Litter & microplastics features in table salts from marine origin: Italian versus Croatian brands

Monia Renzi*, Andrea Blašković

Bioscience Research Center, Via Aurelia Vecchia, 32, 58015 Orbetello, GR, Italy

A B S T R A C T

This study estimates litter content, including microplastics, mesoplastic, and macroplastic in marine table salts coming from Italy and Croatia. Both high (HC) and low (LC) costs commercial brands easily found at the supermarket were analysed. Any macroplastic or mesoplastic were recovered while microplastics and other litter impurities significantly affect table salts of all tested brands. Average microplastic values ranged within 1.57 (HC) – 8.23 (LC) (Italy) and 27.13 (HC) – 31.68 (LC) items/g (Croatia). Microplastics sizes (min-max) ranged within 4–2100 μm (Italy) and 15–4628 μm (Croatia). In samples from both Nations, a significant general positive correlation between the average number of items/g recorded and the total amount of general impurities was recorded. Concerning microplastic shapes, in Italy, fragments dominated even if fibres, granules, films, and foams are frequently recorded. On the contrary, clear PP fibres dominated in Croatian brands even if also other shape classes were recorded.

1. Introduction

Plastic debris is currently a huge problem of global concern (Hollman et al., 2013) that affects the environment entirely. From sediments (Blašković et al., 2017; Cannas et al., 2017; Renzi et al., 2018a, 2018b) and water (Eriksen et al., 2013; Zettler et al., 2013) to the animals (Fossi et al., 2016) and humans (EFSA, 2016). Is widely established the transmission of the microplastic through the food chain (Avio et al., 2015). Now more than ever we are aware of the possible action of the plastic litter on human's health. Litter transfer from marine ecosystems to humans represent an important task that should be better described and clarified by the literature to achieve Horizon 2020 targets concerning the Marine Strategy Framework Directive principal purposes (2008/56/EC).

Humans are exposed by diet to plastic litter intake (EFSA, 2016) and seafood represent the most explored source of pollution coming from marine trophic web (Avio et al., 2015).

In coastal area, salt flats represent important coastal areas of great ecological and economical interest at the interface between land and sea and exposed, as coastal transitional ecosystems (Renzi et al., 2012; Renzi et al., 2013), to different kinds of human pressure. That activity could produce significant effects on plastic litter accumulation both in local trophic webs and commercial products. In spite of that, recent literature focuses their research on plastic pollution risks related to natural seafood and maricultured products (Avio et al., 2015; Dehaut et al., 2016; Pellini et al., 2018) and shows that seafood, as well as pelagic and benthonic fish species is affected by microplastics and, in particular, by fibres (Neves et al., 2015). Mussels and other filter feeding bivalves show a large number of individuals affected by the plastic litter (Karlsson et al., 2017; Van Cauwenberghe et al., 2015; Renzi et al., 2018a, 2018b). There are different types of table salts related to the salt origin as well as sea salt, lake salt, rock salt, river salt, well salt. Production processes are different for different salt types; sea and lake salts are obtained by water evaporation, rocky salt is obtained by mining, while river and well salt is obtained from wells in non-coastal zones (Iñiguez et al., 2017).

Very few studies are performed on table salt from marine origin that represent another important commercial product coming from the sea and potentially affected by marine pollution by plastic litter. Two studies performed recently evaluated levels of plastic litter in table sea salts (Iñiguez et al., 2017; Karami et al., 2017). Karami et al. (2017) performed plastic litter determinations on 17 salt brands from 8 different countries and evidenced that microplastics (171–515 μm) were present in almost all brands at concentrations included between 1 and 10 MPs/kg of salt. Related to shape, authors evidenced that fragments were dominant (63.8%) followed by fibres (25.6%) and films (10.6%). A research performed in Spain by Iñiguez et al. (2017) on 21 different samples of commercial table salts, evidenced ranges within 50–280 MPs/kg salt with any differences between samples due to the origin (sea salts and well salts) and treatments (before and after packaging).

* Corresponding author.
E-mail address: monia.renzi@bsrc.it (M. Renzi).

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Nevertheless, researches on this field are far to be exhaustive.

The aim of this study was to evaluate litter content, including microplastics, in eleven different commercial brands of iodate fine table salts produced in by six different Italian brands and five Croatian brands of sea salt produced locally. Analysed salt samples were easily found at the supermarket and sold in carton (Italian; Croatian) and plastic (Croatian) packs. Total amounts, principal features of recovered litter (including microplastic, mesoplastic, macroplastic), and possible differences among high cost (HC) and low cost (LC) brands were checked.

2. Materials and methods

Eleven different brands of table salt of marine origin were selected in this study. All selected brands were fine sea iodate salt. Commercial name of products cannot be made public for privacy reasons nevertheless commonly used brands were selected to improve representativeness. We sampled six Italian and five Croatian brands available at the supermarkets coming from different production plants. Geographical locations of the production site of sea salt were not always indicated on packaging but sampled sea salts were always declared to be national produced. Italian brands were all sold in carton packs; only one brand from Croatia was sold in carton while the other four brands were sold in clear plastic envelopes. Selection was performed on the basis of the final cost: three high cost (0.45–0.50 Euro, named HC1, HC2, HC3) and three low cost (0.35–0.37 Euro, named LC1, LC2, LC3) brands were selected in Italy. Also Croatian brands were grouped concerning costs (high cost HC4, HC5, HC6; low cost LC4, LC5). Salts were sampled statistically to ensure representativeness.

For each experimental replicates, 360 g of sea salt was completely dissolved in 1 L of deionized water mixed for 20 min in a glass backer and filtered using a filtrating vacuum pump apparatus on a filter fibre (0.45 μm) disks successively dried in oven at 40 °C. Total suspended solids (TSS, method APAT CNR IRSA 2090) were determined on a solution of 200 g/L of salt samples in statistic replicated to evaluate salt contamination by total solid materials. In this case, fibre filters were weighted before and after filtration of the salt solution and successively dried in oven at 60 °C till constant weight. Observations were performed by stereomicroscopy (Nikon, SMZ-800 N). A rigid protocol of identification was applied to minimize operator classification mistakes as reported: (1) no structures of organic origin should be visible; (2) fibres should be equally thick and have a three-dimensional bending to exclude a biological origin; (3) particles should be clear and homogeneously coloured; (4) transparent or whitish particles must be examined under high magnification to exclude a biological origin (Löder and Gerdts, 2015).

Classifications were performed according to type, shape, size, and colour following criteria reported by the literature (Hidalgo-Ruz et al., 2012; JRC EU, 2013; Galgani et al., 2013; Alomar et al., 2016). Multicolour and Violet colour classes were added to the classification reported by classification criteria adopted by the literature (Galgani et al., 2013; Fastelli et al., 2016). Coloured images were collected by high-resolution digital camera linked to the microscope (Nikon, DS-Fi2). Nikon's software for the imaging analysis was applied to the litter dimensional measurements (Nikon ACT-1). Experimental blanks were performed to evaluate possible crossover contamination during air exposure in laboratory as detailed reported by previous studies performed by the research group (Fastelli et al., 2016; Blašković et al., 2017). Confirmations were performed by μFT-IR (Nicolet iN10 MX, Thermo Scientific) technique.

3. Results

Plastic litter recovered during this study are represented in Fig. 1. In particular, black foam (Fig. 1a); tubular unrecognized black (Fig. 1b); multicolour fragment (Fig. 1c); violet fibre (Fig. 1d); various granules (Fig. 1e, f); clear fibres (Fig. 1g); plastic knot (Fig. 1h) of determined microplastics are reported.

General features of tested samples are reported in Table 1. In particular, in Table 1, two price-sizes are reported grouping samples in two classes: High Cost (HC) and Low Cost (LC) brands. HC1 and HC3 are produced in the same geographical area (North-Adriatic Sea) but are sell as different brands. On the contrary, Italian LC table salt is produced from different regions (Southern Tyrrhenian, Southern-Adriatic, and Northern-Adriatic seas). As regards as Croatian marine analysed salts, originated from two different areas. Any macro- or meso- plastics are recovered. All recovered plastic litter are microplastics ranging within 4–2100 μm (Italian salts) and 15–4628 μm (Croatian salts). Statistics performed evidenced a significant difference ($p < 0.01$) among HC and LC brands concerning n. items/g. Total Suspended Solids (TSS, μg/g of salt) is considered as indicator of the total amount of impurities. A comparison among average levels of TSS and microplastic average items/g of salt measured is reported in Fig. 2. Data evidence a general positive correlation between the average amount of MPs items and TSS recorded (Test T; $p < 0.001$) in both Nations.

Average (SD) shape frequencies of microplastics recorded are reported in Table 2 as percentage of each shape versus the total amount recorded. Concerning Italian salts, fragments are the dominant percentage of microplastic recovered followed by fibre, films, foam, and granules. The percentage of unrecognized shape class is closed to 2% in almost all LC brands. Percentages are quite similar in tested brands evidencing a common structure of the shape distribution even if considering number of items/g LC brands evidenced numbers generally higher than HC ones. On the contrary, Croatian salts show a percentage of fibres exceeding 80% in almost all analysed samples. Films, fragments and granules are also recovered. In Fig. 3, average colour fingerprint of tested table salts is reported for both LC and HC brands and expressed as average % (SD). As observed by the figure, colour fingerprints are similar on a National basis even if percentages of items/g of salt are generally higher in LC compared to HC brands. In Italian brands, black is the dominant colour followed by grey and blue. Orange, brown, green, pink, yellow and violet are represented in each brands. High frequencies of multicolour microplastics (Fig. 1) are recorded in both price-sizes even if higher levels are recorded in LC ones. As regard as Croatian salts, the clear colour is the principal observed in recovered microplastics followed by blue, black, white, and yellow colours. Concerning microplastic size, a wide range of dimension is recorded in both Croatian and Italian salts (Table 1) even if the size class < 100 μm of maximum diameter is the most represented for both HC and LC prize-size of table salt tested.

4. Discussion

Sea salt production is performed in salt flats by pumping marine water into evaporation ponds where wind and sun increase evaporation rates and determine the precipitation of salt crystal on the bottom of ponds. Crystallized salt is cut and collected and subjected to different physical processes before packaging (Iñiguez et al., 2017). On the basis of the results obtained by this study, all Italian and Croatian sea salts tested resulted polluted by microplastic and other impurities. Total average measured by this study in tested samples of different brands ranged within 1.6–8.2 (Italian) and 13.5–19.8 (Croatian) items per gram of salt. These levels are significantly higher values compared to data reported by literature (Karami et al., 2017; Iñiguez et al., 2017). Observed higher levels should probably be related to different factors. First of all, possible overestimations of plastic numbers could occur for the particles with a diameter minor than 500 μm during the first step of visual identification of plastic items. Literature considers visual determinations as a valid method for particle dimension larger than 500 μm (Hidalgo-Ruz et al., 2012) while reports a percentage of classification mistakes by visual identification ranging within 20% (Eriksen et al., 2013) - 70% (Hidalgo-Ruz et al., 2012) that increases with the

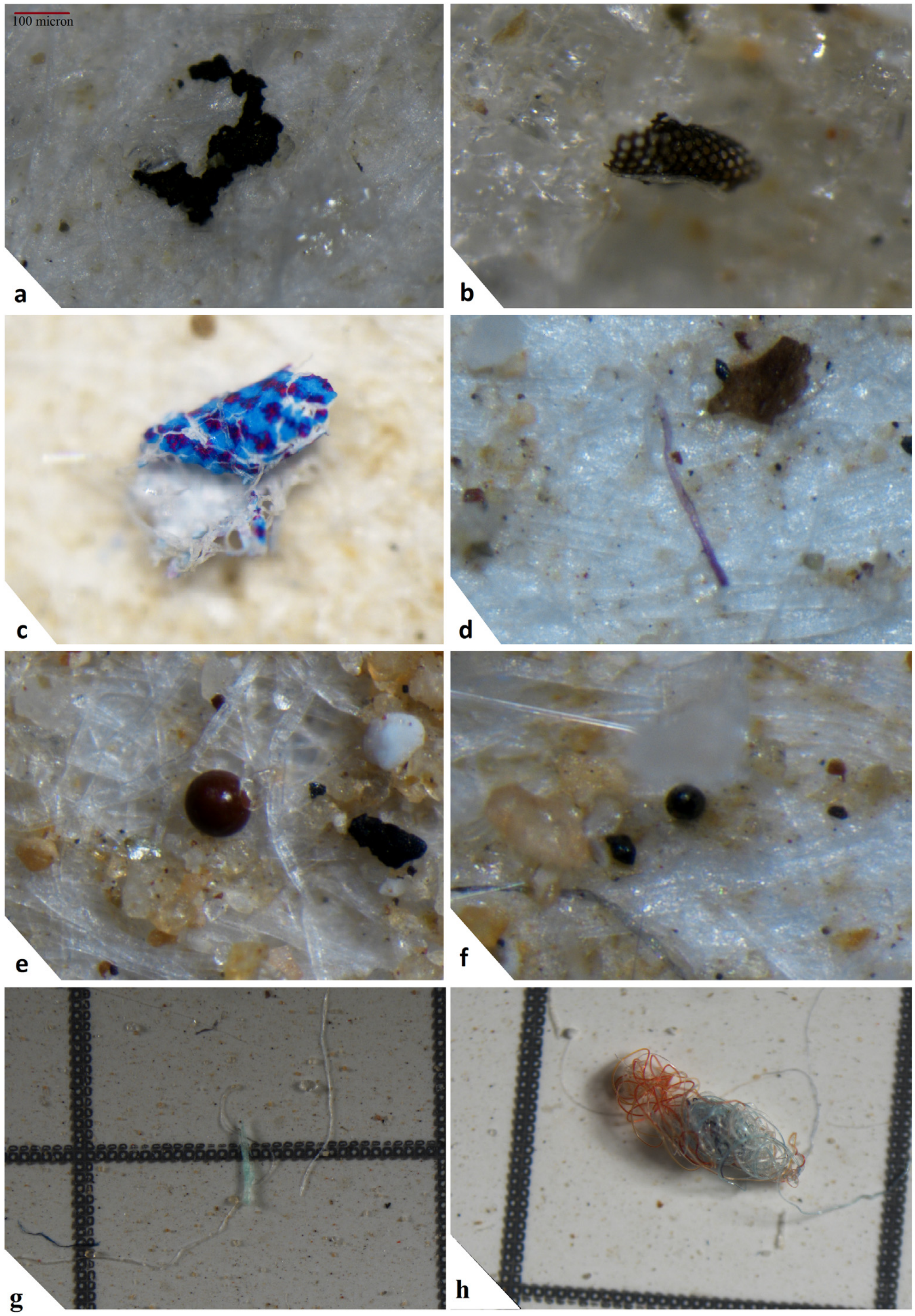

Fig. 1. Plastic litter recovered during this study in Italian (a–f) and Croatian (g–h) analysed salts. Images are to be considered only representative of the whole variability in terms of type, shape, colour of the litter recovered. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

decreasing of the particle size. If we consider only solid data (i.e. data on particles higher than 500 µm), results obtained in this study as average number of items per kg of marine salts are consistent with levels reported for Spain table salts of marine origin supporting the hypothesis that Italian and Croatian table salt are more polluted than Spanish salt as Italian and Croatian solid values are included respectively within 22–594 and 13,500–19,800 items/kg table salt.

Another possible cause of observed higher values of microplastic pollution in Italian and Croatian table salt compared to other countries is due to pollution of marine water and production sites. Salt production for human diet starts from the evaporation process of marine water in natural coastal salty lagoon areas. This process required the air exposure to very long time of saturated salty water to deposition process and this phase is sensible to the accumulation onto salt crystals of plastic litter that are present in marine water and plastic litter absorbed from the air through air deposition due to winds (Iñiguez et al., 2017). In Italy and Croatia, coasts represent over 8000 km of extension; nevertheless, for historical and commercial reasons, salt flats are usually not far from urban settlements and from river inputs as the coastal area is highly overexploited by human settlements and human

Table 1
General features of tested samples. Italian (1–3) and Croatian (4–6) iodate fine marine table salts analysed in this study are grouped per Nation of production. Price-size is, also, reported grouping samples in two classes: High Cost (HC) and Low Cost (LC) brands. Total Suspended Solids (TSS, μg/g salt) are reported as average (standard deviation, SD) as general indicator of the presence of impurities. Recorded micro-litter is reported as average number of items/g of salt (SD) and as average size (minimum- maximum length, μm).

Production data		TSS (μg/g)		Litter items/g d.w.		MPs size (μm)	
Nation	Price-size	Average	SD	Average	SD	Minimum	Maximum
Italy	HC1	85	7	1.57	0.39	7	1839
	HC2	172	30	5.66	1.00	14	2014
	HC3	122	35	3.47	0.94	15	1391
	LC1	128	28	8.23	2.58	4	1761
	LC2	220	57	7.64	0.87	11	1524
	LC3	152	57	6.01	1.63	12	2100
Croatia	HC4	195	7	24.5	1.8	31	3531
	HC5	105	92	27.9	1.4	24	2082
	HC6	165	78	29.0	2.9	26	3847
	LC4	430	28	39.8	9.4	44	4628
	LC5	270	57	23.5	1.5	15	1227

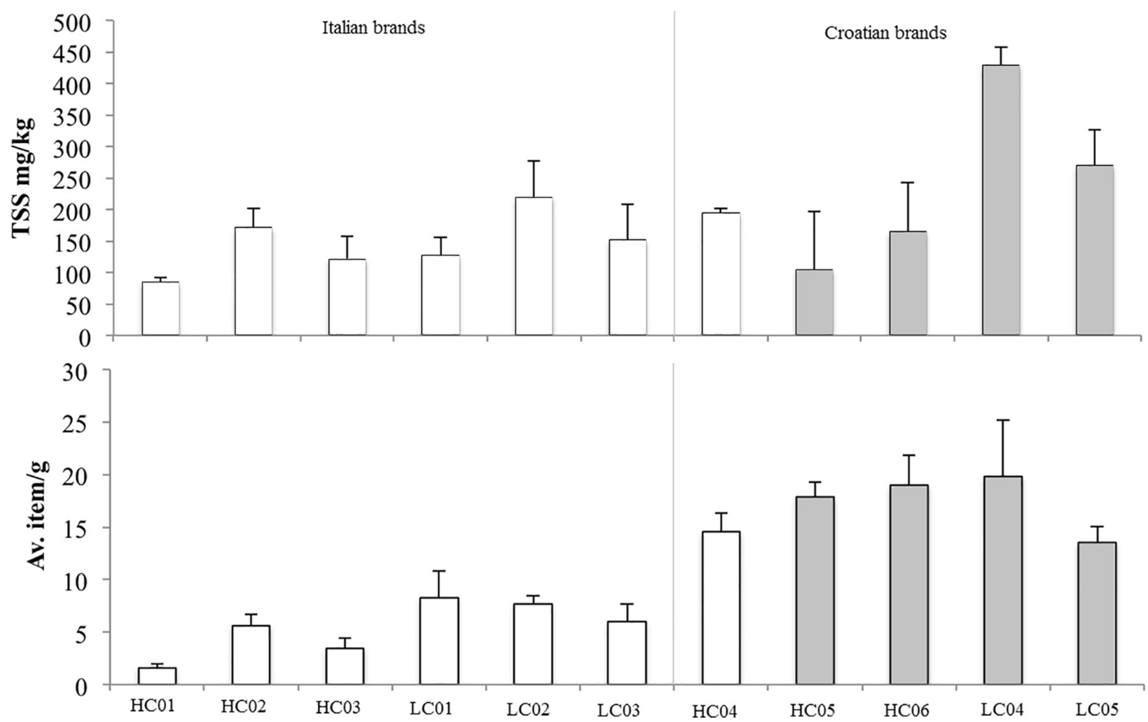

Fig. 2. Comparison among total impurities and microplastic recorded. Data are represented as averages (+SD) evidencing prize-size and Nation for both TSS and microplastic items. Bar colours are referred to the packaging used (white = carton; grey = plastic packs).

Table 2
Microplastics shape according to the price-size. Data are reported as average number of items (expressed as percentage) related to the total average number of items determined in each sample. Data are grouped according to shape classes proposed by the literature (Galgani et al., 2013). Italian trademarks are numbered from 1 to 3 while Croatian from 4 to 6.

	Fiber (FI)	Film (FILM)	Fragment (FR)	Granule (G)	Pellet (P)	Foam (FO)	Unrecognized (UN)
HC1	25.3	10.6	47.6	6.5	0.0	8.8	1.2
HC2	20.4	17.1	43.1	6.6	0.0	12.2	0.6
HC3	18.9	15.3	52.3	4.5	0.0	9.0	0.0
LC1	17.9	10.3	46.4	6.5	0.0	17.5	1.5
LC2	16.4	10.7	37.7	6.6	0.0	27.0	1.6
LC3	26.6	12.5	42.2	8.3	0.0	8.9	1.6
HC4	82.1	10.7	7.1	0.0	0.0	0.0	0.0
HC5	79.1	7.0	0.0	14.0	0.0	0.0	0.0
HC6	87.4	5.5	4.3	2.8	0.0	0.0	0.0
LC4	98.8	0.0	0.0	1.3	0.0	0.0	0.0
LC5	80.6	3.2	6.5	9.7	0.0	0.0	0.0

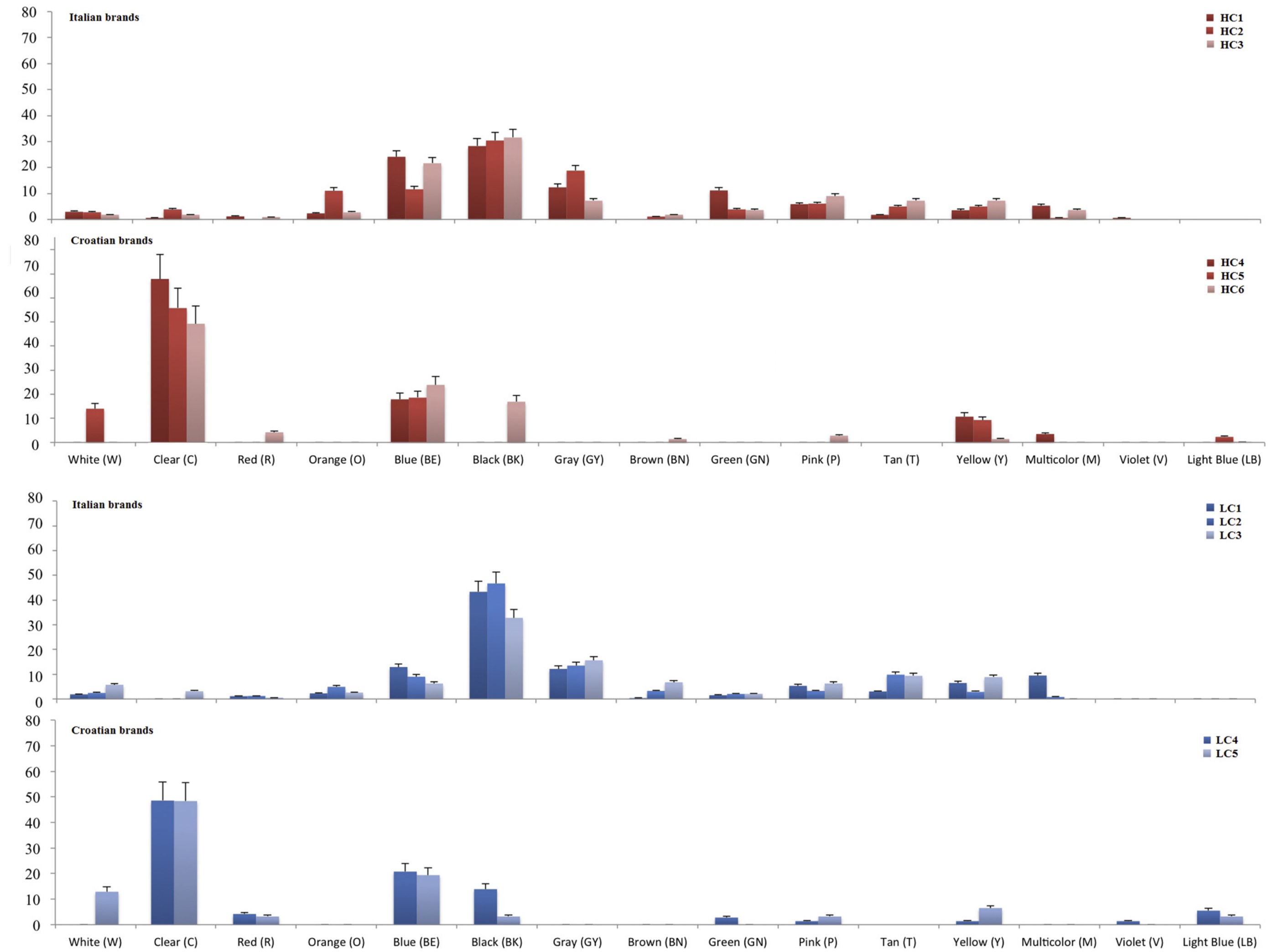

Fig. 3. Fingerprints of average colours. Data are reported as average percentages (+SD) and grouped according Nation and salt price-sizes. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

activities. Also marine ecosystems along Italian coasts are highly overexploited so microplastic pollution could be significantly high in Mediterranean Sea and in particular along Italian and Cratian coastal areas than along Spanish coasts facing on Mediterranean Sea or Spanish coast facing Atlantic Ocean.

Significant differences are reported in this study concerning salt price-size in Italian and Croatian salts. Low cost Italian brand range within 344–594 average items/kg salt, values that are higher than Spanish range 50–280 items/kg calculated on the whole size class range (Iñiguez et al., 2017) and not only on the > 500 μm as in this case. Concerning Croatian table salts, HC salts are less polluted than LC ones even if differences between price-size are < 15% on average. Even if further studies are needed to clarify this aspect, it could be due to the less effective purification process applied to realize low cost salts that could determine a significant increase of both microplastic and other impurities (i.e. black carbon, paper fragments, sediments, etc.) in commercialized products. A better purification technique during productive process could improve salt quality and reduce final product impurities.

As regards as total amount of microplastic recovered, Croatian marine salt tested in this study is, on average, higher contaminated by microplastics than Italian table salt (respectively 28.9 versus 5.4 items/g of salt) and Croatian salt resulted about 5 times more polluted than Italian salt. Even if further researches should be performed to clarify Italian and Croatian brands chemical composition, μFT-IR analyses were performed to evaluate if the large amount of recovered clear fibres in Croatian table salt samples could be due to the release from the plastic envelope used in the packaging process or during the opening of the packages before the use or before the analysis. Results obtained testing a percentage of clear fibres collected excluded the correlation between the envelope and the presence of fibres inside the product. A recent study reported that PET (polyethylene-terephthalate) is the principal component of plastic found by literature in table salt (Yang et al., 2015), probably due to the high density of PET (1.30 g/cm^3) in comparison to PE (0.94 g/cm^3) and PP (0.90 g/cm^3). This feature causes PET to remain with the salt during the crystallization process in the salt production. This research evidences that analysed clear fibres founded in Croatian salts are not related to the PE envelop used during packaging but are PP fibres (Fig. 4). This fact is also supported by the presence of high percentages of clear microplastic fibres also in salt packaged in carton envelope. The presence of PP fibres in Croatian salts could be originated from clothes dressed by operators during the industrial process. Also, fibres could be originated from the pollution of marine water used for the salt production by municipal wastewater effluents. Further studies are needed to better understand PP fibres origin.

The amount of salt intake in the adult's diet according to the World Health Organization (WHO) should not exceeded 5 g/day (Iñiguez et al., 2017). In Table 3, MPs intakes by salt consumption per day calculated considering the suggested maximum daily dose by WHO are

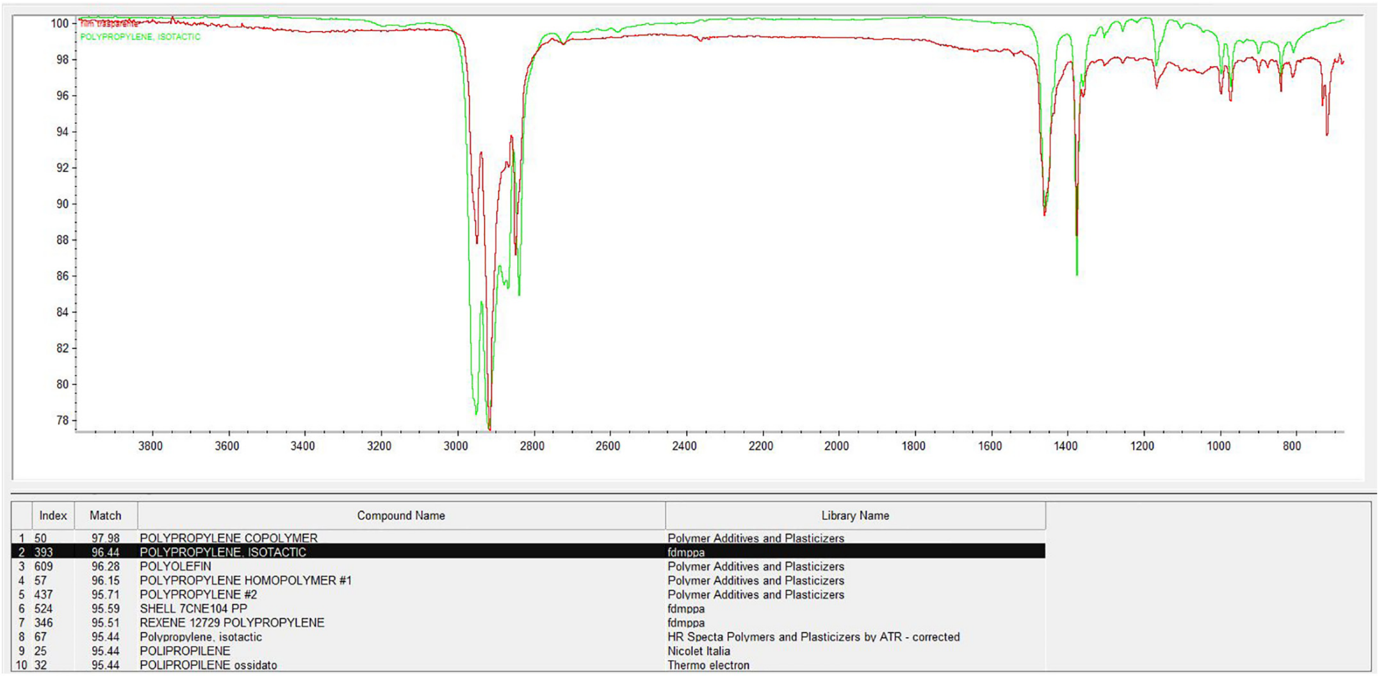

Fig. 4. Chemical analysis performed by μFT-IR of clear fibers collected in table marine salts from Croatia to evaluate possible pollution by packaging.

Table 3
Microplastic features normalized according literature. Table salts analysed in this study are reported separately for each Nation, and brand. Price-size is reported grouping samples in two classes: High Cost (HC 1–6) and Low Cost (LC 1–5) brands. Italian brands are numbered from 1 to 3 while Croatian from 4 to 6. Litter as average number of items/g of salt (SD) is reported both as total amount and as confirmed microplastic items (solid data). Human intakes are calculated on the basis of the maximum suggested salt assumption dose (5 g/day) by WHO. Italian people exceed daily-recommended values by the assumption of about 12 g/day (unpublished data). For this evaluation, we assumed that the whole suggested daily dose is due to the external addition of fine marine salt to injested foods.

Price size	Litter total amount			Microplastic items		
	Items/g		Human intake/day	Items/g		Human intakes/day
	Aver.	SD	WHO	Aver.	SD	WHO
HC1	1.6	0.4	7.9	0.0	0.0	0.1
HC2	5.7	1.0	28.3	0.2	0.1	0.8
HC3	3.5	0.9	17.4	0.3	0.1	1.3
LC1	8.2	2.6	41.2	0.6	0.4	3.0
LC2	7.6	0.9	38.2	0.3	0.2	1.7
LC3	6.0	1.6	30.0	0.6	0.1	2.8
LC4	39.8	9.4	199.0	14.5	1.8	72.6
HC4	24.5	1.8	122.5	17.9	1.4	89.4
HC5	27.9	1.4	139.5	19.0	2.9	95.0
LC5	23.5	1.5	117.5	19.8	5.4	99.1
HC6	29.0	2.9	145.0	13.5	1.5	67.7

calculated. According to these data, annual MPs intakes for Italian peoples ranging within 40.6–1,085.2 items/year. Intakes for HC price-size brands are similar to values reported for Spanish people (510 items/year; Iñiguez et al., 2017) at the suggested salt intakes but higher than Spanish people considering low cost brands. Croatian annual intakes are about 50 times higher than values calculated for Italian people.

5. Conclusions

Salt flats represent coastal areas of particular interest due to the presence of high human pressures but also high human productive interests. This research evidences as litter and microplastics pollute Italian and Croatian fine iodate table salts from marine origin. This research should be of some inputs: i) to better define production areas and industrial steps affecting microplastic levels in commercial products; ii) to reduce impurities in final products; iii) to reduce associated exposure by consumption. Future researches should be performed to better define on a chemical basis microplastic features in sea salts and to define the origin of observed pollution to propose technical strategies to be applied by producers to reduce impurities and to improve salt quality. Furthermore, this study should help stakeholders to better understand problems associated to litter pollution and to define well-sized strategies to correctly face and control the problem.

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