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Microparticles and microplastics contamination in African table salts

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ABSTRACT

The presence of micro/plastic particles has been reported in various seafood products. However, information on microplastics contamination in salts from African continent is very limited. This study analysed 23 brands of table salts from 8 African countries for microplastics using microscopic/spectroscopic techniques. South Africa showed the highest microplastics concentration (0–1.33 ± 0.32 particles/kg), Nigeria, Cameroun, and Ghana (0–0.33 ± 0.38 particles/kg each); characterized as polyvinyl acetate, polypropylene, and polyethylene. Other countries have no detectable microplastics at 0.3 μm filter pore size. To our best knowledge, this is the first study to characterize micro-fibres/plastics in table salts across African countries, confirming that it is an emission source of micro-fibres/plastics into the human food chain, highlighting the overarching need to understand their effects on human health.

1. Introduction

Plastic pollution has been identified as contributing the greatest proportion of marine litter globally (Bergmann et al., 2015) with unabated flooding of oceans with plastics. This is due to anthropogenic activities and mismanaged plastic waste that eventually end up in the aquatic environment. The disintegration of plastics into smaller particles (micro/nanoplastics, ≤5 mm) which are more mobile and can penetrate different barriers, constitute a great risk to aquatic animals. Likewise, the marine environment has been a source of food reservoir to man, raising a universal concern about human safety through seafood consumption and other useful products obtained from the marine ecosystems. For instance, recent findings have identified plastic particles from different seafoods including canned sardine, mussel and seafish (Karami et al., 2018; Li et al., 2018; Ribeiro et al., 2020; Santillo et al., 2017) with their potential threats to human health attracting intense attention due to the widespread detection (Zhang et al., 2020).

Salt is one of the sea products useful for human consumption worldwide. Salt has found its application in industries such as pharmaceutical (Morris, 2002), paint, water treatment, textile, aluminium, metals, soaps, and detergents. Likewise, it is used domestically for food preparation, seasonings, preservation, disinfection, relieving sore throat, reducing fatigue, and removing dead cells or plaque (Buss and Robertson, 1973). Furthermore, sodium, an essential ion in the human body is recommended by World Health Organization to be consumed at 2 g/day per adult which is equivalent to 5 g of salt per day (Mente et al., 2018; Grimes et al., 2009). It plays a pivotal role in osmoregulation, fluid maintenance within the body, muscle contraction and nervous system coordination ("Dietary Guidelines Advisory Committee Reports", 2015; Strom et al., 2013) and this is majorly obtained from salt.

However, salt consumption has been reported as one of the exposure routes of human to microplastics. Microplastics contamination in commercial table salts has been monitored and reported in different countries around the globe, such as Spain (Iñiguez et al., 2017), Turkey (Gündoğdu, 2018), India (Seth and Shriwastav, 2018), Taiwan (Kim et al., 2018), Italy (Renzi and Blašković, 2018), China (Yang et al., 2015), Australia, France, Iran, Japan, Malaysia, New Zealand, Portugal and South Africa (Karami et al., 2017). Table salts can be contaminated from the water drawn from seas to manufacture salt that may contain microplastics, organic materials and sand particles as well as during the manufacturing process (Gündoğdu, 2018; Yang et al., 2015). With microplastics in table salt entering the human body through the digestive tract (Zhang et al., 2020), a study has reported microplastics in human stools suggesting that humans are being exposed to microplastics

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through the food chain (Schwabl et al., 2019). Although recent studies have stressed on the importance of investigating the potential risk of microplastics transferred through the food chain to humans (Seltenrich, 2015), health risk assessment of microplastics remains in its infancy with limited information on exposure routes, biological fates, and health effects (Zhang et al., 2020). While the effects of microplastics ingestion in humans is still a subject of research (Fadare et al., 2020a), studies have shown some of its deleterious effects on aquatic species, which constitute a significant part of human diets. This includes its ability to induce behavioural and physiological changes, embryotoxicity, reproductive and transgenerational effects (de Sá et al., 2018; Fadare et al., 2020b). As a result, the safety of marine products processed for human consumption is raising a global concern.

To date, however, there have been few studies on the source and occurrence of microplastic in table salts;, with data on the extent of pollution in the African continent lacking; although table salts provide essential elements for humans (Yang et al., 2015). Therefore, it is necessary to monitor the presence of microplastic contaminants in table salts. Herein, this study aimed to investigate the prevalence of microparticles, microfibres, and microplastics contamination in commercial table salts obtained in open markets as well as supermarkets in some selected countries across Africa; providing a baseline for micro-fibre/plastics contamination in table salts across eight African countries. The presence, abundance and composition of the plastic particles were examined using microscopic and spectroscopic techniques.

2. Materials and methods

2.1. Chemicals and materials used

Hydrogen peroxide (H_2O_2) was purchased from Sigma-Aldrich (St Louis, MO, USA). All solutions were prepared using ultra-pure water (resistivity of 18 MΩ cm) provided by a Millipore Milli-Q system (Bedford, MA). The digested salt solutions were filtered through a MF-Millipore cellulose nitrate membrane filter (having 0.3 μm pore size and 47 mm diameter) obtained from Merck KGaA, Ireland.

2.2. Sample collection

Twenty-three different brands of commonly consumed commercial table salts were obtained from open market/supermarkets in the following African countries (Fig. 1): Nigeria (4), Cameroon (2), Ghana (3), Malawi (1), Zimbabwe (1), South Africa (6), Kenya (5) and Uganda (1), between August and September 2019. The collected brands

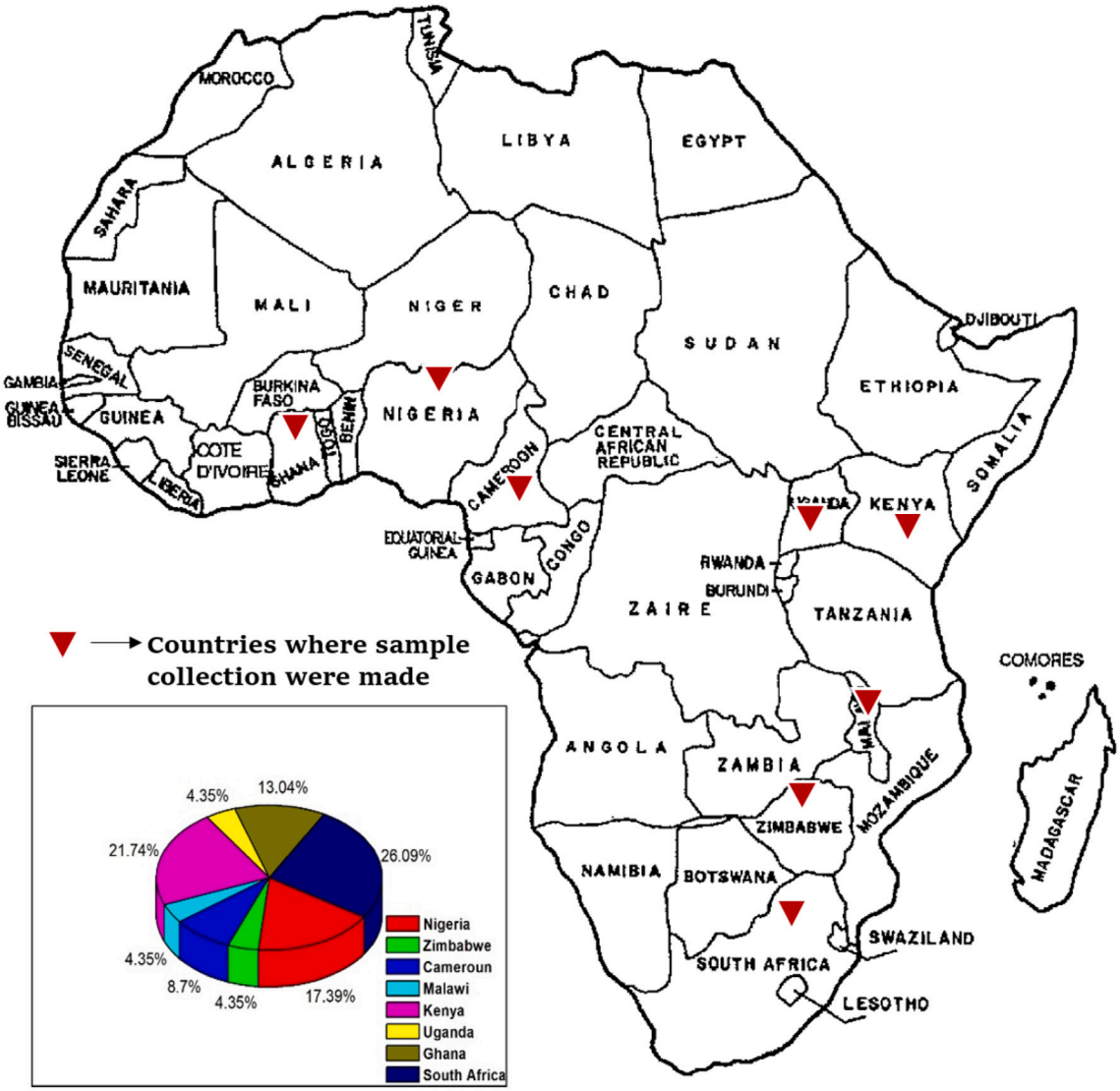

Fig. 1. Map of Africa showing the eight countries from where the salt samples were purchased namely: Nigeria, Cameroon, Ghana, Malawi, Zimbabwe, South Africa, Kenya, and Uganda. The pie chart represents the percentage distribution of the sampled salts.

represent the majorly available brands in the market. Further details on the individual brands, including information on country of origin, production date, expiry date, batch no (if indicated), town/city/place of purchase, colouration at the time of coming into the laboratory and weight per package have been provided in the Supplementary information (SI) Table S1 and Fig. S1. Company's name and brand's name were omitted for privacy reasons. The packaged weight of the sampled salts ranged from 200 g to 1 kg and the weight of each brand were reported in the supporting information, Table S1.

2.3. Extraction of particles

Extraction of micro/plastic particles in the sampled salts was carried out using a previously reported procedure (Yang et al., 2015) with slight modification. In brief, each salt sachet (in triplicates) was carefully transferred into 2 L conical flask. 100 mL of 30% hydrogen peroxide was added to each flask in order to digest any organic matter present in the salt samples. The flasks were corked with a glass cover to minimize atmospheric contact and to prevent the introduction of foreign particles, placed in a chamber (KLC2-1220A, Yataikelong, Beijing, China) at a temperature of 65 °C, and agitated at 80 rpm overnight. Thereafter, 800–1500 mL of filtered Millipore water was added to each sample. The flasks were then gently shaken to ensure complete dissolution of the salts. Thereafter, the salt solutions were filtered using a filtration setup under vacuum. In an effort to avoid contamination from air, particles extraction/all the experiments were carried out inside a laminar flow chamber. To prevent contamination, a 100% cotton laboratory coat was worn during all steps of analysis. All glassware's, containers, beakers, filtering units and devices used for processing samples were rinsed with filtered Millipore water before used. All laboratory working benches and working space were cleaned with 70% ethanol throughout the experiment. Whenever samples were not being processed (extraction and characterization), they were covered with aluminium foil. To further account for contamination, two controls were set up, which are: water used in the extraction of the particles (procedural blank, C1), water left open in the experimental lab for the period of extraction (air blank, C2).

2.4. Characterization of particles

The residues on the filter papers were visually examined with the help of an optical microscope. All detected/counted particles were classified into microparticles (MPs), microfibres (MFs), and microplastics (MPPs), and those considered to be polymers were analysed using Infrared Imaging Microscope Fourier-transformed infrared (FT-IR) spectrometry (Nicolet iN10 MX, Thermo Scientific, WI, USA) to identify the surface functional groups. All the sample spectra were recorded as 32 scans in the spectral range of 650–4000 cm^{-1} at a resolution 4 cm^{-1} and were compared to the polymer library (KnowItAll, Bio-Rad) to identify the polymer type. The plastic particles were then placed on an adhesive carbon tape mounted on an aluminium sample holder. The morphological characterization of the particles was carried out using a scanning electron microscope/energy dispersive X-ray system (SEM/EDX, ASPEX LLC - 3020, Aspex Cooperation, Delmont, PA, USA) to obtain the surface morphology of the microplastics.

2.5. Image analysis and particle size measurement

The SEM images of each MP were obtained and analysed with ImageJ software (NIH, USA) to obtain the particle size. OriginPro 8 software (Northampton, MA, USA) was used for the plots.

3. Result and discussion

3.1. Abundance and morphology of microparticles

In this study, we isolated and characterized the various particles in 23 brands of commercial table salts across eight (8) African countries. Fig. 2 shows some of the particles isolated from the salts which are typically fragments, fibres, granules, and microplastics. Also, different impurities like sand, paste, small pebbles and unidentified particles were observed in various brands. Similar observation has been reported by Renzi and Blašković (2018). The origin and production process of table salt from different marine environment has been identified as the possible source of various impurities extracted in sampled salts (Yang et al., 2015). For instance, the presence of microplastics has been reported in the marine system globally, which are ingested by aquatic animals and has been found in seafoods processed for human consumption (Van Cauwenberghe and Janssen, 2014). Renzi and Blašković (2018) reported litter content recovered from marine table salts from Italy and Croatia which include foam, fragments, fibres, granules, micro/meso/macroplastics of different types, shapes and colours. The presence of these microparticles was attributed to the overexploitation of the coastal area which is the major source of the salts by anthropogenic activities, including plastic pollution (Kim et al., 2018). In a similar study, Kosuth et al. (2018) identified microplastics in all sea salts analysed with the most frequent plastic type being fibres and not particles. Seth and Shriwastav (2018) also found microplastics in all salt samples, with 37% and 63% of the extracted microplastics being fibres and particles, respectively. The accumulation of microplastics in the oceans and other aquatic ecosystem is raising concerns due to the possible impact on global food safety and human health (Rist et al., 2018). Furthermore, there are possibilities that additives may be introduced during production by the manufacturers to enhance their products such as synthetic folates or folic acid (Modupe et al., 2020). For instance, the presence of paste-like materials (Fig. 2a and e) in some of the sampled salts can only be rationalized as additives incorporated during production.

To enhance the quality of salt production in order to minimize impurities or unwanted particles especially in Africa, there is a need to improve the production processes of table salt. Each stage of production must be upgraded, or modern equipment can be introduced as this is crucial to food safety. Also, where additives are introduced into a product, this should be clearly stated on the label to keep consumers informed about the content of the product purchased.

In all the table salt samples analysed, there were concerning levels of different particles with a vast majority of particles being fibres which represented about 93.8% of the isolated plastic-like particles in all the salt samples (Fig. 4c: Supporting Information Fig. S2). Textile fibres have been reported as the abundant microplastic type in the marine environment (Henry et al., 2019), suggesting that microplastics contamination in table salt may originate from the plastic polluted seawater. The total concentration of the plastic-like particles ranged from 24.08 ± 16.21 particles/kg to 78.9 ± 38.53 particles/kg, while the mean concentration was found to be 38.42 ± 24.62 particles/kg. The concentration of the plastic particles detected in the samples was classified into two categories: microfibres (fibres) and the microplastic particles which were a total of 1246 and 82 particles respectively. Studies have shown that microfibres are the most abundant plastic particles in the marine environment (Suaria et al., 2020). From Fig. 3a, there is no significant difference between the mean microfibre concentration from all the countries and that of the 2 controls, especially C1 (procedural blank). However, microplastics concentrations (Fig. 3b) were significantly different from the control (no particles found in the control). The high levels of microfibres detected in the controls highlights the importance of including controls when assessing levels of contamination to avoid overestimation. It is possible that these fibres were from airborne contamination (as in Control C2). Since most of the microfibres detected in all countries were not significantly different from the control (Fig. 3a), we focus on the microplastic particles which amounted to a total of 82 particles in all countries under study.

Microplastics were found in all triplicate salt sample in all countries (except one salt brand from South Africa, SAS-4) with a total

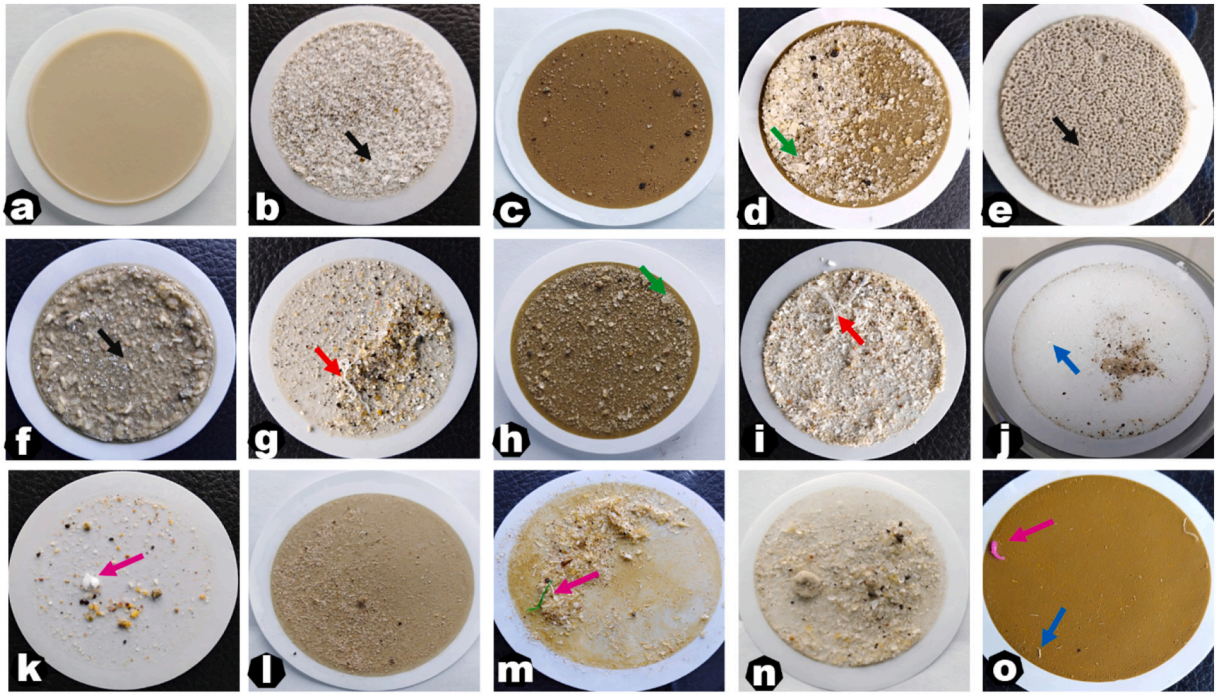

Fig. 2. Representative particles extracted from different table salts obtained from eight African countries; fragments (blue arrows, j and o), fibres (red arrow, g and i), granules (green arrow, d and h) unidentified particles (black arrow, b, and f) and microplastics (pink arrow, k, m, and o). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

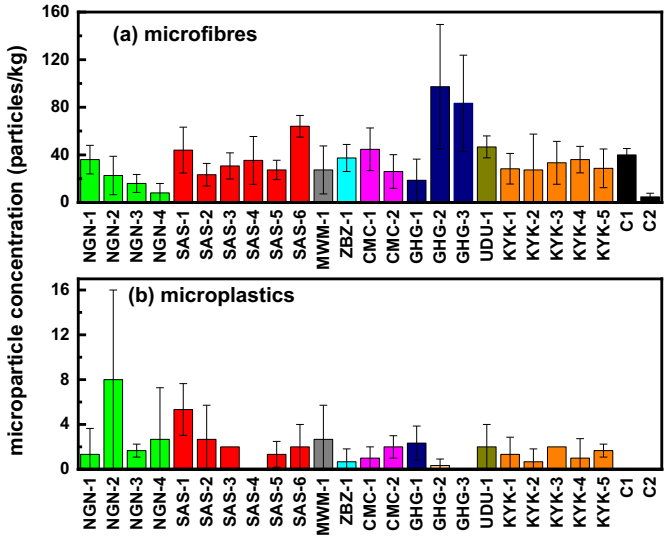

Fig. 3. Mean of the plastic-particle concentration (a) microfibres (b) microplastics detected across all samples examined. Error bars represent the standard deviation between triplicate runs.

concentration ranging from 0.67 ± 1.15 particles/kg to 3.42 ± 4.94 particles/kg. The overall mean concentration of microplastics was calculated to be 1.68 ± 1.83 particles/kg. The distribution of the total particle count per mass of salt is presented in Fig. 4. Among all 8 countries, table salt samples from Ghana had more total particle counts per mass (including impurities like sand and small pebbles) compared to other countries (as corrected in Fig. 4a below).

3.2. Characterization of microplastics in table salts

Due to the particle size limitation (>50 μm), about 10.9% of the 82 isolated microplastics were eventually analysed using FT-IR. These were made up of polyvinyl acetate (PVA), polypropylene (PP) and polyethylene (PE) (Fig. 5a). FTIR analysis was carried out on the bulk packaging material used for the salts, with the data presented as a pie chart in Fig. 5b. The bulk packaging mainly consisted of polyethylene (PE), polyethylene isophthalate (PEI) and polyethylene terephthalate (PET). Comparing Fig. 5a and b shows that PP, PVA and PE-PP were not identified in the bulk packaging, which implies that some of the microplastics isolated from the salt samples did not originate from the bulk packaging. Hence, we infer that some of the microplastics detected in the table salt samples originated from the environment or salt sources during production. Kim et al. (2018) reported PE, PET and PP in sea salt samples from Senegal, with PET being the only microplastics not detected in our study. However, Karami et al. (2017) identified only PET in sea salts samples analysed from South Africa. Seth and Shriwastav (2018) have also reported several types of microplastics such PE, PET, polyamide (PA), polyesters (PES), and polystyrene (PS) in salt samples, with the types of microplastics present reported to be independent of the salt brand and packaging type. The observation made by Seth and Shriwastav (2018) goes a long way to support the findings of this study regarding bulk packaging and the identified microplastics in salts. However, Iñiguez et al. (2017) analysed packed and un-packed salt from the same well but concluded that the packing process had no influence on the microplastics content in salt samples.

Scanning electron microscopy (SEM) is recognised as a useful analytical tool providing high-resolution particle surface features (morphology) (Gniadek and Dąbrowska, 2019). SEM analysis of the microplastic particle surfaces often revealed characteristic features, which may suggest aging, oxidative or mechanical degradation of the plastic surface (Wang et al., 2017; Yin et al., 2019). For instance, mechanical disintegration of plastic can result in physical features such as grooves on the surface of the material. In the current study, we investigated the surface morphology, oxidative and mechanical degradation of the identified microplastics. The images (Fig. 6) revealed different surface morphological features such as irregular (Fig. 6C), rough (Fig. 6D), hollow (Fig. 6E), porous (Fig. 6F), and cracks (Fig. 6A and B)

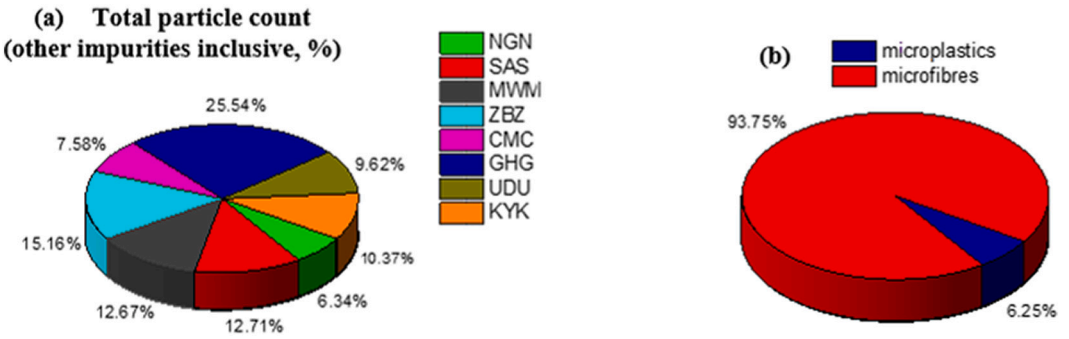

Fig. 4. (a and b) Contribution of each country to the total concentration of plastic particles (microfibres and microplastics) detected across all triplicate runs (c) distribution of particle type detected in all samples under examination.

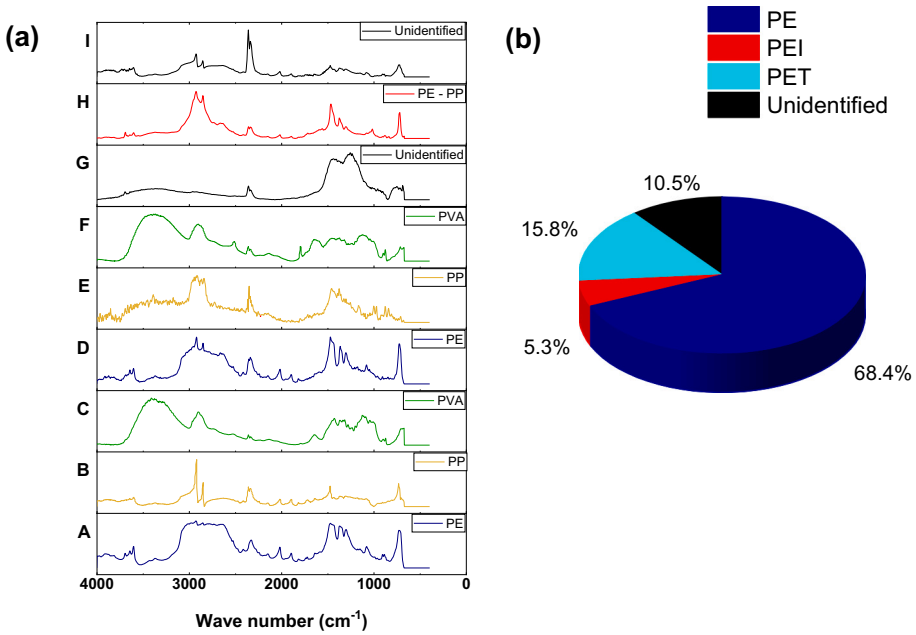

Fig. 5. (a) FTIR spectra of the sampled microplastics. (b) Chemical composition of the bulk packaging materials. Polyethylene; PE, polypropylene; PP, polyethylene terephthalate; PET, polyethylene isophthalate; PEI and polyvinyl alcohol; PVA. NB: Unidentified means the particle spectra did not match a plastic material from the library database.

in the analysed microplastics. The primary degradation morphological features exhibited by most of these microplastics were cracks and fractures, which might have resulted from oxidation in the environment because the physical appearance of the isolated microplastics shows that they do not emanate from the bulk packaging materials. Their source can only be attributed to the salt source, which is the marine environment. The observed cracks could result in increased particle surface area, which is known to enhance the adsorption of hydrophobic organic chemicals on the surface of the microplastics. The high degree of oxidative degradation is expected due to the impact of salt on the microplastics and warm to high temperature may as well be a contributing factor, as Africa is mostly tropical region.

3.3. *Abundance of microplastics in African table salts*

Particles detected in all samples were below the 5 mm size definition

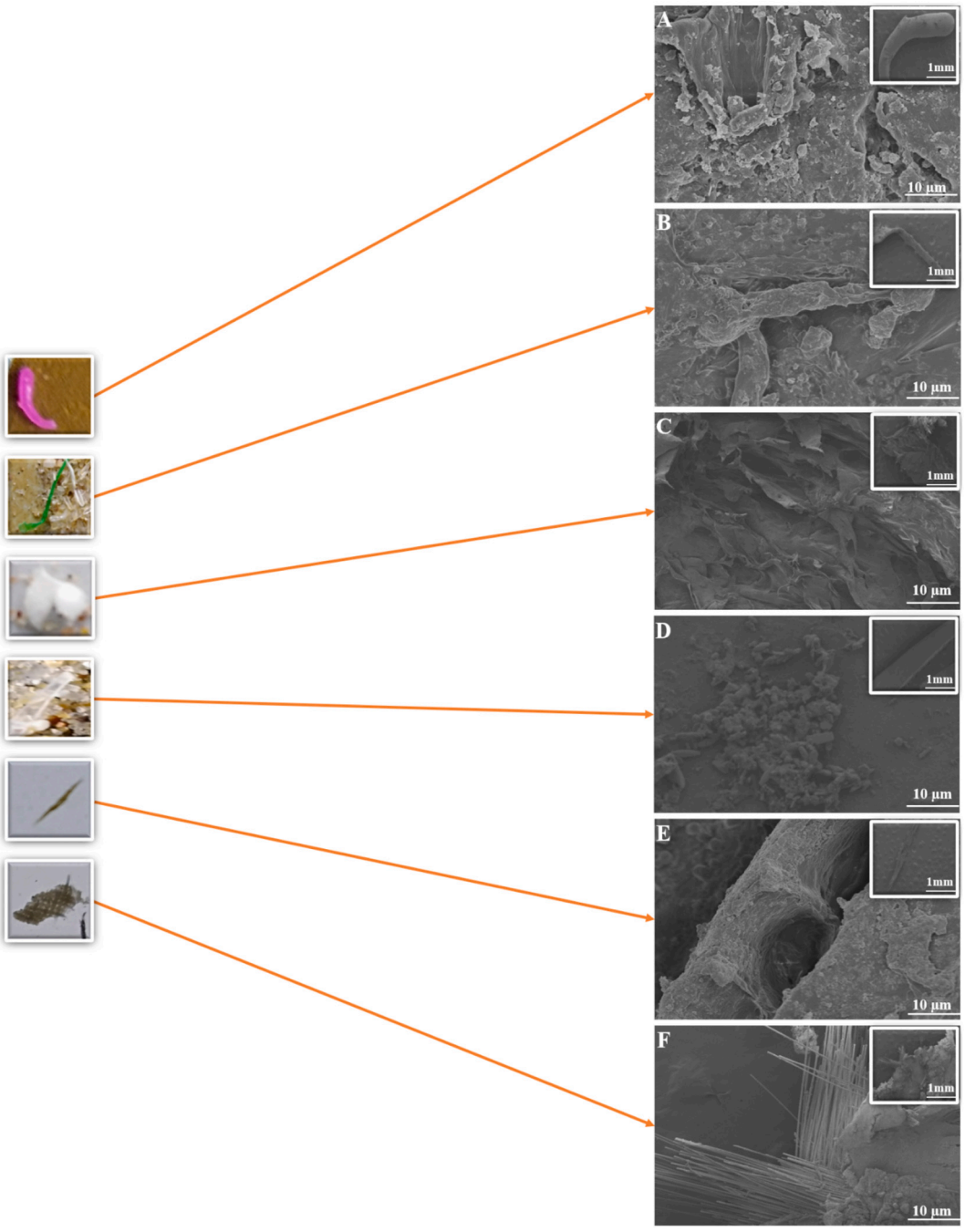

Fig. 6. SEM images of some of the identified microplastics.

hence are referred to as microplastics. Image J analysis showed that the size of the microplastics ranged from 3.3 to 4660 µm which were similar to the ranges reported for microplastics in sea salts in studies around the world. It is important to note also that this study is one of the few studies that report a size as low as 3.3 µm. For example, Kosuth et al. (2018) reported particle sizes of between 40 and 5000 µm for microplastics identified in sea salt samples. Kim et al. (2018) also reported particles sizes of microplastics in sea salts to be between 100 and 5000 µm. However, the two reported studies from Africa observed particles sizes of microplastics to be (160–980 µm) and (100–3000 µm) for microplastics for South Africa (Karami et al., 2017) and Senegal (Kim et al., 2018), respectively.

From the FT-IR analysed microplastics, salt samples from South Africa showed the highest concentration of microplastics which ranged between 0 and 1.33 ± 0.32 particles/kg, followed by Nigeria, Cameroun and Ghana with a value range of 0–0.33 ± 0.38 particles/kg each. Salt samples from Malawi, Zimbabwe, Uganda, and Kenya has no detectable MPs within the filter pore size (0.3 µm). It is possible that these numbers are underestimates since the FTIR instrument could not be used to analyse plastic particles lower than 50 µm (Klein et al., 2018). The possibility of the unconfirmed particles being of natural polymeric origin cannot be overruled as has been observed in another study (Kim et al., 2018). Also, Kim et al. (2018) established a correlation between microplastics abundance in table salts and the surrounding environment. Since all the table salt samples were of marine origin, the results of the study (the observed microfibres and microplastics) may reflect the extent of pollution in this system. However, there is a lack of data on the levels of micro-fibres/plastics pollution in most of the countries that

samples were collected. Hence, attempting to relate the abundance of micro-fibres/plastics in seawater and micro-fibres/plastics detected in table salts may be difficult. Nonetheless, as microplastics accumulate in the environment, there is a need to introduce the protocols for detection, isolation, quantification, and characterization of microplastics in the quality control procedure for table salts by various food monitoring agencies to assist in risk assessment.

As far as we aware only two studies have reported on microplastics concentrations in table salt samples from Africa, the countries being South Africa (Karami et al., 2017) and Senegal (Kim et al., 2018). The number of microplastics observed in this study for samples from South Africa was similar to the concentrations in sea salts (1–3 microplastics/kg) reported by Karami et al. (2017) from South Africa. On the other hand, the concentration of microplastics reported in sea salt samples from Senegal (250 microplastics/kg) (Kim et al., 2018) was higher than the microplastics observed in all the salts samples analysed in this study. It should, however, be noted that samples from Senegal were not included in this study. A growing body of research has also identified microplastics in table salts from other parts of the world. For instance, Yang et al. (2015) reported higher concentrations of microplastics (550–681 particles/kg) in sea salts, (43–364 particles/kg) in lake salts and (7–204 particles/kg) in rock/well salts in samples from China compared to our study. In another study, Karami et al. (2017) observed microplastics in 88% of salts samples analysed from eight countries (Australia, France, Iran, Japan, Malaysia, New Zeeland, Portugal, and South Africa). The microplastics concentrations ranged from 0 particles (French sea salt) to 10 particles/kg (Portuguese sea salt) which were similar to the amounts observed in our study. Renzi and Blašković (2018) reported somewhat higher concentrations of microplastics in all sea salt samples investigated from Italian (22 to 594 particles/kg) and Croatian (13,500 to 19,800 particles/kg) salt samples. Gündoğdu (2018) studied sea, rock and lake salts sample from Turkey and observed microplastics concentrations in the range of 16 to 84 particles/kg in sea salts, 8 to 102 particles/kg in lake salts, and 9 to 16 particles/kg in rock salts. Kim et al. (2018) analysed microplastics in salts samples from 21 countries and observed microplastics in 92% of the salts analysed. The concentrations of microplastics ranged between 0 and 1674 particles/kg (the authors exclude one outlier of 13,629 particles/kg) in sea salts, 0 to 148 particles/kg in rock salt, and 28 to 462 particles/kg in lake salt. Kosuth et al. (2018) identified microplastics in all salts samples analysed from different world regions (Atlantic Ocean, Celtic Sea, Himalayan region, Mediterranean Sea, Mexico, North Sea, Pacific Ocean, Sicilian Sea, USA, and Utah Salt Lake), with a concentration range of 47 to 806 particles/kg which was higher than reported in this study. It is obvious from the above studies that microplastics contamination in table salts should be of a major public health concern.

4. Conclusion

Human exposure to micro/plastic particles through the consumption of seafood products is currently a source of concern. In this study, we reported the presence and abundance of micro-fibres/plastics in some brands of African table salts. The levels of microplastic contamination recorded in salt samples are comparable to other studies. The total concentration ranged from 0.67 ± 1.15 particles/kg to 3.42 ± 4.94 particles/kg. On the basis of our results, the consumption of table salts in African countries represents a major source of micro-fibres/plastics exposure to humans. The types of microplastics detected in the salt samples show that they did not originate from the salt packages but likely from contamination either during salt production or from sea pollution. Nevertheless, this needs to be further investigated in order to reduce the contamination of salts used for human consumptions in African countries. In this regards, further studies are required to appropriately establish the sources of micro-fibres/plastics in table salts which may be from marine origins due to plastic pollution levels in aquatic systems or poor handling during salts production. We also suggest that future studies should include more detailed analysis of micro-fibres/plastics in salt samples for instance by using Pyrolysis GC/MS, as well as Raman spectroscopy to investigate the presence of small micro-fibres/plastic particles isolated in the salt samples.

CRediT authorship contribution statement

Oluniyi O. Fadare: Conceptualization, Methodology, Investigation, Visualization, Writing – original draft, Writing – review & editing, Resources. **Elvis D. Okoffo:** Methodology, Investigation, Visualization, Writing – review & editing, Resources. **Emmanuel F. Olasehinde:** Methodology, Investigation, Visualization, Writing – review & editing, Resources.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.marpolbul.2021.112006.

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