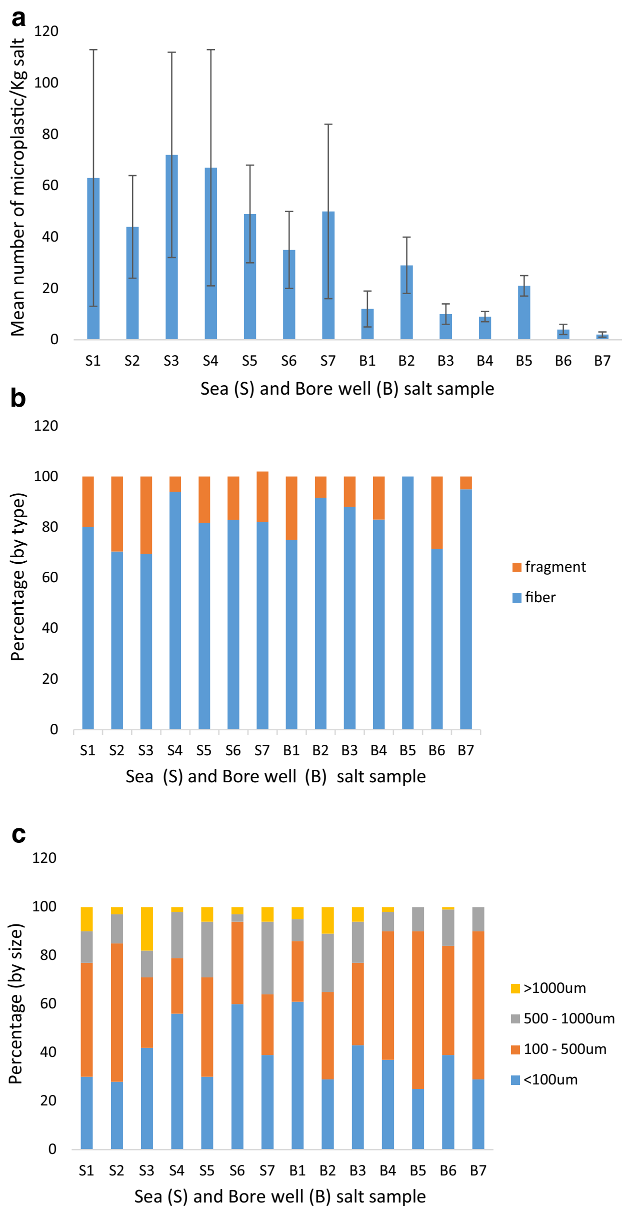

Fig. 2 Abundance (**a**), type (**b**), and size (**c**) of MPs in sea salt (S1–S7) and bore well salt (B1–B7). Error bars represent standard deviation, where three different replicate packages of each brand salts were used

and 2 ± 1 to 29 ± 11 items/kg in bore-well salt. The higher values of abundance of MPs in sea salts are recorded in S3 and S4 where untreated sewage input, fishing activities, and industries contribute to the coastal MP pollution. There is statistically significant difference between the amount of MPs found in sea salt samples and bore-well salt samples ($P < 0.05$). The amount of MPs in sea salt is found to be higher than in bore-well salt, which is obviously due to the contaminated seawater. Several recent studies have showed that the sea is a major sink of MP pollution (Sathish et al. 2019; Qu et al. 2018; Teng et al. 2018; Chen et al. 2017; Jambeck et al. 2015), and the pollution of the sea will undoubtedly lead to the pollution of sea products. Seth and Shriwastav (2018) investigated the MP contamination in Indian sea salt and found that the number of particles ranged from 56 ± 49 to 103 ± 39 kg^{-1} of salt. The levels of MPs in the salt samples analyzed by the present study are found to be lower than those in the sea salts and well salt from China and Spain (Yang et al. 2015; Iñiguez et al. 2017). Some recent studies have reported the presence of MPs in the beach sediments and fishes of the coastal environments of Tuticorin (Sathish et al. 2019; Kumar et al. 2018). Microplastic abundance in sea salts may be attributed to the fact that sea salt is a direct product of the coastal water (Seth and Shriwastav 2018). MPs of lower density also can be transported horizontally and vertically by soil cracks and also by earthworms, collembolan, and other organisms from surface soil into deeper soil layers (Yu et al. 2019a, b; Huerta Lwanga et al. 2017; Maass et al. 2017; Rillig et al. 2017). The production of salt being an open process, it also might be influenced by airborne contamination (Prata 2018; Yang et al. 2015; Bouwmeester et al. 2015). This finding is in line with the results obtained for microplastic pollution of honey and sugar (Maria et al. 2017; Liebezeit and Liebezeit 2013).

As for the shapes of MPs, this study observed more fiber (83%) than fragment (17%) (Fig. 2b). Similar results have been obtained by Gündogdu (2018) for salts in Turkey. The shapes of fiber and fragment indicate that the MPs found in this study are secondary MPs formed through the processes of photolysis, thermo-oxidation, thermo-degradation, and biodegradation (Zhao et al. 2016; Laglbauer et al. 2014). Presumably the high level of fiber pollution might have originated from commercial fisheries, laundry, domestic wastewater, and other local human activities in Tuticorin region. Additionally, fishing gear and airborne MPs contributed to high accumulation of fiber type of MPs. Breakdown of larger plastic debris forms fragment-shaped MPs. Depending on their size, the MPs found in this study are classified into four categories: < 100 µm; 100–500 µm; 500–1000 µm; and > 1000 µm. The size distribution of the MPs observed in this study vary from 55 µm to 2 mm in all the samples (Fig. 2c). In sea salt, 40% and 36% of particles belong respectively to < 100 µm and 100- to 500-µm category. In bore-well salt, 37% and 43% of particles belong respectively to < 100-µm and 100- to 500-µm range. A majority of the samples accounts for < 500-µm range. Yang et al. (2015) found that the MPs of < 200 µm represented the majority of those in sea salts. Because of the very small size range, the particles in the salt samples are not visible to the naked eye, and they could easily enter into the consumer's body and prove harmful to human health. Unfortunately, the impact of MPs on food safety and human health are still unknown (Van Cauwenberghe and Janssen 2014). The MPs identified in this study possess different coloration ranging from semi-transparency, through black and blue to red. From this, we can infer that the particles are derived from different color sources.

Identification of MPs

A preliminary characterization was done by a combination of visual identification under microscope, fragmentation test with tweezers, and application of the hot needle test (De Witte et al. 2014). The chemical composition of MPs was studied by FTIR-ATR. Because it is difficult to analyze thin MPs with ATR, 75 particles were selected for identification. Of which 85.3% (50 particles from sea salt and 25 particles from bore-well salt) were established as plastic, 10.6% as unidentified particles, and 4% as nonplastic particles, such as cotton, metal wire, natural fibres, and other nonsynthetic materials. The FTIR analysis revealed the presence of four types of polymers: polyethylene (PE), polypropylene (PP), polyester (PES), and polyamide (PA) (Fig. 3). Some non-plastic items, such as black enamel paint, also were identified in the seawater salt samples. It might have originated from boat washing, abandoned structures, and grounded ships (Andrew et al. 2009). In sea and bore-well salts, the most common polymer is polyethylene followed by polypropylene (Table 1). This might be due to the massive production and consumption of polyethylene plastic material. As of 2017, more than 100 million tons of polyethylene resins are produced annually, accounting for 34% of the total plastics market (Plastics Europe 2017; Geyer et al. 2017). Polyethylene has been observed in salts from other countries also (Gündoğdu 2018; Iñiguez et al. 2017; Karami et al. 2017; Yang et al. 2015). Kumar et al. (2018) reported that polyethylene and polypropylene were widely distributed in the marine environment of Tuticorin. Sathish et al. (2019) also identified PE, PA, and PS polymers in the beach sediments of Tuticorin. Wastewater intrusion and fishing activity in the marine environment might be the cause of the accumulation of microplastic in seawater. Most of these polymers also have been observed in different marine environments by other workers (Sebille et al. 2015; Desforges et al. 2014; Andrady 2011). This research also supports that the fibres (PE and PP) found in bore-well salts could have originated from PE envelopes used in packaging and from clothes worn by operators during the industrial process (Renzi and Blaškovic 2018).

In general, polymers undergo degradation through physical and chemical processes as well as through photo- and biodegradation. FTIR spectra of PE and PP have a characteristic peak at 2914 cm^{-1}, 2847 cm^{-1}, 1470 cm^{-1}, and 718 cm^{-1} (PE) and 2915 cm^{-1}, 2945 cm^{-1}, 2838 cm^{-1}, 1455–800 cm^{-1} (PP). But some of the PE and PP spectra contain additional degradation peaks at 3386 cm^{-1}, 1713 cm^{-1}, 1635 cm^{-1}, and 1031 cm^{-1} for a hydroxyl group, carbonyl group, alkenes, and ester linkage with the characteristic peak due to oxidative weathering (aging) processes (Fig. 4). The formation of additional functional groups on the surface of MPs increases with increasing solar radiation, thermal oxidation, and hydrolysis (Singh and Sharma 2008). The relative amount of formation of carbonyl groups is related to the progressive aging of MPs. The carbonyl index values of MPs calculated in this study vary from 0.4 to 1.32 for PE and from 0.8 to 2.1 for PP. The CI normally increases with increasing degree of photodegradation (Endo et al. 2005). This is congruent with a long residence time in the marine environment. During aging, plastic debris can become weathered, gain more surface area, and generate oxygen groups, which can increase their polarity, charge, roughness, and porosity (Fotopoulou and Karapanagioti 2012). Thus, the microplastic staying for a long time in polluted marine environment has a tendency to accumulate other element onto its surface (Ryan 2008; Wang et al. 2017). The present study showed that the PE and PP of MP particles have undergone various weathering processes, and this can greatly facilitate the adsorption of inorganic pollutants (Li et al. 2019; Yu et al. 2019a, b).

Surface Morphology of MPs

SEM-EDAX is a useful tool for imaging the surface morphology of MP particles and provides useful data on the inorganic elements present on the surface of MP particles. The SEM images exhibit different features on the MP surface; for example, PE particles of bore-well salt show small protrusions (Fig. 5a) and PE and PP of MPs of sea salt show cracks, pits, and the particle (Fig. 5c, e, g) adhering to their surfaces. These variations, due to the different levels of weathering undergone by the plastic particles through wave action and sand grinding (Fig. 5d, f), demonstrate that the presence of naturally occurring elements (i.e., Ca, Si, F, Cl, S, K, and Al) on MPs is consistent with the environmental exposure to which seawater is subject during the crystallization process. The presence of other elements, such as Fe, Ni, and As (Fig. 5h), is consistent with the levels of existing environmental contaminants. These results bear out the great relationship between the degree of plastic aging and sorption capacity to heavy metals. Santhanakrishnan et al. (2016) reported the presence of heavy metals, such as Fe and Ni, in the salt pan sediment of Tuticorin, which they attributed to the effluents of nearby industries and the domestic dumps. Fe in the MPs might have been derived from the fly ash from the Tuticorin Thermal Plant (Baskaran et al. 2002), whereas Ni may be indicative of inputs from petroleum-related activities in the surrounding areas (Muthu Raj and Jayaprakash 2008).

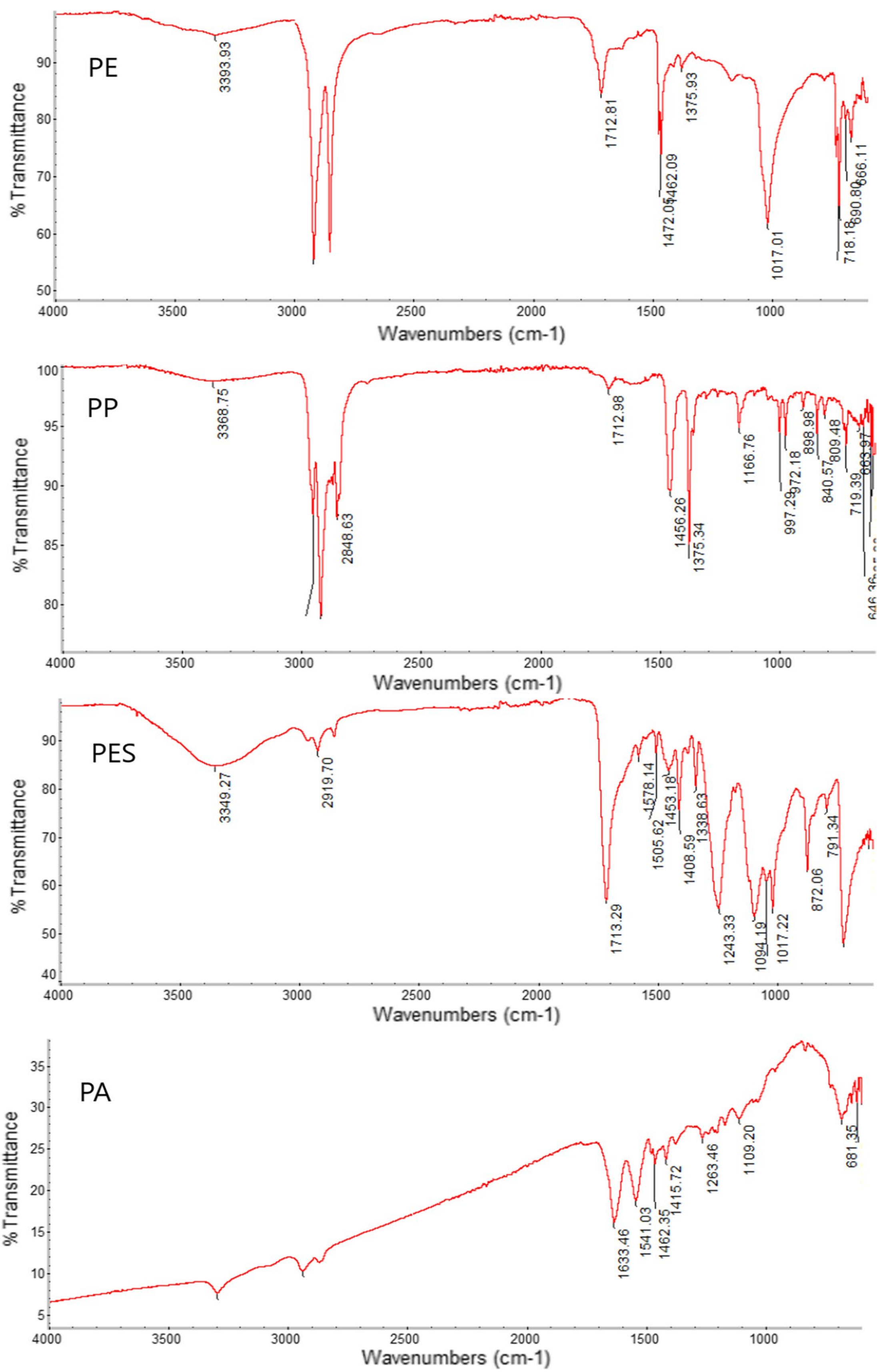

Fig. 3 FTIR-ATR spectra of representative MPs extracted from sea salt and bore-well salt samples

Table 1 Types of microplastics identified in salt samples (75 particles were selected, of which 85% of the particle are confirmed as polymer by FTIR)

Type of particle	Sea salt		Bore-well salt		Total	
	N = 50	Percentage	*N* = 25	Percentage	*N* = 75	Percentage
Plastic	44	88	20	80	64	85.3
Polyethylene	24	54.5	9	45	33	51.6
Polypropylene	10	22.7	6	30	16	25
Polyester	9	20.4	5	25	14	21.8
Polyamide	1	2.2	0	0	1	1.6
Nonplastic	1	2	2	8	3	4
Unidentified	5	10	3	12	8	10.6

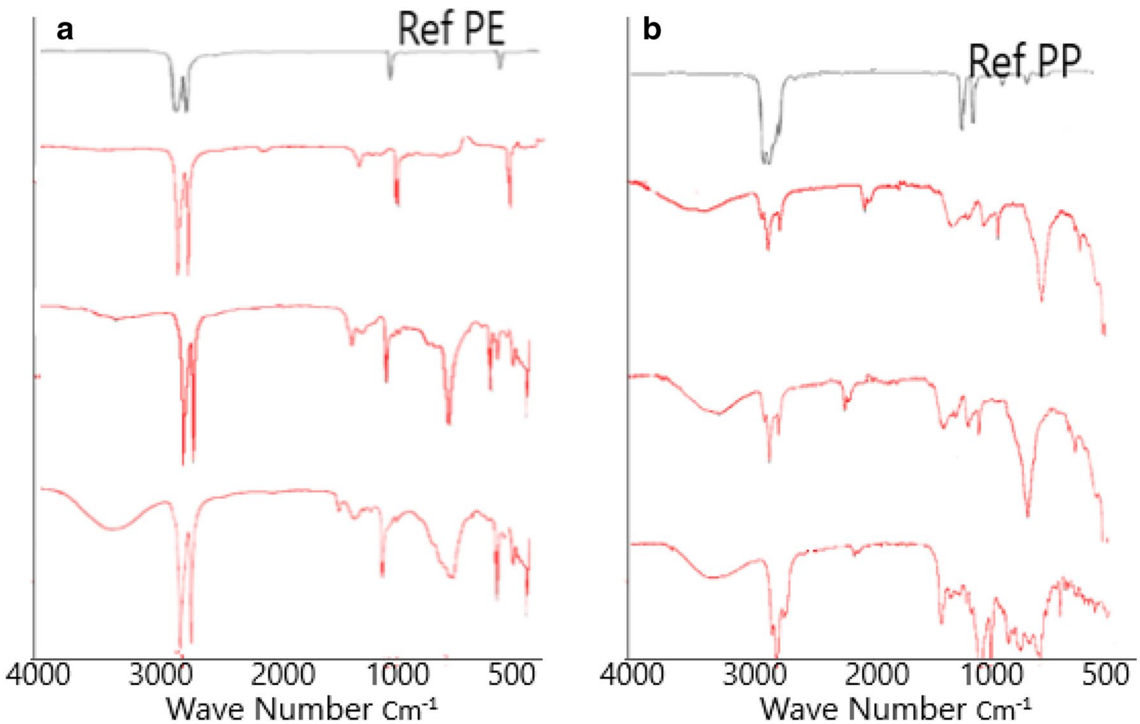

Fig. 4 FTIR spectra showing the chemical weathering of PE (**a**) and PP (**b**) microplastics

Implication of MPs

Humans consume food products containing salt throughout their life. The total amount of MP intake by humans through contaminated salts, therefore, is expected to increase. Johnson et al. (2017) reported that an average Indian consumer takes 10.98 g of salt per day, which is twice the limit of 5 g per day recommended by the WHO (2012). As the results of the present study show, the average number of MPs is 54 ± 13.4 items/kg in sea salt and 12 ± 9.5 items/kg in bore-well salt. At this rate, people consume 216 items of MPs per year via sea salt and 48 items per year via bore-well salt if the average person has a daily salt intake of 5 g. MPs adversely affect many animals (Mohsen et al. 2019; Wang et al. 2019; Magni et al. 2018; Martins and Guilhermino 2018). They get translocated across living cells to the lymphatic and circulatory systems in humans (particle size 0.2–150 μm), rodents (30–40 μm), rabbits (0.1–10 μm), and dogs (3–100 μm), possibly via Peyer's patches in the intestine (Waring et al. 2018; Rieux et al. 2005; Hussain et al. 2001). Because of their prolonged incubation in the water, MPs also have the ability to adsorb organic (POPs) and inorganic pollutants persistent in the environment and transfer them to the sea resources, thus increasing the risks of human exposure to these chemicals.

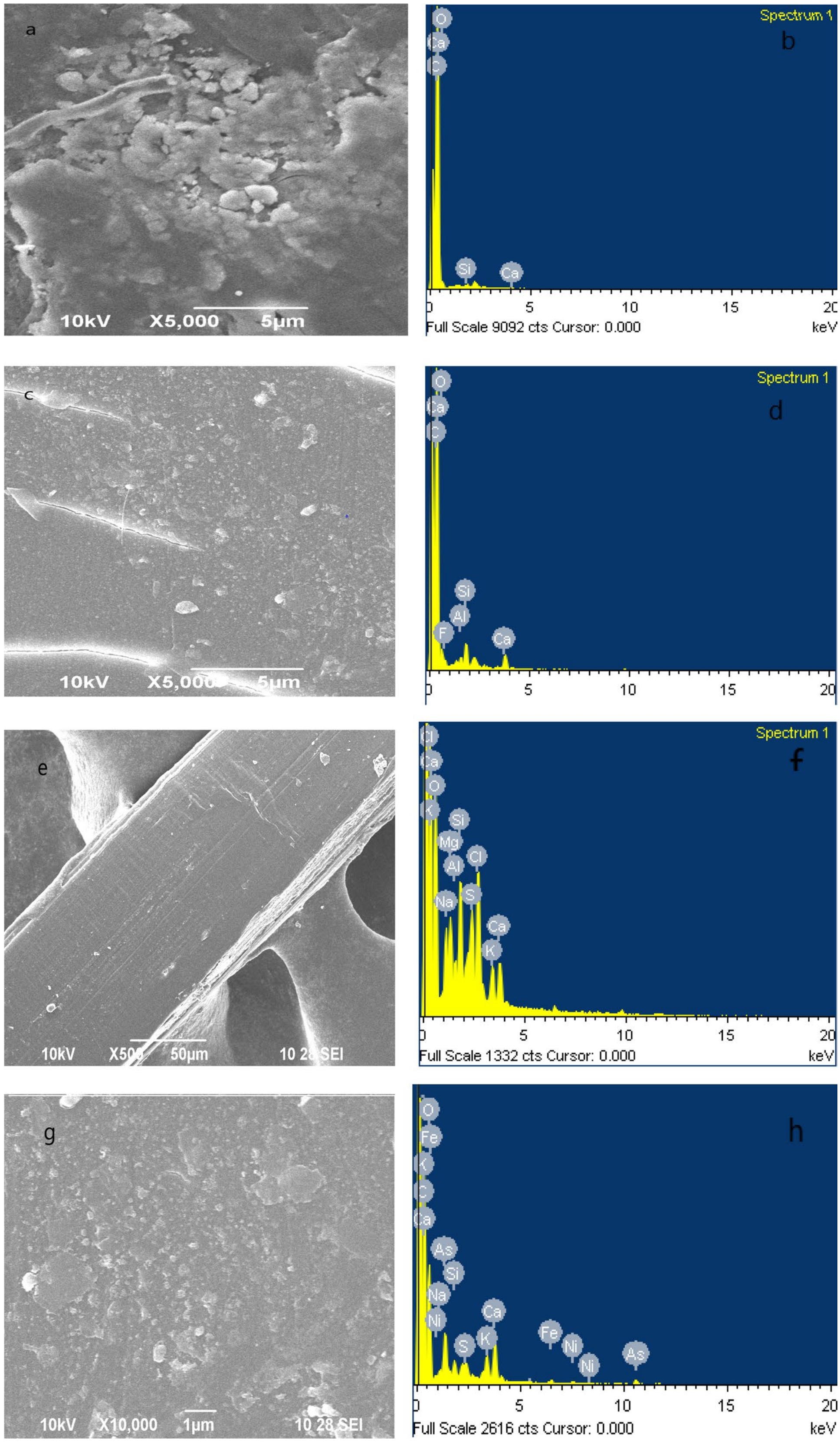

Fig. 5 SEM/EDS image of PE (**a**, **b**) MP from bore-well salt, PP (**c**, **d**) and PE (**e**–**h**) MPs extracted from sea salt showing protrusion, cracks, pits, and tiny particle with inorganic element

Conclusions

This study reveals the presence of MPs in food-grade salts manufactured from two sources: seawater and bore-well water. Fiber and fragment types of MPs of various size categories of different polymers were extracted from the salt samples; polyethylene is the most common in sea salt and bore-well salt. The study shows that sea salts are more contaminated with MPs than bore-well salts. SEM-EDAX images obtained in the study exhibited the different types of surface morphology of weathered MPs, such as pits, cracks, and adherent particles. The presence of elements, such as Ni and Fe, associated with MPs might be due to pollution from fly-ash and petroleum-related activities in the surrounding area. The level of MP contamination is still low, but it may increase in time because of the increasing use of plastic and the continuing improper methods of its disposal. This is the first study in this region on the contamination of salt with MPs. It quantifies with reasonable approximations the mass concentration of MPs. Further studies are needed to understand the mechanism involved in adsorption of MPs in human tissue and their potential effect. Regular monitoring of MPs in various sea resources is necessary for food safety and human health.

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