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Proceedings of the International Conference on Microplastic Pollution in the Mediterranean Sea

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Contents

Sub-Basin Scale Heterogeneity in the Polymeric Composition of Floating Microplastics in the Mediterranean Sea

Giuseppe Suaria, Carlo Giacomo Avio, Francesco Regoli and Stefano Aliani

1 Introduction

Mainly owing to limited outflow of surface waters, a densely populated coastline and intensive fishing, shipping, touristic and industrial activities, substantial amounts of plastic litter are accumulating in the Mediterranean Basin which—together with the main five oceanic gyres—is now recognized as one of the greatest accumulation zones for floating plastic debris in the world [1–5].

The generic term "microplastics", however, encompasses a wide range of different polymers, while most of the field studies mainly relied on the visual identification of particles or characterized only a restricted subset of them. Thus, detailed knowledge of the actual polymeric diversity of this emerging pollutant is lacking. Such information is urgently required to identify sources and sinks of microplastics and to better understand fate and impacts of the different polymers at sea, so that knowledge-based reduction and prevention measures can be effectively implemented.

Here, we present the results of a large-scale survey of floating microplastics (<5 mm) and meso-plastics (5–20 mm) in central-western Mediterranean waters, providing the largest polymeric characterization ever performed (n = 4050 particles). We confirm the Mediterranean Sea as severely contaminated by plastic pollution and describe for the first time the complex mixture of synthetic polymers floating on its surface, testing the hypothesis that plastic distribution and composition are not homogeneous and that geographical differences exist between sub-basins.

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2 Materials and Methods

2.1 Sampling and Laboratory Analysis

74 offshore samples were collected during two expeditions carried out in the Mediterranean Sea on board the Italian research vessel Urania between May 9 and June 24 2013. Samples were collected using a 200 μm Neuston net towed for ~5 min at a speed of 1.5–2 knots. Once on board, the plankton samples were transferred to 50 mL falcon tubes and fixed with 80% EtOH. In the laboratory, all samples were examined under a dissecting stereomicroscope by two different researchers to reduce operator bias during sorting. Plastic particles were carefully hand-picked using laboratory tweezers and transferred to glass jars. All particles were then counted, weighed, dried and classified according to their shape and colour. Due to the high risk of airborne contamination, all filaments and fibres suspected of having a textile origin were not considered in density calculations. Total plastic concentrations (expressed as g/km^2 and particles/m^2) were then computed and plotted in Fig. 1.

2.2 FTIR Analysis

The polymeric identity of all collected particles >700 μm (n = 4050) was verified through FTIR analyses. The subset was considered highly representative since it comprised 96.16% of the total weight of collected plastic. Analyses were performed using a Cary 660 FTIR spectrometer (Agilent) equipped with ATR (GladiATR Diamond Crystal Plate, Pike technologies). 128 scans per particle were performed, and CO_2 interference (2300–2400 cm^{-1}) was removed for clarity. For each particle,

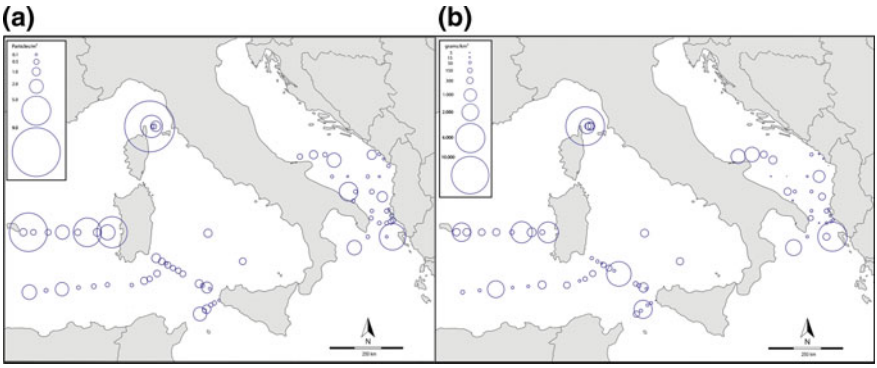

Fig. 1 Map of the study area showing the location of all sampling stations and the distribution of total plastic concentrations expressed as **a** number of particles/m^2 and **b** grams/km^2. Reprinted from [4], Sci. Rep. 6, 37551 under open copyright licence

scans were performed with a resolution of 4 cm^{-1}. Agilent Resolution Pro v5.2 was used for the output spectra, and identification of polymers was performed by comparison with a library of standard spectra. Only polymers matching reference spectra for more than 60% were accepted.

2.3 Statistical Analysis

Principal component analysis (PCA) was based on a variance–covariance matrix of the relative frequencies of the seven most common polymers: PP, PE, PS, PA, PVC, nylon and paint. Mann–Whitney U test was used to verify significant differences between sub-basins. Normality and homogeneity of variance was checked, and the level of statistical significance was set at $p < 0.05$.

3 Results

Plastic-like particles were found in all samples with a mean total concentration of 1.25 ± 1.62 particles/m^2 and 703.16 ± 1573.95 g/km^2. Plastic concentrations showed a very high spatial heterogeneity spanning two or three orders of magnitude across the study area. The highest value (9.23 particles/m^2) was found in the Corsica Channel, while the lowest concentration was observed in the southern Adriatic (0.04 particles/m^2). Overall, plastic particles were significantly ($p = 0.002$) less abundant in the Adriatic Basin (0.83 ± 1.05 particles/m^2; 485.07 ± 1153.07 g/km^2; $n = 30$) than in the western Mediterranean (1.54 ± 1.87 particles/m^2; 851.85 ± 1803.66 g/km^2; $n = 44$). Most of the 14106 collected particles were visually classified as irregularly shaped fragments (93.2%), while pellets, films and foams constituted only a small fraction of the total (2.2, 1.6 and 3.1%, respectively).

FTIR analysis revealed the presence of 16 different polymers (Fig. 2). Polyethylene (HD-PE and LD-PE) was the predominant form with an overall frequency of 52%, followed by polypropylene (PP) (16%) and synthetic paints (7.7%). Polyamides (PAs) accounted for 4.7% (excluding nylon which accounted alone for 1.9%), whereas polyvinyl chloride (PVC), polystyrene (PS) and polyvinyl alcohol (PVA) represented 2.6, 2.8 and 1.2%, respectively. Other less frequent polymers (<1%) included polyethylene terephthalate (PET), polyisoprene (synthetic rubber), poly (vinyl stearate) (PVS), ethylene-vinyl acetate (EVA) and cellulose acetate. Ten fragments of polycaprolactone, a biodegradable polymer, were found in seven different samples throughout the study area, while 201 fragments of epoxy resin (polyepoxide) were collected in the Balearic Sea. Similarly, residues of paraffin wax were exclusively found in an offshore sample in the Adriatic Sea. The molecular characterization also revealed that 4.4% of all analysed particles did not consist of plastic but were rather made of cotton, chitin, cellulose and other non-synthetic materials.

Geographical differences between Mediterranean sub-basins were found also in the relative occurrence of different polymers (Fig. 3). The composition of western Mediterranean samples was dominated by low-density polymers such as PE and

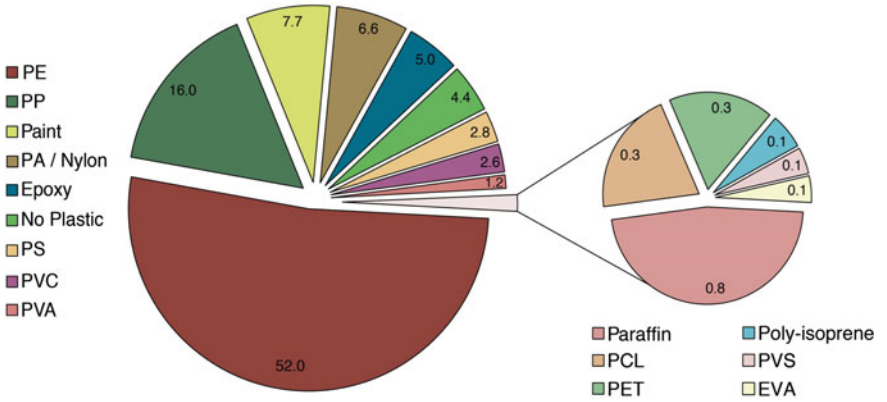

Fig. 2 Polymeric composition of all particles >700 μm characterized through ATR FTIR analysis (n = 4050 particles). Values are expressed in percentages. Reprinted from [4], Sci. Rep. 6, 37551 under open copyright licence

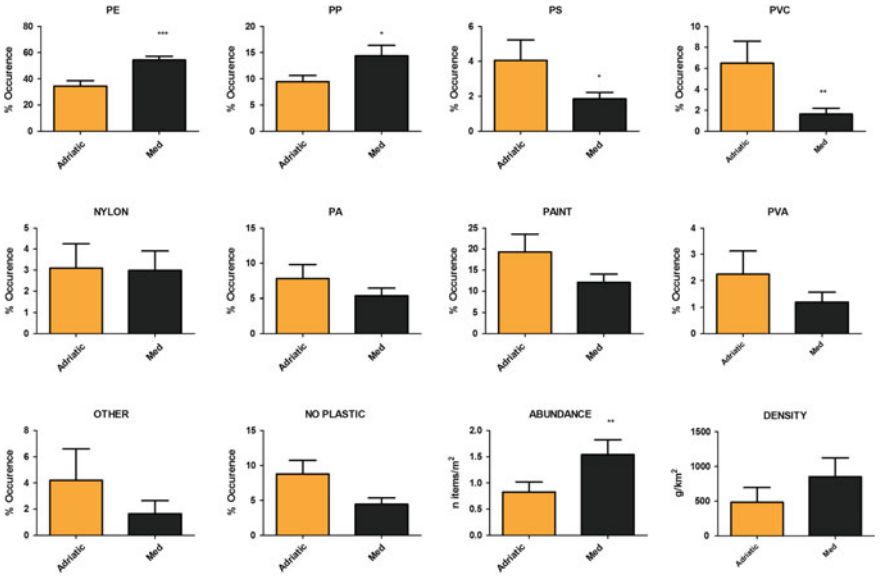

Fig. 3 Differences between Adriatic (n = 30) and western Mediterranean samples (n = 44) in the relative frequencies of the most common polymers identified through ATR FTIR (n = 4050 particles). Differences in the total abundance (items/m^2) and density (g/km^2) of plastic particles are also shown. Reprinted from [4], Sci. Rep. 6, 37551 under open copyright licence

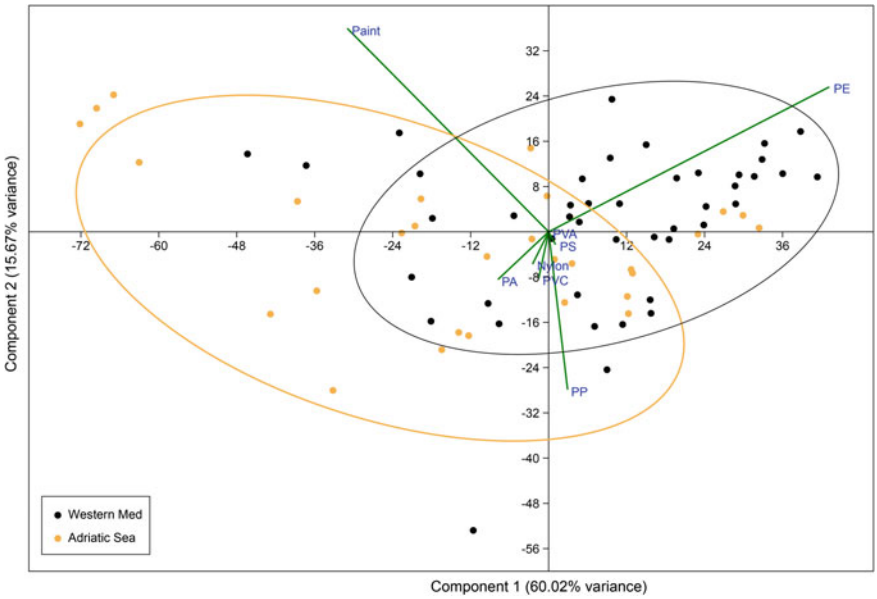

Fig. 4 PCA ordination based on the occurrence of the seven most common polymer typologies. Orange dots represent Adriatic samples (n = 30), while western Mediterranean samples are in black (n = 44). Distance biplot of the eigenvectors is not in scale with data points. Reprinted from [4], Sci. Rep. 6, 37551 under open copyright licence

PP. Adriatic samples instead were more heterogeneous and rather characterized by a higher presence of paint chips, PS, PVC, PVA and PAs. PCA ordination of samples produced a two-dimensional pattern, with the first two components explaining 75.7% of the total variance (Fig. 4). Despite some overlapping, most of the separation between sub-basins occurred along PC1 axis, mainly referring to PE (0.80) and paint (−0.57). On the other side, PP (−0.52) determined most of the separation along PC2.

4 Discussion and Conclusions

No clear accumulation pattern and a high small-scale variability in plastic abundance and composition emerged from our survey, similar to what was previously reported in the same area for floating macro-debris [4]. With this respect, the formation of permanent accumulation zones in the Mediterranean Sea is probably hampered by the highly dynamic character of the surface circulation, of which the observed heterogeneity in plastic distribution is very likely a reflection.

Our concentration values are substantially higher than most studies performed in the Mediterranean Sea. Nevertheless, a closer agreement is obtained when comparing

densities expressed in terms of mass concentrations, rather than particle counts. After removing two outliers from our data set for instance, our mean density drops to 463.5 g/km^2, which is incredibly similar to the values reported from the inner accumulation zones of all main oceanic gyres [6] and to the values of 423 and 579.3 g/km^2 obtained in two other large-scale surveys of the Mediterranean Sea [3, 5].

The proportion of different polymers found in our study roughly corresponds to the global production stocks of plastic materials, with polyolefins (PE and PP) accounting for 62% of the global plastic demand [7] and for 68% of our collected particles. Being widely used in the disposable packaging industry and having lower densities than sea water, it is not surprising that polyolefins consistently account for the majority of the plastic particles floating in surface waters worldwide. Also being less susceptible to sinking, the contribution of these low-density polymers has been shown to increase with distance from land [8], hence potentially representing a proxy of the distance from pollution sources. From this perspective, the higher heterogeneity in the polymeric composition of Adriatic samples, together with a higher occurrence of high-density polymers, would indicate shorter residence times of particles at sea and a closer proximity to pollution sources, likely reflecting the distinctive hydrological features of the Adriatic Basin. Although the mechanisms through which high-density polymers can persist at the sea surface have to be clarified, the presence of paint and paraffin wax seems to suggest a high influence of ship-based pollution in the Adriatic Basin. Paint chips are typically generated during repair, maintenance and cleaning of vessel decks and hulls, and large quantities of synthetic paints have been related to intense shipping activities in Korean waters [9]. Paraffin wax, on the other hand, is transported in large quantities by cargo ships, and tank-washing residues may be legally discharged at sea beyond the 12 nautical miles, according to MARPOL regulations. Paraffin clumps were previously reported along the coasts of the North and Baltic Sea and in the stomach contents of northern fulmars [10]; nevertheless, its presence in Mediterranean waters had never been explicitly recorded before.

Polycaprolactone (PCL) instead is a synthetic polyester which is marketed as biodegradable, even though only some signs of degradation appear after 12 months in the marine environment [11]. PCL fragments were found in 9.5% of our samples, and its presence in Mediterranean offshore waters provides further evidence that some “biodegradable plastics” do not readily degrade in natural conditions, thus not representing an a priori solution for reducing marine litter.

In conclusion, our results demonstrate the pervasiveness of plastic pollution in Mediterranean waters and, confirming model predictions, provide further evidence that microplastic abundances are amongst the highest in the world. Sinks, sources, fate and residence times of different polymers at sea are the main knowledge gaps that need to be addressed, so that the global mismatch between plastic sinks and inputs can be eventually explained. The polymeric characterization of microplastics is of paramount importance for the effective identification of specific solutions and alternatives. However, irrespective of different sources and typologies, the problem of plastic pollution is a social and behavioural issue, whose causes required to be mostly sought upstream the consumption chain.

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Floating Microplastics in the Northwestern Mediterranean Sea: Temporal and Spatial Heterogeneities

Mel Constant, Philippe Kerherve, Jennifer Sola, Anna Sanchez-Vidal, Miquel Canals and Serge Heussner

1 Introduction

Attention on marine litter, and their economic, social, and environmental issues, has been increasingly paid over the last few decades [1]. Litter has been found everywhere, from rivers to central open oceans, in coral reefs and polar oceans, floating at the sea surface, deposited on the seafloor, stranded on beaches or ingested by marine fauna (e.g., [2–7]).

Plastics can represent more than 70% of items described within floating marine litter [8]. Among plastic litter, an important part has a size below 5 mm (e.g., >98% [9]). These small size particles, called microplastics (MPs), can be ingested by small organisms, extending the problem to a large part of the marine trophic network.

Mediterranean ecosystems are exposed to high anthropogenic pressures such as densely populated coastlines, busy shipping routes, and strong tourism activities. In addition, the semi-enclosed morphology of the Mediterranean Sea leads to an accumulation and a density of marine litter as important as within the great accumulation zones in the oceanic gyres [10].

The Mediterranean Sea is one of the most sampled areas for floating MPs. However, only a few investigations have been conducted at small spatial and temporal scales in coastal areas. To overcome this gap, MPs (<5 mm) were

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collected off the mouths of two contrasted rivers discharging into the Northwestern Mediterranean Sea: the Rhône River, the largest continental inflow to the Mediterranean Sea, and the Têt River, a small typical Mediterranean river.

2 Materials and Method

2.1 Sea Surface Sampling

Between May and November 2016, 13 samples were taken at different seasons in the Rhône area. Two transects were performed at the sea surface close to the river mouth. During the same period, a total of 18 samples were obtained in the Têt area. A triangle of three transects close to the river mouth was performed every month (Fig. 1).

Transects were carried out using a manta trawl (60 × 25 cm opening; 333 µm mesh) towed at the top 20 cm of the sea surface at 2 knots for 30 min–1 h. Sampled water volumes were recorded with a mechanical flowmeter (Hydro-bios; model 438 110). Once on board, the trawl was rinsed, the content of the collector was emptied over a 200 µm metal sieve and then transferred to glass bottles.

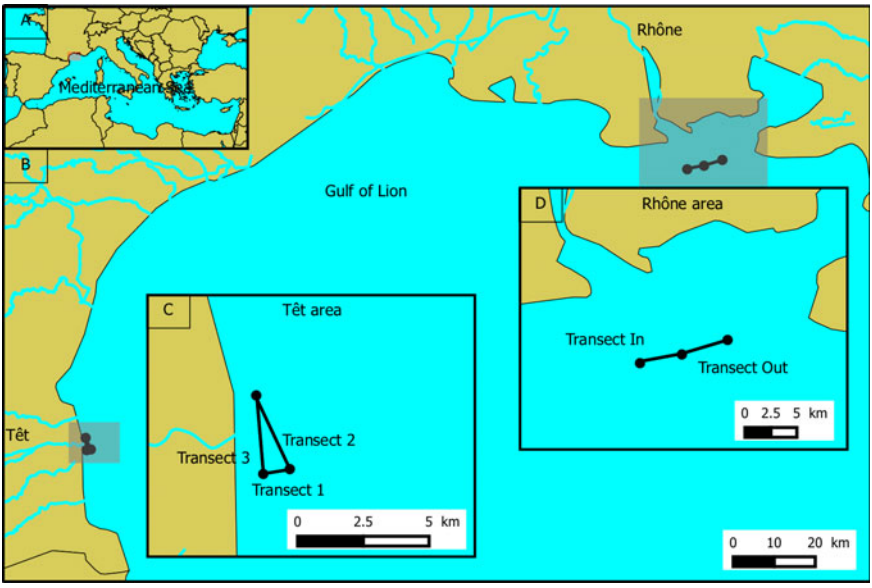

Fig. 1 Map of sampling sites. **A**: Mediterranean Sea; **B**: Gulf of Lion; **C**: Têt area; **D**: Rhône area. Grey rectangles represent zoom box **B**, **C** and **D**

2.2 Laboratory Work

Contamination of the samples was minimized by wearing laboratory cotton coats and using equipment made of glass or steel. Every step with an air exposure was performed under a laminar flow cabinet and beakers were covered by an aluminum foil.

2.2.1 Preparation of Samples

Collected materials were transferred into beakers and biological debris was removed using hydrogen peroxide H_2O_2 (modified from [11]). Two mL of H_2O_2 40% were added repeatedly until most of the organic matter was digested. During the entire digestion process, beakers were heated at 50 °C on a heating plate. The resulting solutions were filtered on paper filters (Whatman). Filters were examined under a Wild Heerbrugg dissecting stereomicroscope (25 × and 50 × magnifications). MPs were separated into four categories: fibers (including filaments and fishing lines), fragments (3-D pieces of plastic, including spherules), films (2-D pieces of plastic), and foams (pieces or spherules with a spongy, soft structure).

2.2.2 Polymer Identification

Forty fibers and 80 other items of each category were randomly selected and analyzed for polymer nature. Fibers were analyzed with an FTLA2000 FTIR spectrometer (ABB). Other particles were analyzed with a Frontier FTIR spectrometer (PerkinElmer). Following background scans, 100 scans per particle were performed. For each particle, scans were performed between 4000 and 700 cm^{-1}. Essential FTIR trial version software was used for the output spectra and identification of polymers was performed by comparison with a self-collected spectrum database.

2.3 Data Analysis

FTIR analyses allowed to estimate the number of plastic items collected. For each category, a "plastic" ratio was applied to the number of collected particles. This ratio results from the division of the number of particles which have a "plastic spectra" with the total number of particles analyzed. Finally, concentrations of MPs were estimated by dividing the adjusted numbers of plastic particles by the sampled water volumes.

3 Results and Discussion

3.1 Results

Adjusted concentrations ranged from 0.05 to 0.45 items.m^{-3} for the Têt area with an average of 0.18 items.m^{-3} (Fig. 2a) and from 0.08 to 0.41 items.m^{-3} with an average of 0.19 items.m^{-3} for the Rhône area (Fig. 2b), which is not significantly different (t-test; p-value = 0.96).

Concentrations inside the Têt area were most of the time higher at transect 3 (Fig. 2c), but differences between the three transects are not significant (ANOVA; p-value = 0.168). Concentrations for each transect have changed highly during the

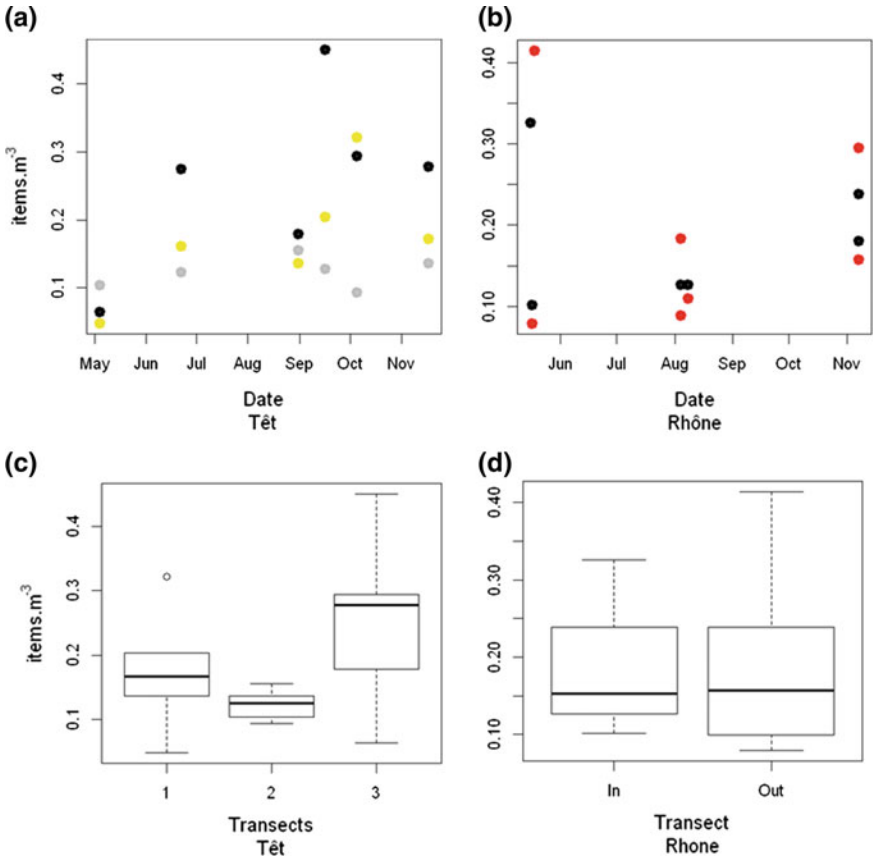

Fig. 2 Concentration of MPs (items.m^{-3}) during the year 2016 **a** in the Têt area (yellow dots: transect 1; grey dots: transect 2; black dots: transect 3) and **b** in the Rhône area (black dots: transect In, red dots: transect Out); Boxplot of MPs concentration according to transects **c** in the Têt area and **d** in the Rhône area. "In": in the Rhône plume, "out": upstream the Rhône plume

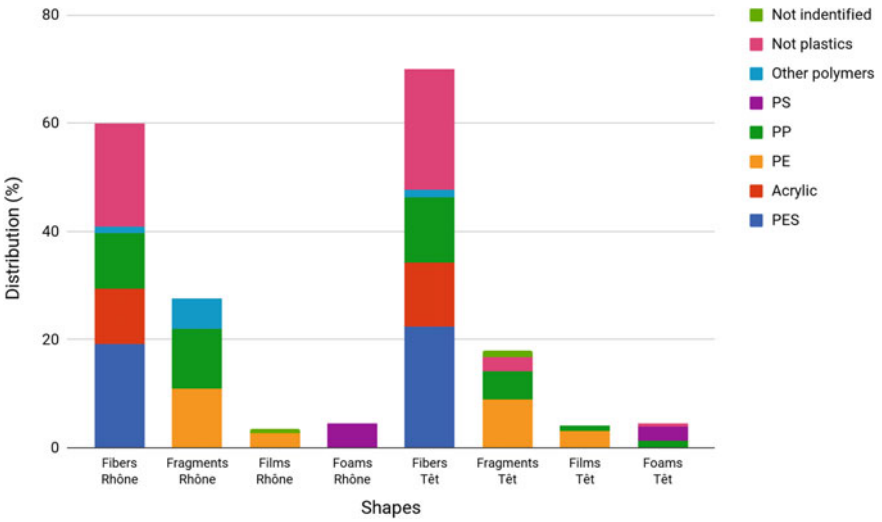

Fig. 3 Distribution of shape categories and corresponding polymer nature

year but combined concentrations did not significantly differ between months (ANOVA; p-value = 0.293).

Similarly, concentrations inside the Rhône area were not significantly different between the two transects (Fig. 2d; t-test; p-value = 0.91) or between the three different sampling periods (ANOVA; p-value = 0.274). Nevertheless, concentrations could occasionally change by a factor of 5 between two consecutive days at the same location (e.g., May samples), and by a factor of 3 between two consecutive trawls on the same day (e.g., August samples).

The Rhône and the Têt areas had similar categorical distributions (Fig. 3). Fibers were the most abundant shape (60–70%), followed by fragments (18–28%). Foams and films were less represented (3–5%). FTIR analysis indicates that fibers were polyester (32%), cotton (32%), PP (17%), acrylic (17%), and PA (2%). Fragments and films were mostly PE (54%) and PP (17%), while foams were essentially made of PS (67%).

3.2 Discussion

The Rhône River is the largest source of freshwater and sediments into the Mediterranean Sea while the Têt River is a typical small Mediterranean coastal river, with extreme low-water stages and occasional massive flood events. MPs concentrations in the downstream parts of both rivers ranged between 1 and 2 items.m^{-3} (Constant, unpublished results), thus, about 10 times more than off

both mouths. This strong decrease in concentration has been mainly attributed to the oceanic dilution [12].

The Rhône River has an average water flow 200 times higher than the Têt River. The total amount of MPs discharged by the Rhône River, so far not precisely calculated, should be, due to the close riverine concentrations, much higher than those released by the Têt River. One could have thus expected surface concentration differences more in connection with the large differences in inputs rather than the observed similarities between both areas. These preliminary results suggest that the influence of rivers on the spatial distribution of MPs rapidly decreases with distance to the river mouths, a hypothesis supported by the fact that highest concentrations off the Têt River were observed in the transect closest to the river mouth. Alternately, the similarities in offshore MP concentrations could also indicate that the floating plastics collected within our experimental areas are not provided by straight, direct river inputs. Some mixing and dispersion processes must rapidly take place once the MPs enter the marine environment.

Concentrations estimated in this study are relatively close to previous observations from the Northwestern Mediterranean (0.1–0.3 items.m^{-3} [13–15]) or in the costal water of the East China Sea (0.2 items.m^{-3} [12]), but 45 times lower than concentrations observed along the Israeli coast (7 items.m^{-3} [16]) or in the Southern California coastal waters (7.3 items.m^{-3} [17]). Such differences may have numerous reasons ranging from hydrodynamic features to the use of different trawls and methodologies. Indeed, most of the old investigations did not check the polymeric nature of their collected particles and especially fibers, probably leading to overestimations.

No spatial or temporal trends were found but high changes at daily and kilometer scale were observed. Presence of lint composed of hundreds of fibers can partly explain the high differences observed at small scales, as well as fast-changing river inputs.

Strong spatial heterogeneities of floating MPs have been previously reported at regional scales, up to a factor of 40 [14] and 30 at subregional scales [13]. Van der Hal et al. [16] indicated a factor of 300 at subregional and seasonal scales. Collignon et al. [18] investigated fluctuations of a costal transect on a semi-monthly basis and observed differences up to a factor of 60 during the same week. Wind had been found to be a factor of heterogeneity [15]. Large hydrological structures, like central oceanic gyres, accumulate MPs [2, 10] but influence of meso and small structures, like eddies and fronts, remain unknown.

4 Conclusions

This study confirms that heterogeneities of floating MPs can act at small scales. These extended observations in the NW Mediterranean coastal environment underline the necessity of performing replicate sampling to get a better insight into the spatial and temporal distribution patterns of these worrying pollutants.

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Microplastic Abundance and Polymer Types in a Mediterranean Environment

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1 Introduction

Microplastics have become a more and more dominant threat to marine ecosystems. The ubiquity of microplastics is one of the major problems; from the sea surface and water column to the beach and seabed sediment or even ingested by marine organisms, small plastic particles have been found. This study is focused on monitoring and assessment of microplastic pollution (plastic particles <5 mm) on the sea surface, in beach sediments, and in marine biota in the Corfu Island area (Northern Ionian Sea). Corfu is one of the main touristic destinations in Greece, and thus tourism together with maritime traffic is the main source of litter on the beaches as well as in the marine environment. This study analyzes microplastic pollution of the region by determining microplastic abundance, types, and polymer composition in three environmental compartments (sea surface, beach sediment, and marine biota).

2 Materials and Methods

2.1 Study Area and Sampling

Samples from water, sediment, and marine biota were collected from the Northern Ionian Sea. All sampling was conducted in 2014 and 2015 according to [1]. More precisely, sea surface samples were collected during April 2014 and October 2015,

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