

Presence of Microplastic in Personal Care and Cosmetic Products from Markets in Punjab, Pakistan

Sajjad Hussain¹, Nuzhat Sial^{1*}, Zarqa Nawaz², Muhammad Naeem³, Rana Zeeshan Habib⁴, Thies Thiemann^{5*}

¹Department of Zoology, The Islamia University of Bahawalpur, Bahawalpur, Pakistan

²Department of Chemistry, The Women University Multan, Multan, Pakistan

³Department of Pharmacy, Shah Abdul Latif University, Khairpur, Pakistan

⁴Department of Biology, College of Science, United Arab Emirates University, Al Ain, United Arab Emirates

⁵Department of Chemistry, College of Science, United Arab Emirates University, Al Ain, United Arab Emirates

Email: *nztial@gmail.com, *thiesthiemann@yahoo.de

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Abstract

In order to assess microplastic content in personal care products in Pakistan, 103 body scrubs and face washes were randomly selected in August-October 2021 from different markets in Jhang, Multan, and Bahawalpur in Punjab, Pakistan. It was found that 47 (45.6%) products incorporated plastic microbeads, including 44 (42.7%) products that exhibited polythene microbeads. Overall, a higher proportion of imported products exhibited plastic microbead content than locally manufactured products.

Keywords

Rinse-Off Cosmetics, Body Scrubs, Plastic Microbeads, Microbeads, Plastic Pollution

1. Introduction

Microplastics (MPs) have become a pervasive environmental concern globally due to their widespread presence in water bodies and their potential to enter the human food chain. In 2021, global plastic production amounted to 390.7 million tons [1] [2], an increase of 4% over the preceding year. A significant amount of the plastic reaches the environment, especially at the end of life or due to spillage during production and transport. It is estimated that 10% of the plastics produced worldwide enter the oceans, thereby contributing 80%~85% of the marine litter [3]-[5] and >90% of the floating debris [6], constituting between 2.1×10^{23} and 1.7

$\times 10^{24}$ plastic particles [7] [8]. The fate of single-use plastics is especially worrisome. Its production, use and fate at end of life are governed by reglementary laws in a number of regions. Historically, there have been products whose plastic constituents would automatically be released into the environment upon use. Typical such products are plastic abrasives for blast cleaning surfaces and microplastic (MP) containing personal care products [9]-[12]. In both instances, the plastic particles are less than 5 mm in size, hence microplastics, and “soft” enough not to damage the surface to be cleaned, may it be the skin, tooth enamel, or the hull of a ship, and in both instances the particles belong to the class of primary MPs. Primary MPs as opposed to secondary MPs are plastic particles that are purposefully produced in their small size for certain applications. Secondary MPs, on the other hand, derive from the degradation of larger plastic pieces, such as meso- and macroplastics. In the last decade, a number of countries (Figure 1) started to severely reduce the use of MPs in rinse-off cosmetics that include toothpastes and body scrubs. These efforts range from outright banning MPs in rinse-off cosmetics to governmental recommendations [13]. The first culmination of this effort was the Microbead-Free Waters Act of 2015 [14] [15], which was passed by the 114th Congress of the United States and which prohibits the sale and distribution of rinse-off cosmetics containing plastic microbeads. Bans on microbeads in rinse-off cosmetics were also put in place in Sweden (2018), UK (2018), Italy (2019), France (2017), the Netherlands (2014), India (2020), Thailand (2017), South Korea (2017), Taiwan (2018), Canada (2018) and New Zealand (2017). In addition, in September 2018, in an effort to restrict the manufacture and sales of products with intentionally added MPs, the European Parliament called on the Commission to implement a MP ban in cosmetics, personal care products, detergents and cleaning products in all European Community (EC) member states by 2020, with the idea that the manufacturers of cosmetics operating in the EC would have phased out MPs voluntarily at that time. The adoption of this ban has taken more time than expected but has been voted on positively by the EC member states and as amending Annex XVII to Regulation (EC) No 1907/2006 of the European Parliament and of the Council concerning the Registration, Evaluation, Authorisation and Restriction of Chemicals (REACH) as regards synthetic polymer microparticles is currently scrutinized by the Council and European Parliament [16] [17]. Many of the industrialized countries such as Australia focused on a voluntary industrial phase-out of plastic microbeads found in rinse-off personal care and cosmetic products [18]. In Australia, the voluntary phase-out was led by Accord Australasia (Accord), and a recent study has shown that of 8100 unique stock keeping units of rinse-off products scrutinized at different shops in the country, only 58 (0.7%) exhibited plastic microbeads [19], most of which were facial and body scrub products.

Some of us pursued a study on MP content of body scrubs sold in the United Arab Emirates in 2018 - 2020 [20] [21], and it was shown that gratifyingly only a small percentage of products contained MPs, the share of which even declined

during the time of the study, and this in a country that had not signed off on legislation banning or restricting plastic microbeads in rinse-off cosmetics. These studies were augmented by a survey on MPs in toothpastes available in UAE [22], in addition to toothpastes bought in Syria. None of them showed any MPs. The results seemed to indicate that MPs were being phased out in cosmetics, including in toothpastes, generally, even in regions where no ban on microplastics had been announced. Similarly, O'Farell and Harvey [19] reported that none of the toothpastes analyzed in Australia showed MPs, and Lei *et al.* found no plastic microbeads in the 135 toothpastes from 23 brands they studied in China [23]. Disparagingly, however, from some parts of Asia such as India and China came other reports of continuing significant use and release of MP into the environment of MPs from cosmetic products [24] [25], including from toothpastes [26] which seemed to indicate a more complicated situation worldwide. Therefore, when the opportunity presented itself to undertake a study on MP content of cosmetic products in Pakistan, the authors were happy to seize it.

In recent times, a number of studies have appeared that analyzed for MP concentrations in different environmental compartments in Pakistan, predominately in aquatic environments such as in nearshore waters [27], and along the coastline of the Arabian Sea [27]–[29], but also in relatively secluded lakes such as the Mahodand Lake, Swat, Khyber Pakhtunkhwa District [30] and Rawal Lake, Margalla Hills, Islamabad Capital territory [31] [32]. MP concentrations were found to be significant. Also, the studies made clear that MPs are taken up by marine organisms including fish such as the Indian mackerel (*Rastrelliger kanagurta*) [28] and the Crescent grunter (*Terapon jarbua*) [29] that are used for local consumption, giving the chance for MPs to penetrate the human foodweb [33] [34]. However, little is yet known about the contribution of different sources to MPs in the Pakistani environment. As plastic microbeads from personal care products have always been seen as contributors of primary MPs to the environment, this study has also been undertaken to understand to what degree cosmetic products contribute to the overall MP contamination in the environment. For this reason, 103 different rinse-off cosmetics were bought from markets in Punjab, Pakistan and analyzed for microplastic beads. The following is the account of this study.

2. Materials and Methods

103 cosmetic products, including scrubs and personal care and cosmetic products (PCCPs), were selected randomly from various local markets located in Jhang, Multan, and Bahawalpur in Punjab, Pakistan. The products were purchased during the months of August, September, and October 2021. The products were purchased without any attention to cost or to the country of manufacture of the products. 30 facial scrubs were purchased from Jhang, 42 were purchased from Multan and 31 scrub samples were bought from Bahawalpur. The products were of different brands, made by diverse manufacturing companies and had diverse countries of origin.

Solid content from the cosmetic products was obtained by addition of 10 g of the selected cleanser to 200 mL of water at 50°C. The resulting mixture was stirred for 10 min. Thereafter, the mixture was filtered through a cotton cloth. This procedure was performed in triplicate for each product. Then, the acquired filtrate was put through an additional round of filtration using a Whatman filter paper (ashless, grade 1001-070, pore size 11 µm) to ensure that all potential microparticles had been removed. The particulates were dried in a Vacuum Oven DZF-6090 drying cabinet at a temperature of 40°C for 10h. Before filtration, the seven products SH3, SH11, SH16, SH20, SH29, and SH96 were discovered to have a significant of non-soluble organic components. These samples were subjected to treatment with Fenton reagent ($\text{FeSO}_4 + \text{H}_2\text{O}_2$) in order to decompose the organic matter. There were also organic solids in the samples SH36, SH48, SH85 and SH92, which could be removed with the organic solvents ethyl acetate and chloroform.

It was necessary to use a microscope (Olympus microscope BX51M) in order to manually separate the particulates in some of the tested products due to the existence of more than one kind of microplastic. In SH36, SH48, and SH85, there were microbeads present that had a variety of colors, including blue, pink, yellow, and white. These microbeads were separated through the process of floatation using a variety of differently dense organic solvents, such as chloroform (CHCl_3 , $d = 1.40 \text{ g/mL}$) and ethanol ($\text{C}_2\text{H}_5\text{OH}$, $d = 0.789 \text{ g/mL}$).

In the case of digesting products with Fenton's reagent, 5 g of the product sample was treated with a mixture of 5 mL of a solution containing hydrogen peroxide (H_2O_2) and 2.5 mL of a solution containing Fenton's catalyst. The solution was pH 5. To prepare Fenton's catalyst, 20 g of iron (II) sulphate heptahydrate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$) were dissolved in 1L distilled water [35]. A reaction between the sample and the solution was allowed to proceed at 25°C for approximately 15 min. After this, 10 mL of water were added to the solution, and the resulting suspension was filtered using a Whatman filter paper (ash less, grade 1001-070, 11 µm pore size). A final washing of the filtrate was carried out with 315 mL distilled water. The filter paper with the filtered solid was then dried in an oven (Vacuum Oven DZF-6090) at 40°C.

The filtered, washed and dried microbeads were analyzed for, size, shape and total count. An electric balance type ABT 220-5DM (detection limit: 0.01 mg) was used for weighing, whereas microphotos of the beads were taken with an Olympus microscope BX51M. Three replicates from each product were used for the photos and quantification procedures. For characterization and Feret's diameter of the microbeads, the photos were analyzed by Fiji Image J software [36] [37]. The Feret's diameter represent the size of microbeads. The Feret's diameter corresponds to the longest distance between two points along the microbeads boundary [38].

Fourier Transform Infrared (FT-IR) spectroscopy (model Bruker ALPHA platinum) was used to determine the composition of extracted microbeads for each product. The obtained spectra were matched with OMNIC 9 software to identify

the type of polymer.

To obtain an insight in the inorganic constituents, the filtered solids of the cosmetic products were subjected to ashing, where samples were weighed in crucibles of 40 ml volume and were heated in a Vacuum Oven DZF-6090n for 1.5 h at 550°C. The amount of ash that was left behind and which constitutes the inorganic content of the microbeads is shown in the text as w%.

During isolation and analysis of the product samples, suitable contamination control measures took place to prevent microplastic and fiber contamination. White cotton laboratory coats were worn, and single-use latex gloves were used throughout the study. The number of persons in the laboratory was kept to a minimum, and the laboratory door was closed at all times. Surfaces were cleaned carefully prior to use. Randomly, filter paper was scrutinized under the microscope for any contamination before use.

3. Results

Of the products sampled for this study, 26 products had been manufactured locally by Pakistani companies, 34 samples had been produced in India, 8 products in the United Arab Emirates, 12 products in Thailand, 11 products in China, 2 products in South Korea, 5 products in the United States of America, and 5 products were made by companies in France (**Figure 1**).

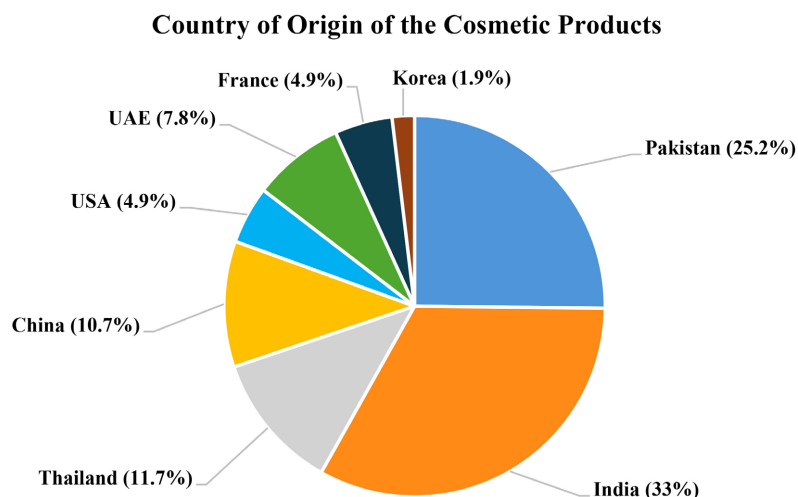


Figure 1. Country of origin of the cosmetic products investigated in this study.

From the evaluation of the solid content of the cosmetic products under study, it could be seen that out of 103 products, 27 products (26.2%) did not contain any microbeads at all, 20 products (19.4%) contained walnut shells as abrasive, 3 products (2.9%) contained hydrated silica as the only solid abrasive, and 6 products (5.9%) exhibited microcrystalline cellulose as the sole solid abrasive (**Table 1, Figure 2**). 10 other products (9.7%) incorporated microcrystalline cellulose in their formulation, but then invariably in combination with polythene microbeads. It was found that 47 (45.6%) products incorporated plastic microbeads, including

44 (42.7%) products that exhibited polythene microbeads. 10 (9.7%) of the products which revealed polythene had microcrystalline cellulose as a co-constituent.

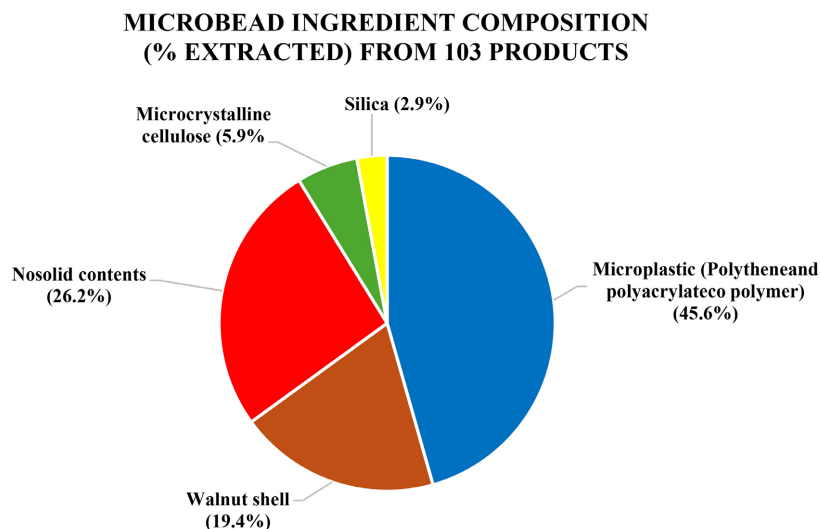


Figure 2. Composition of solids of the 103 cosmetic products sampled for the study.

Table 1. Country of origin, color, shape and polymer compositions collected from 103 facial scrubs purchased from different markets of Punjab, Pakistan.

Sample	Country	Color	Shape	Polymer (MBs) composition by FTIR
SH1	Pakistan	-	-	no solid particles were present
SH2	Pakistan	golden	irregular	walnut shells
SH3	United Arab Emirates	brown	irregular	walnut shells
SH4	India	-	-	no solid particles were present
SH5	USA	colorless	irregular	polyethylene
SH6	India	red	irregular	polyethylene
SH7	India	white	spherical	microcrystalline cellulose
SH8	Pakistan	-	-	no solid particles were present
SH9	Pakistan	brown	irregular	walnut shells
SH10	Pakistan	yellow, green	spherical, granular	polyethylene
SH11	India	white, red	irregular	polyethylene
SH12	Thailand	white	granular	polyethylene
SH13	India	-	-	no solid particles were present
SH14	India	-	-	no solid particles were present
SH15	India	-	-	no solid particles were present
SH16	Korea	blue	irregular	polyethylene, microcrystalline cellulose
SH17	USA	-	-	no solid particles were present
SH18	Pakistan	-	—	no solid particles were present
SH19	Korea	-	—	no solid particles were present

Continued

SH20	India	red	spherical	ethylene acrylate co-polymer
SH21	UAE	-	-	no solid particles were present
SH22	India	-	-	no solid particles were present
SH23	Pakistan	-	-	no solid particles were present
SH24	UAE	-	-	no solid particles were present
SH25	China	-	-	no solid particles were present
SH26	India	-	-	no solid particles were present
SH27	India	-	-	no solid particles were present
SH28	Pakistan	-	-	no solid particles were present
SH29	India	colorless	spherical	walnut shells
SH30	India	-	-	no solid particles were present
SH31	India	-	-	no solid particles were present
SH32	Pakistan	brown	irregular	walnut shells
SH33	United Arab Emirates	red, blue	granular	polyethylene
SH34	Pakistan	yellow	irregular	polyethylene
SH35	India	brown	irregular	walnut shells
SH36	India	blue, white	irregular	polyethylene
SH37	India	blue	spherical	polyethylene
SH38	France	colorless	irregular	polyethylene
SH39	India	white	spherical	hydroxylated silica gel
SH40	Pakistan	-	-	no solid particles were present
SH41	Pakistan	blue, yellow	irregular	polyethylene
SH42	India	red	irregular	polyethylene, microcrystalline cellulose
SH43	India	brown	granular	ethylene acrylate co-polymer
SH44	Pakistan	brown	irregular	polyethylene
SH45	Pakistan	-	-	no solid particles were present
SH46	Thailand	blue	irregular	polyethylene
SH47	United Arab Emirates	red	irregular	polyethylene
SH48	Pakistan	brown, blue	irregular	polyethylene
SH49	India	-	-	no solid particles were present
SH50	India	red, blue	irregular	polyethylene
SH51	China	white	irregular	polyethylene
SH52	Pakistan	red	irregular	polyethylene
SH53	India	-	-	no solid particles were present
SH54	USA	brown	irregular	polyethylene, microcrystalline cellulose
SH55	India	blue	irregular	ethylene acrylate co-polymer
SH56	Thailand	white	Irregular	polyethylene

Continued

SH57	India	-	-	no solid particles were present
SH58	United Arab Emirates	red	irregular	polyethylene
SH59	Pakistan	red, blue	irregular	polyethylene, microcrystalline cellulose
SH60	Thailand	brown	irregular	walnut shells
SH61	India	-	-	no solid particles were present
SH62	China	golden	irregular	walnut shells
SH63	India	colorless	irregular	polyethylene, microcrystalline cellulose
SH64	Thailand	blue	irregular	polyethylene
SH65	China	red	irregular	walnut shells
SH66	Thailand	brown	Irregular	walnut shells
SH67	United Arab Emirates	golden	irregular	polyethylene, microcrystalline cellulose
SH68	Pakistan	brown	irregular	walnut shells
SH69	India	blue	irregular	polyethylene
SH70	USA	brown	spherical, granular	polyethylene, microcrystalline cellulose
SH71	Thailand	red, blue	irregular	walnut shells
SH72	China	red	irregular	walnut shells
SH73	France	red	spherical, granular	microcrystalline cellulose, polyethylene
SH74	Pakistan	blue	irregular	polyethylene
SH75	France	white	irregular	microcrystalline cellulose, polyethylene
SH76	India	red, blue	spherical, granular	polyethylene
SH77	Pakistan	brown	irregular	hydroxylated silica gel
SH78	China	golden	irregular	walnut shells
SH79	USA	red, blue	spherical, granular	microcrystalline cellulose, polyethylene
SH80	Thailand	red	irregular	walnut shells
SH81	United Arab Emirates	white	irregular	polyethylene
SH82	India	-	-	no solid particles were present
SH83	Pakistan	brown	irregular	walnut shells
SH84	China	colorless	irregular	polyethylene
SH85	Thailand	blue, white	spherical, granular	polyethylene
SH86	France	red	irregular	ethylene acrylate co-polymer
SH87	France	red, blue	irregular	polyethylene
SH88	India	white	irregular	microcrystalline cellulose
SH89	Thailand	golden	irregular	walnut shells
SH90	China	white	irregular	microcrystalline cellulose
SH91	Pakistan	blue	spherical, granular	microcrystalline cellulose
SH92	China	white	irregular	polyethylene
SH93	India	red	irregular	walnut shells

Continued

SH94	Pakistan	brown	irregular	walnut shells
SH95	Pakistan	colorless	irregular	polyethylene
SH96	Pakistan	blue	spherical, granular	polyethylene
SH97	Pakistan	-	-	no solid particles were present
SH98	China	brown	irregular	walnut shells
SH99	Thailand	white	irregular	microcrystalline cellulose
SH100	India	brown	irregular	hydroxylated silica gel
SH101	China	red	irregular	polyethylene
SH102	Thailand	Blue	irregular	polyethylene
SH103	India	Colorless	irregular	microcrystalline cellulose

Figure 3 shows a typical example of an IR spectrum depicting polythene. The spectrum was obtained from the solids of product SH-12. The characteristic absorptions are 2915, 2847, 1472, 1465, 730, 717 cm^{-1} . A peak at around 2915 cm^{-1} corresponds to the asymmetric stretching of C-H bonds in CH_2 groups, the peak around 2847 cm^{-1} corresponds to the symmetric stretching of C-H bonds in CH_2 groups, the two peaks at 1472 cm^{-1} and 1465 cm^{-1} correspond to the bending deformation of the polymer backbone, the peaks at 730 cm^{-1} and 720 cm^{-1} correspond to the rocking deformation of the CH_2 groups [39].

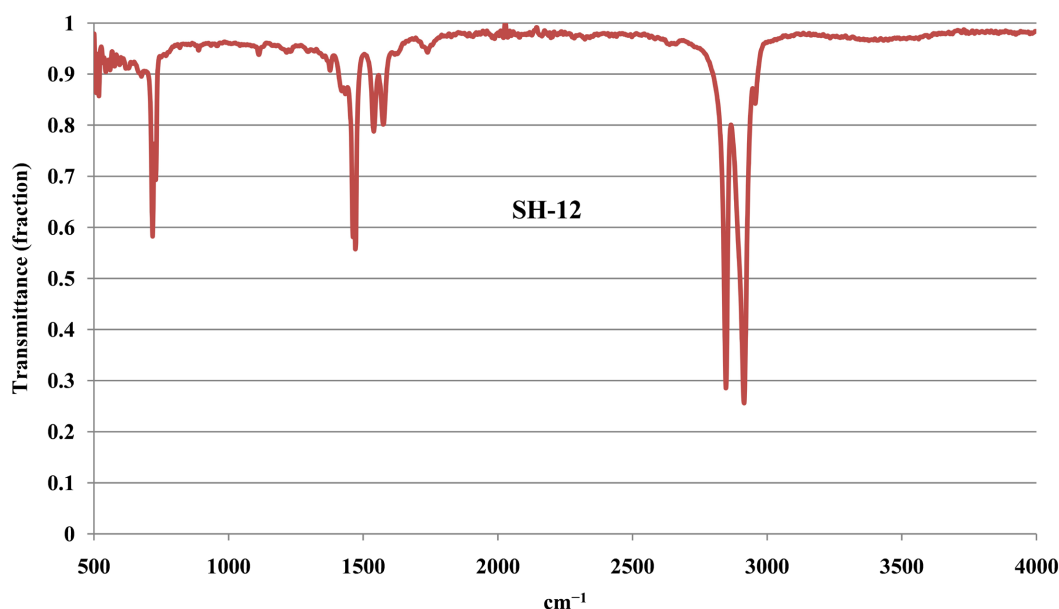


Figure 3. FT-IR spectrum of polythene containing microbead of product SH-12.

Ferret diameter size range (μm) graphs of the microbeads collected from 76 products are listed below, in **Table 2** and in **Figure S1** (Suppl. Data) for products SH-2 to SH-103. The size distribution graphs were plotted to compare and analyze the size range of microbeads existing in the 76 products. The filter paper used in

the filtration process gives a size limit of about 12 μm for microbeads that could be retained in this experiment. The average size of 3 samples was used to plot the distribution graphs for each product on an excel sheet by using the pivot graph option. To facilitate a comparison of the microbead size distribution of different products, the start and end values for the presentation of the distribution were fixed at 1 and 350 μm , respectively, with a difference of 50 μm per bin for all 76 products.

The distribution pattern of 76 products revealed that 29 products (SH-2, SH-3, SH-4, SH-7, SH-10, SH-12, SH-16, SH-20, SH-32, SH-42, SH-60, SH-69, SH-70, SH-71, SH-73, SH-85, SH-86, SH-87, SH-90, SH-91, SH-93, SH-94, SH-95, SH-96, SH-98, SH-99, SH-100, SH-101 and SH-103) have a non-uniform size distribution, 4 products (SH-11, SH-43, SH-76 and SH-92) have a left-skewed size distribution, 43 products (SH-6, SH-9, SH-29, SH-33, SH-34, SH-35, SH-36, SH-37, SH-38, SH-39, SH-41, SH-44, SH-46, SH-47, SH-48, SH-50, SH-51, SH-52, SH-54, SH-55, SH-56, SH-58, SH-59, SH-62, SH-63, SH-64, SH-65, SH-66, SH-67, SH-68, SH-72, SH-74, SH-75, SH-77, SH-78, SH-79, SH-80, SH-81, SH-83, SH-84, SH-88, SH-89 and SH-102) have a right-skewed size distribution.

In this study, the microbead size range of 46 samples was represented by the size bin 1 μm - 50 μm . The smallest size bead, however was found to be 12.7 μm . This can also be seen as the lower size range of MPs that could still be retained in the filter paper. The upper size range of 2 samples was represented by the size bin ending at 300 μm , and in 2 samples were beads with a size slightly over 300 μm . Overall, the majority of the analyzed cosmetic products were found to have the majority of their beads with a diameter size ranging from 12.7 μm to 200 μm .

Table 2. Size, quantity and ash contents of the MBs collected from the 76 personal care products (facial scrubs) that contained solid content, purchased from different markets and shopping malls of Punjab, Pakistan.

Sample	Size range of the microbeads (μm)	Average size of microbeads (μm)	Microbeads weight in g per 10 g of product	Weight (%) of ash obtained from the particles
SH2	53.6 - 201.3	78.7 $\pm$ 19.6	0.301 $\pm$ 0.019	3.1
SH3	76.2 - 256.4	89.3 $\pm$ 15.9	0.210 $\pm$ 0.031	4.9
SH5	65.7 - 234.5	76.4 $\pm$ 17.1	0.310 $\pm$ 0.032	0.9
SH6	101.6 - 298.7	115.3 $\pm$ 20.4	0.192 $\pm$ 0.045	4.3
SH7	98.9 - 167.8	101.3 $\pm$ 29.7	0.261 $\pm$ 0.032	6.9
SH9	132.3 - 224.9	160.6 $\pm$ 13.9	0.265 $\pm$ 0.035	8.4
SH10	87.1 - 206.4	95.8 $\pm$ 11.6	0.124 $\pm$ 0.044	32.6
SH11	57.0 - 110.9	78.3 $\pm$ 23.9	0.217 $\pm$ 0.044	4.9
SH12	25.2 - 196.8	95.3 $\pm$ 19.3	0.253 $\pm$ 0.043	34.1
SH16	145.7 - 263.9	168.3 $\pm$ 25.3	0.264 $\pm$ 0.023	0.39
SH20	98.6 - 153.9	115.4 $\pm$ 17.4	0.123 $\pm$ 0.024	<5.0
SH29	54.4 - 198.5	69.3 $\pm$ 12.4	0.272 $\pm$ 0.027	2.9
SH32	98.2 - 156.9	115.9 $\pm$ 18.4	0.213 $\pm$ 0.026	3.7

Continued

SH33	165.1 - 203.4	187.3 ± 36.4	0.265 ± 0.036	0.95
SH34	69.9 - 208.2	89.4 ± 23.0	0.213 ± 0.034	6.9
SH35	72.8 - 300.6	98.9 ± 15.3	0.251 ± 0.014	4.6
SH36	89.9 - 156.7	110.8 ± 17.4	0.127 ± 0.066	9.1
SH37	35.8 - 199.2	74.7 ± 16.4	0.112 ± 0.026	3.0
SH38	143.7 - 205.9	178.4 ± 36.5	0.212 ± 0.026	13.9
SH39	46.0 - 109.6	93.4 ± 23.3	0.321 ± 0.086	3.9
SH41	98.5 - 165.9	142 ± 27.0	0.219 ± 0.058	5.2
SH42	96.2 - 176.8	110.0 ± 15.8	0.193 ± 0.017	0.58
SH43	53.8 - 145.9	74.9 ± 16.3	0.201 ± 0.053	6.2
SH44	24.6 - 90.7	78.4 ± 12.4	0.194 ± 0.074	3.9
SH46	25.8 - 154.8	78.3 ± 14.3	0.212 ± 0.035	<6.0
SH47	21.9 - 97.4	85.8 ± 12.3	0.192 ± 0.084	9.3
SH48	16.8 - 183.6	98.3 ± 14.3	0.182 ± 0.026	6.5
SH50	43.1 - 97.9	83.5 ± 21.1	0.113 ± 0.024	7.0
SH51	12.9 - 165.7	99.4 ± 23.4	0.115 ± 0.075	3.9
SH52	24.6 - 97.4	79.0 ± 17.3	0.132 ± 0.040	5.2
SH54	32.1 - 94.6	76.9 ± 14.9	0.134 ± 0.016	9.1
SH55	53.0 - 197.5	110.3 ± 23.9	0.123 ± 0.054	1.9
SH56	34.6 - 87.9	58.3 ± 11.2	0.325 ± 0.028	3.5
SH58	25.7 - 79.5	69.4 ± 17.3	0.371 ± 0.064	7.8
SH59	25.6 - 96.7	75.9 ± 21.4	0.283 ± 0.054	2.8
SH60	23.7 - 106.5	69.4 ± 12.1	0.294 ± 0.086	5.1
SH62	25.1 - 98.7	76.3 ± 11.5	0.220 ± 0.013	7.0
SH63	32.7 - 143.9	83.5 ± 31.7	0.193 ± 0.037	4.3
SH64	12.7 - 163.9	73.9 ± 12.6	0.195 ± 0.054	6.0
SH65	23.4 - 153.7	87.4 ± 17.3	0.372 ± 0.074	3.2
SH66	38.9 - 90.1	73.9 ± 18.3	0.281 ± 0.065	4.9
SH67	21.7 - 142.5	80.4 ± 11.2	0.197 ± 0.069	3.6
SH68	21.4 - 96.6	76.4 ± 11.0	0.328 ± 0.036	5.1
SH69	43.6 - 151.5	73.9 ± 12.3	0.293 ± 0.064	9.1
SH70	32.5 - 206.7	74.9 ± 12.9	0.295 ± 0.076	1.9
SH71	43.8 - 143.8	98.3 ± 17.2	0.192 ± 0.013	3.5
SH72	22.3 - 108.9	68.9 ± 10.2	0.320 ± 0.038	7.8
SH73	43.7 - 176.7	98.3 ± 17.3	0.302 ± 0.054	2.8
SH74	32.4 - 98.9	63.7 ± 17.9	0.294 ± 0.028	5.1
SH75	23.5 - 124.7	76.5 ± 18.3	0.294 ± 0.038	7.0

Continued

SH76	43.1 - 97.7	78.9 ± 11.2	0.232 ± 0.083	4.3
SH77	23.6 - 142.7	87.7 ± 21.2	0.291 ± 0.064	6.0
SH78	19.4 - 156.3	69.4 ± 11.0	0.302 ± 0.028	3.2
SH79	26.4 - 197.1	65.5 ± 10.9	0.274 ± 0.097	4.9
SH80	24.9 - 203.4	95.5 ± 10.2	0.329 ± 0.009	3.6
SH81	29.5 - 175.3	98.3 ± 21.6	0.304 ± 0.056	9.1
SH83	46.8 - 206.9	114.5 ± 23.9	0.239 ± 0.062	1.9
SH84	17.8 - 153.5	85.4 ± 19.5	0.193 ± 0.024	3.5
SH85	27.3 - 145.3	87.9 ± 11.4	0.172 ± 0.038	7.8
SH86	34.6 - 106.8	79.4 ± 17.3	0.193 ± 0.062	2.8
SH87	43.2 - 242.8	118.3 ± 23.1	0.208 ± 0.075	5.1
SH88	36.0 - 94.5	67.3 ± 12.9	0.187 ± 0.056	7.0
SH89	15.8 - 103.6	58.9 ± 9.4	0.231 ± 0.054	4.3
SH90	37.2 - 231.2	132.5 ± 23.1	0.253 ± 0.073	6.0
SH91	32.4 - 102.8	75.3 ± 12.2	0.365 ± 0.028	3.2
SH92	36.0 - 94.5	65.8 ± 11.0	0.377 ± 0.064	4.9
SH93	15.8 - 103.6	58.3 ± 8.3	0.299 ± 0.054	3.6
SH94	37.2 - 231.2	101.7 ± 19.2	0.342 ± 0.053	5.1
SH95	32.4 - 102.8	76.3 ± 12.9	0.254 ± 0.037	9.1
SH96	35.3 - 147.0	83.6 ± 10.2	0.306 ± 0.054	1.9
SH98	48.2 - 156.9	130.2 ± 23.8	0.377 ± 0.054	3.5
SH99	48.1 - 203.4	136.2 ± 17.3	0.2908 ± 0.045	7.8
SH100	39.9 - 208.2	116.9 ± 21.2	0.297 ± 0.027	2.8
SH101	72.8 - 300.6	143.5 ± 32.8	0.328 ± 0.067	5.1
SH102	49.9 - 156.7	110.6 ± 12.9	0.303 ± 0.044	7.0
SH103	45.8 - 199.2	79.4 ± 19.3	0.295 ± 0.084	4.3

The most common colors used in the studied products of the plastic microbeads were blue (35%), red (29%) and white/colorless (25%). The rest of the products had yellow (5%), brown (2%), green (2%) and golden (2%) plastic microbeads (Suppl. **Figure S1**). Over all products with beads (Suppl. **Figure S1**), the color scheme was composed of red, blue, and colorless, equally contributing 21%, golden (6%), yellow (3%) and green (1%). The much larger share of brown is due to the use of walnut shell in the products as solid abrasive material. Interestingly, walnut shell has also been found to be dyed golden and red in some cosmetic products [21]. The predominance of the colors blue and colorless/white can be found in cosmetics from a number of other markets around the world. Often, blue microplastics make up by color the dominant share of microplastics found in the oceans [5] [40]-[42]. The large share of the color red is something that may be specific to the Pakistani market.

4. Discussion

Microbeads play a vital role in some cosmetics products. Their functions depend on the size, shape, and composition of the microbeads [43]. Mostly, they are used as exfoliants [34] [44] [45] in personal care and cosmetic products (PCCPs), where PCCPs can be body and facial scrubs, shower gels and toothpastes, but also other cosmetic products such as lip sticks. Oftentimes, the basic function of the microbeads is to produce a smoother skin by increasing the rate of keratinization through exfoliation [46]-[48]. The composition of abrasive material in solid form in PCCPs has changed over time, beginning with exclusively natural materials such as pumice (pumicite), diatomaceous earth, perlite and tripoli, as well as many plant derived solids, such as chick-peas, barley, almonds, and horseradish seeds, in addition to ash and salt in oral care products [49]. Although early patents for microbeads in personal care and cosmetic products began to appear in the late 1960s with the first patent to include micro-polymer content in cosmetics being awarded in 1965 [50], plastic microbeads did not find entry into rinse-off cosmetics and personal care products in a large scale until the early 1990s. Some of the advantages of plastic microbeads are their low cost, the ease of dyeing, the possibility of tailoring their size and size distribution and their relative softness so that the microbeads do not damage the skin or the enamel of teeth. In the last two decades, the most commonly used synthetic polymers found in plastic microbeads have been polyethylene (PE), polypropylene (PP), polyethylene terephthalate (PET), poly (methyl methacrylate) (PMMA), and nylon, whereas cellulose, pumice, cornmeal, apricot kernels, silica, and walnut husks have been the most common natural or biodegradable materials used in microbead formulations [51] [52].

Zitko and Hanlon [53] were one of the first researchers reporting on potential dangers of plastic microbeads in commercially available personal care products. They commented on skin cleansers that contained granulated polyethene, polypropylene and polystyrene, 40 - 200 mesh (75 - 420 μm) in size. A few years later, Gregory reported on finding solid plastic content in 3 facial scrubs and in 3 face cleansers, sold in New Zealand markets, with contents of 0.19 - 6.91 g plastic microbeads per 100 g of product [54]. Since then, the use of plastic microbeads in cosmetic products has been seen as an international problem as these tend to pollute the environment, once washed off into the wastewater.

MPs from PCCPs, including from cosmetics, often find their way directly into household drains and are transported to wastewater treatment plants (WWTPs) [55]. WWTPs cannot retain the MPs completely, and so WWTPs have been identified as one of the point sources of MP emissions [56] [57], including of plastic microbeads from cosmetics [58]-[60] to the marine environment. Also, plastic microbeads find their way into soil, especially in the case, where sewage sludge is used for agricultural purposes [61] [62]. It must be noted, however, that it is far from easy to pinpoint the source of a microplastic and thus associate a particular microplastic with a PCCP, once the microplastic is released [63] [64].

Table 3. Published studies on the presence of MPs in personal care products from around the world.

Sample type	% of products with plastic microbeads	Country/region	Reference
4 face cleansers	100%	New Zealand	Fendall & Sewell, 2009 [44]
5 body scrubs	100% (PE and PP)	Malaysia	Praveen <i>et al.</i> , 2018 [65]
5 toothpastes	20 %	Malaysia	Praveen <i>et al.</i> , 2018 [65]
9 body scrubs	100% (PE such as LDPE)	PR China	Cheung & Fok, 2017 [25]
68 facial skin care products	64.7% (63% PE)	Macao, PR China	Bashir <i>et al.</i> , 2021 [68]
31 body skin care products	29% (PE)	Macao, PR China	Bashir <i>et al.</i> , 2021 [68]
45 cosmetic products	100% (53.3% PE)	Macao, PR China	Bashir <i>et al.</i> , 2021 [68]
135 toothpastes/23 brands	0%	PR China	Lei <i>et al.</i> , 2017 [23]
126 facial cleansers/16 brands	7.1%	PR China	Lei <i>et al.</i> , 2017 [23]
136 shower gels/30 brands	2.2%	PR China	Lei <i>et al.</i> , 2017 [23]
33 toothpastes	0%	UAE/Syria	Elkashlan <i>et al.</i> , 2022 [22]
74 body scrubs	year 2020: 12% (PE)	UAE	Habib <i>et al.</i> , 2022 [21]
89 body scrubs	year 2019: 12% (PE)	UAE	Habib <i>et al.</i> , 2022 [21]
37 body scrubs	year 2018: 29.7% (PE)	UAE	Habib <i>et al.</i> , 2020 [20]
20 toothpastes	20%	Turkey	Ustabasi & Baysal, 2019 [66]
50 abrasive cosmetics	26%	Poland	Guzik <i>et al.</i> , 2023 [11]
130 body scrubs	55%	Poland	Piotrowska <i>et al.</i> , 2020 [67]
3 shower gels and 2 body sprays	MBs throughout, of unclear composition	Romania	Banica <i>et al.</i> , 2023 [10]
103 body scrubs	45.6% (42.7% PE)	Punjab, Pakistan	this study

Currently, the likelihood of a personal care or cosmetic product to carry plastic microbeads varies, depending on the region, where the products are being sold. In 2018, Praveena *et al.* published a study on 5 body-scrubs and 5 toothpastes, bought in Malaysia. 1 out of 5 toothpastes contained MPs in form of polythene microbeads, however, all 5 body-scrubs contained plastic microbeads made of either polythene or polypropylene [65]. In 2019, in their study of products available in Istanbul, Turkey, Ustabasi and Baysal found polythene containing MPs at contents of between 0.4 and 1 w% in 5 (20%) of the 20 toothpaste products they had analyzed [66]. In 2023, Guzik *et al.* looked at 50 abrasive cosmetic products of Polish manufacturers, and found 13 of the products to contain MPs, but none of them were made of polythene. 49 of the products contained abrasive particles of natural origin [11]. This was a considerable improvement when compared to a previous study from Poland where of 130 analyzed body scrub products, 72 (55%) contained MPs made of PE [67]. Habib *et al.* studied PPCPs in the United Arab Emirates (UAE) and found a substantial reduction of products carrying MPs, looking at the market in 2020 (12%) [20] as opposed to what the market was in 2018 (29.7%) [22].

With 45.6% of the products examined in the current study carrying plastic microbeads, the proportion of PCCPs with MPs in the Punjab region of Pakistan is significant compared to PCCPs in many other countries (Table 3). There was no correlation between the presence of microbeads in a product and the cost of the product. The average size of the microbeads found in the products is similar to sizes found in products sold in other regions [25] [68], with a relatively narrow size distribution, albeit at the lower side of the size ranges that have been reported elsewhere. This also holds true for the weight contribution of MPs in the products [25] [68], which ranged from 1.13 w% to 3.72 w%. Most plastic microbeads in the studied products were found to be made of PE, similar to what has been found in other studies [69] [70]. Overall, this leads to a notable plastic burden in the Pakistani wastewater, where any plastic particles not retained in treatment regimes would reach the environment. This is significant as not all areas in Pakistan are connected to adequate wastewater treatment facilities and a sizable proportion of the wastewater is channeled into natural drains [71] [72], which would impose a heavy plastic burden on the environment.

Of the 103 examined products, 77 products (74.8%) were imported from other countries. While 38% of the Pakistani products included MPs, 48% of the imported products did so (Suppl. Figure S1). Interestingly, 9 out of the 10 products imported from France and USA contained MPs, where both USA and France are countries which have a ban on MPs in rinse-off cosmetics in place. This is similar to what was noted to be true in UAE markets [20] [21], where MP-carrying PCCPs are imported that are manufactured by companies whose headquarters lie in regions where the use of MPs in rinse-off cosmetics is already banned.

Pakistan has issued a new directive [73] which bans the manufacture, storage, and sale of single-use plastic items, which includes single-use polythene bags. Industrial plastic wrapping is excluded from the ban. Rinse-off MP containing personal care and cosmetic products is not mentioned in the ban. Recently, the Pakistan Chemical Manufacturing Association (PCMA) [74] has developed a webpage on MPs to further the Pakistani public's awareness of issues surrounding the use of MP-containing products.

Much has been made of the fact that in order to reduce plastics emissions into the environment, especially into the marine environment [75], international efforts would be needed [76] [77]. In addition, the voluntary phase-out of MPs in personal care products by the cosmetic companies [78] in coordination with the individual governments was much heralded as a way forward. This was seen to proceed in conjunction with better technologies in regard to wastewater treatment [79], although WWTPs themselves are not specifically designed to retain plastic microbeads. These measures would show little effect in regions to which MP laden products would still be exported and which do not have an adequate wastewater treatment infrastructure. Therefore, it would be advisable that international regulations would also focus on the export of MP containing products from countries that have regulations on plastic microbeads in place and on the import of such

products to regions, which have not yet implemented a ban on plastic microbead containing products. Alternatives for plastic microbeads are beads or microparticles made from crushed hazelnut, almond, pecan and especially walnut shells, from apricot and plum kernels, and from jojoba seeds, corn, oat or rice grain/meal as well as from refined wood pulp in form of microcrystalline cellulose. Purely inorganic natural alternatives are pumice, silica, and bentonites, including montmorillonite, or other clays such as talc. Furthermore, degradable, synthetic polymeric microbeads are being developed [80]-[83], and support for further research in this direction would be welcome.

5. Conclusions

This study analyzed 103 personal care and cosmetic products bought in markets in Punjab, Pakistan. 45.6% of the products contained MPs in their formulation, 42.7% of them PE microbeads. Comparing these numbers with studies from around the world and with data released from governmental organizations, the proportion of cosmetic products available in Pakistani markets carrying MPs is high. Especially concerning is the high proportion of MP containing products imported from countries, where a ban of plastic microbeads in rinse-off cosmetics is already in place. With 1.13 w% to 3.72 w%, the weight contribution of MPs to the individual products which contain plastic microbeads is similar to those reported for products available in other countries. As not all households in Pakistan are connected to a viable wastewater treatment facility, it is expected that significant amounts of MPs are emitted into the environment. Therefore, heightened consumer awareness, a reduction in the import of MP-containing rinse-off cosmetics, and continued research into affordable MP replacement materials would be of great value.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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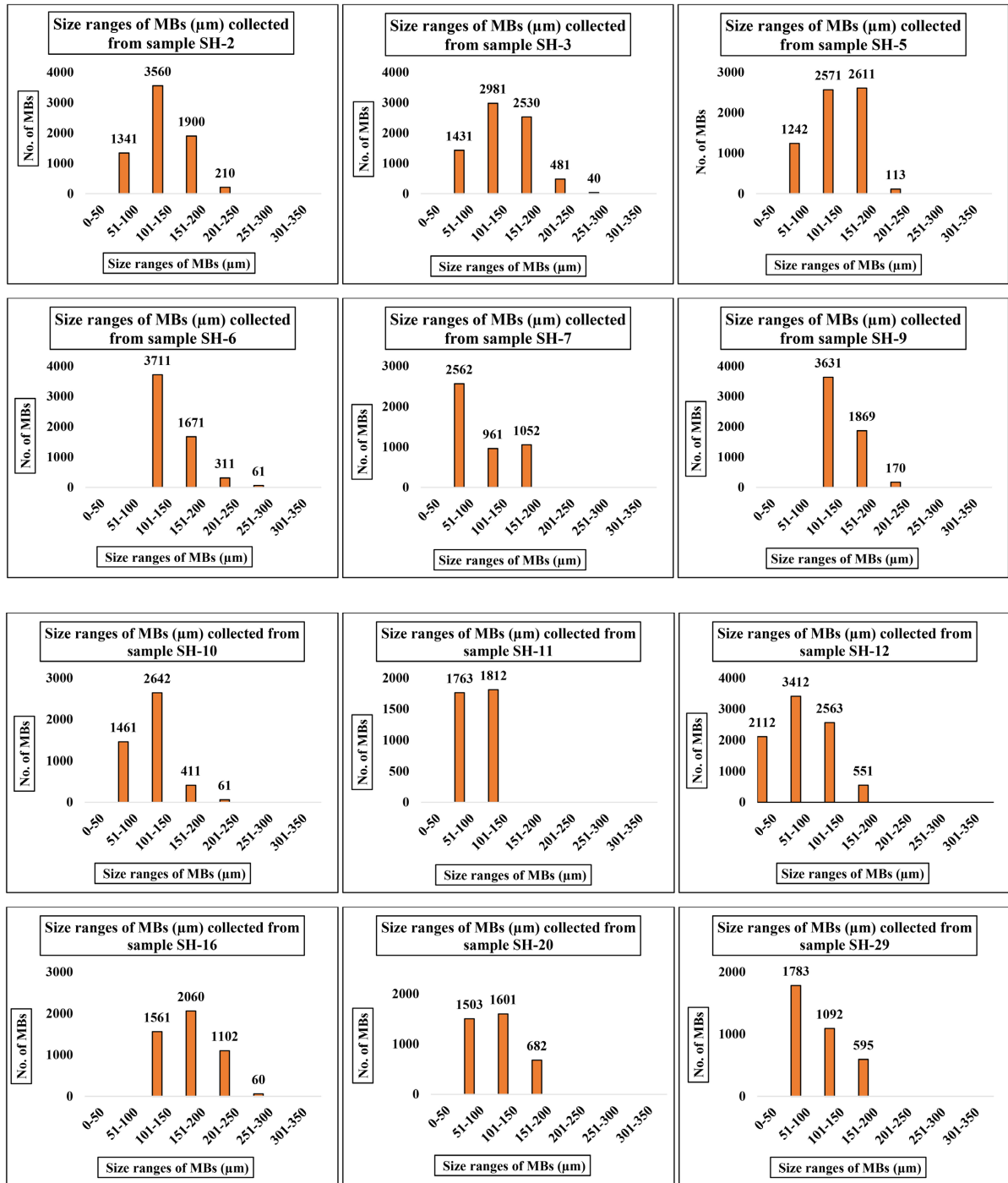
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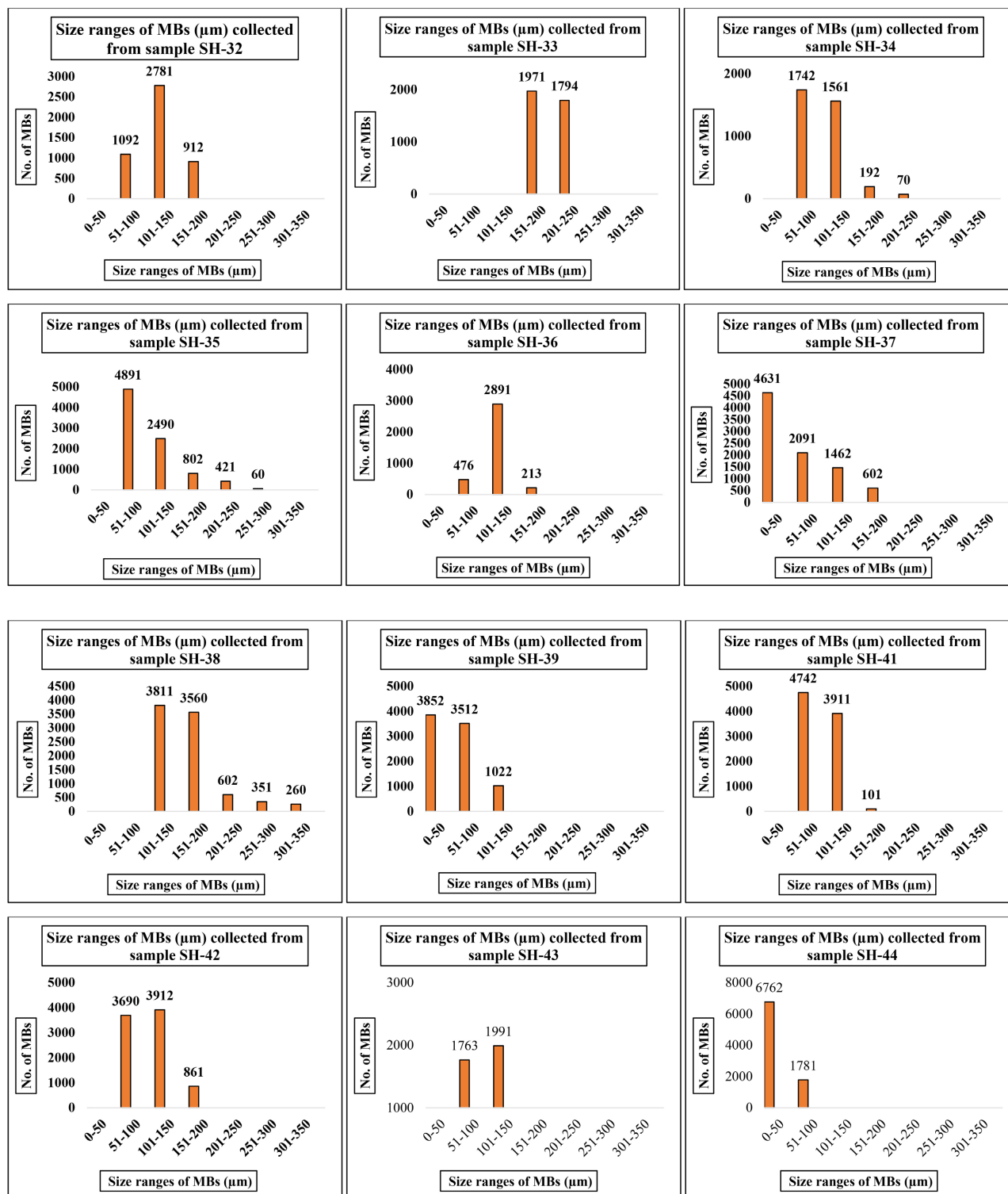
Supplementary Data

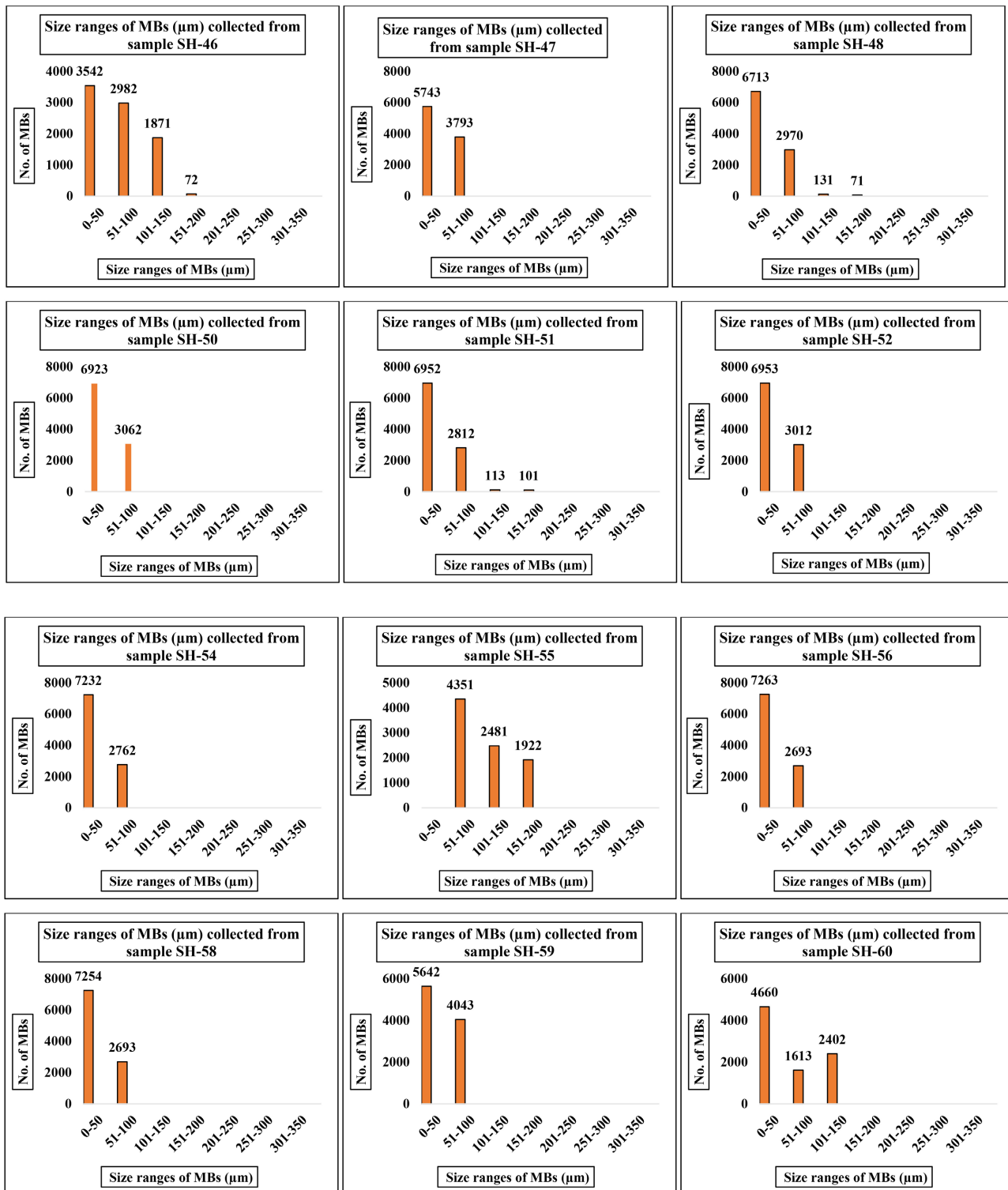
Size distribution graph of microbead containing products (**Figure S1**) pp. 2-14

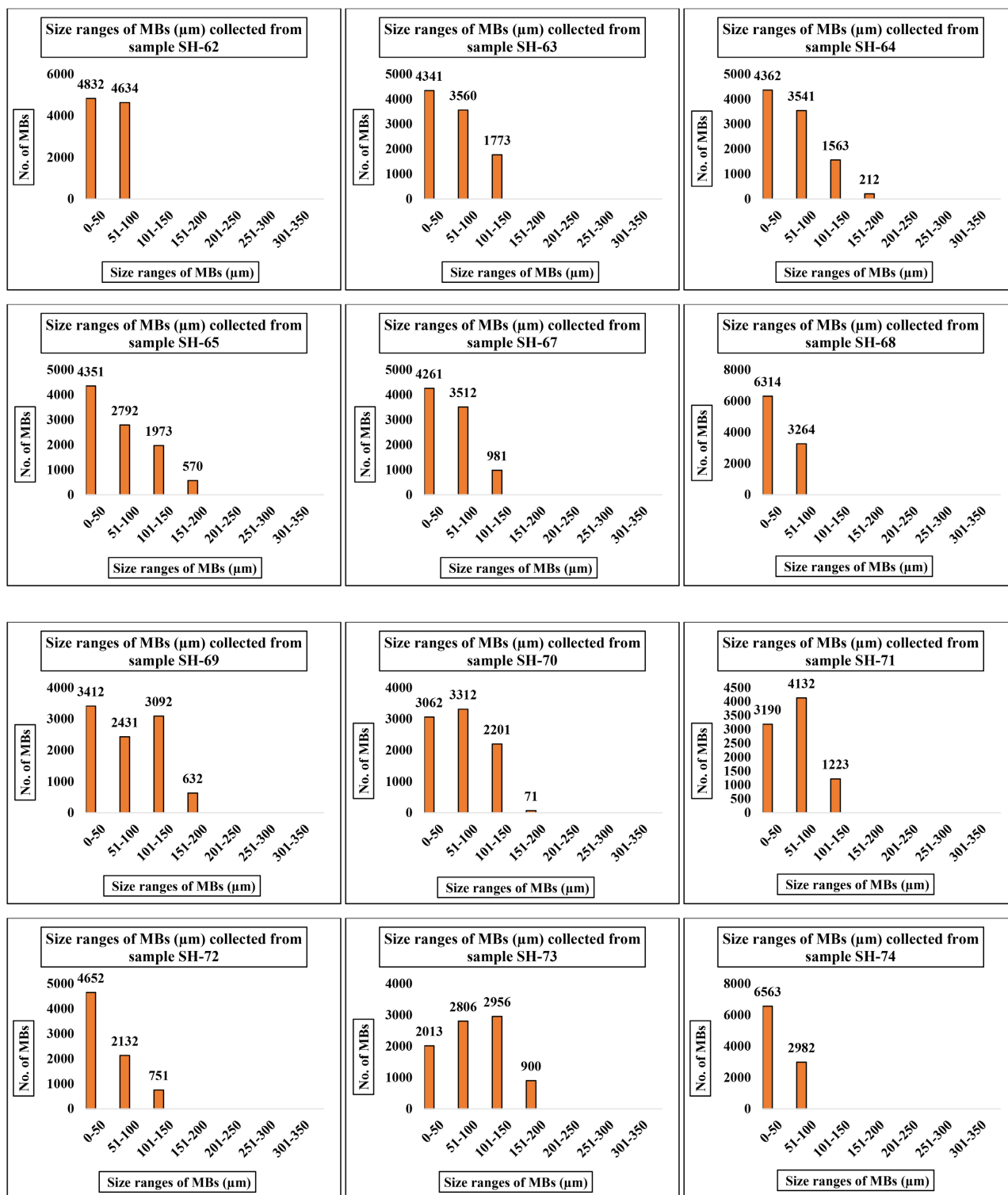
Categorization of the products according to color of the microbeads (**Figure S2**) p. 15

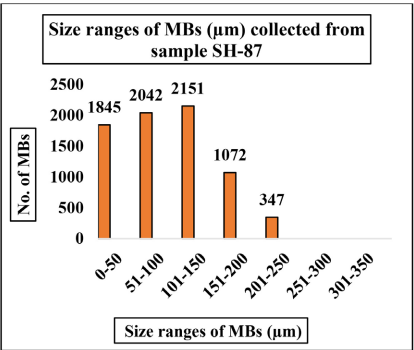
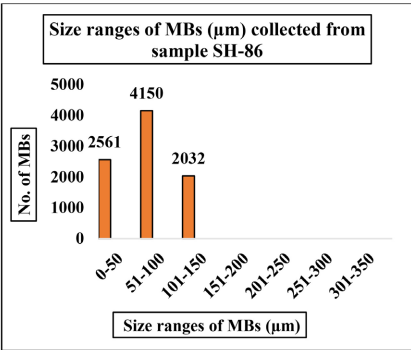
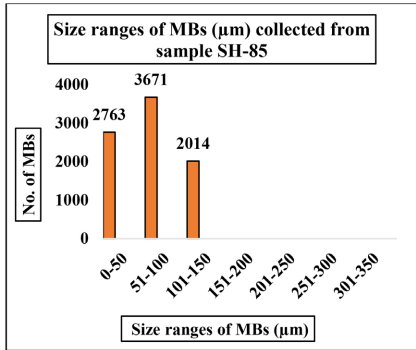
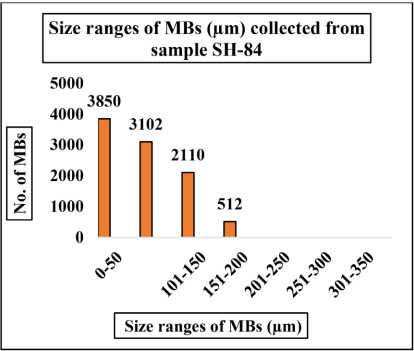
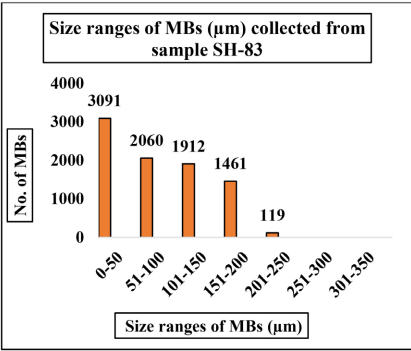
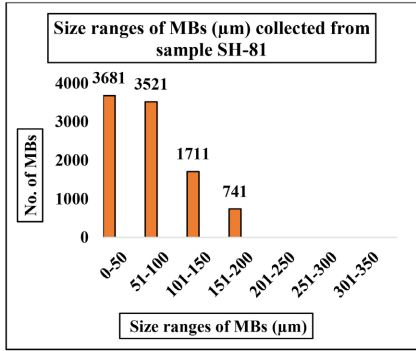
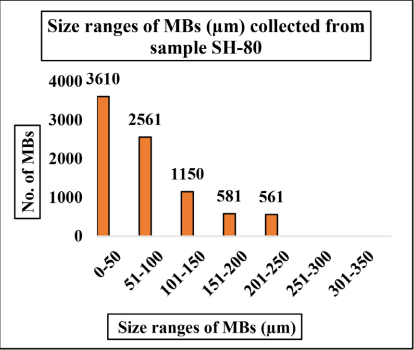
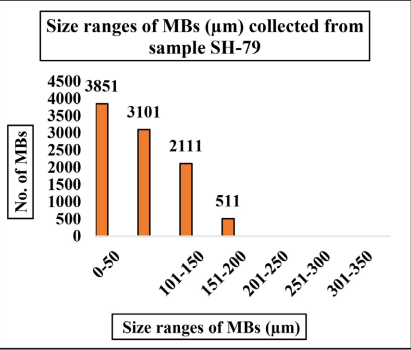
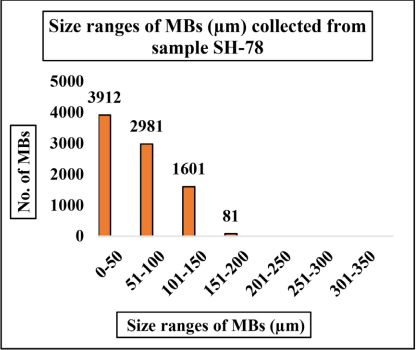
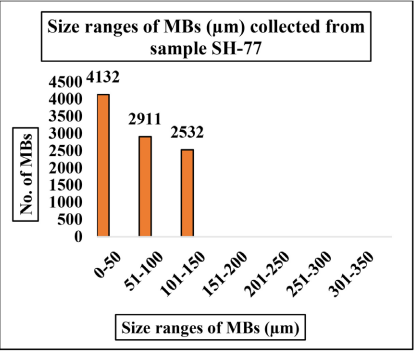
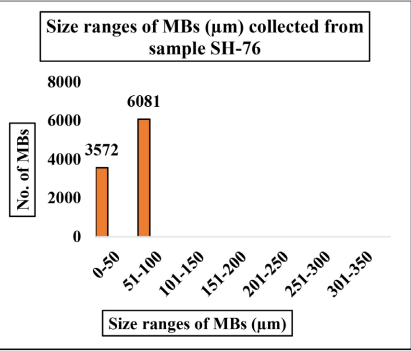
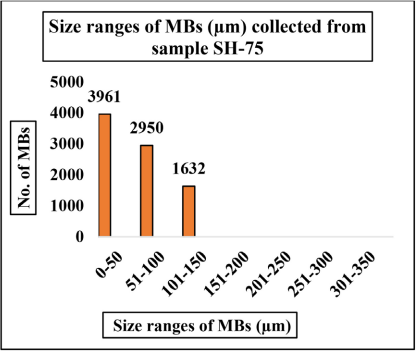
Categorization of the analyzed products by country as to their composition (no microbeads, plastic microbeads and non-plastic microbeads) (**Figure S3**) pp. 16-18

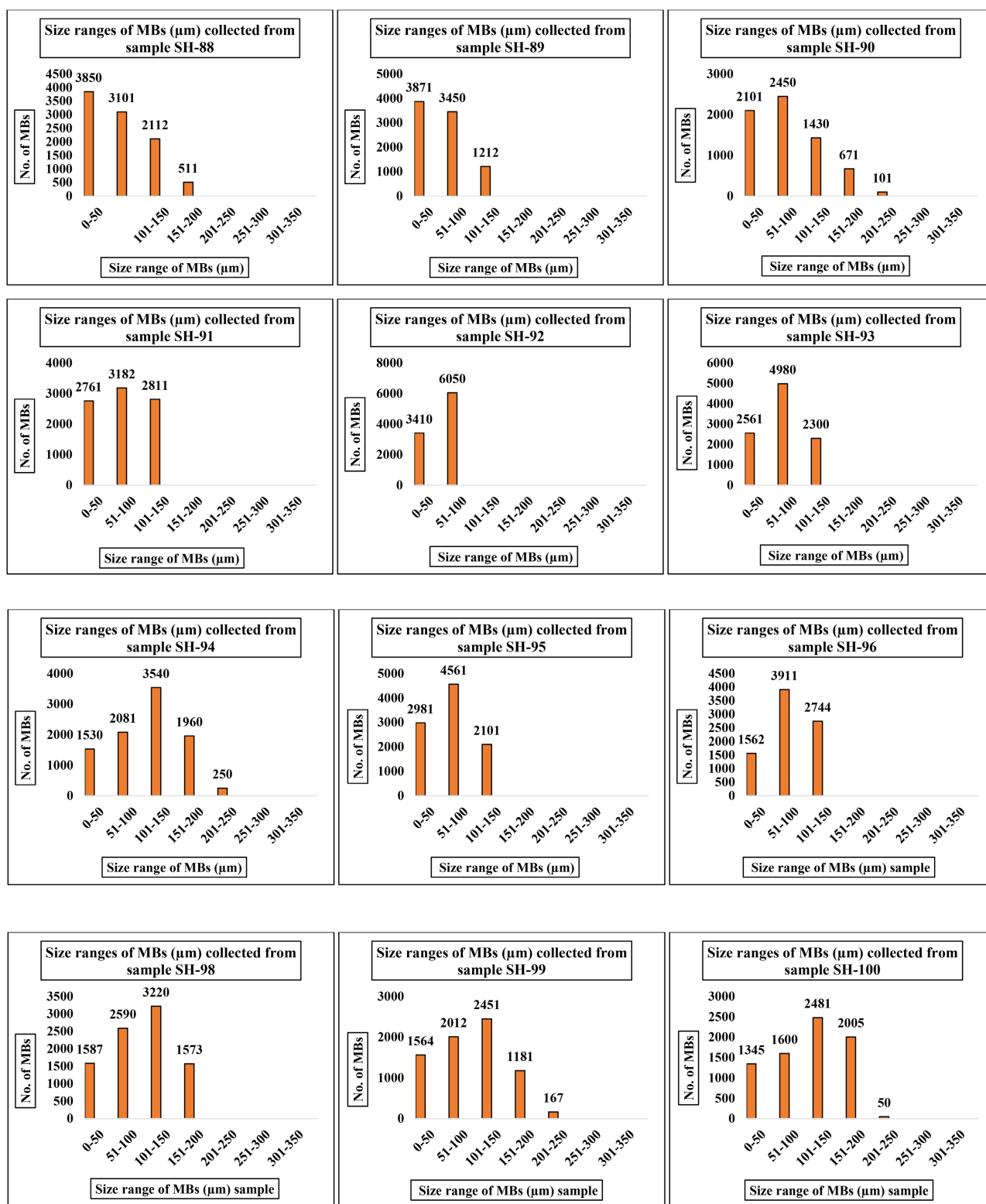


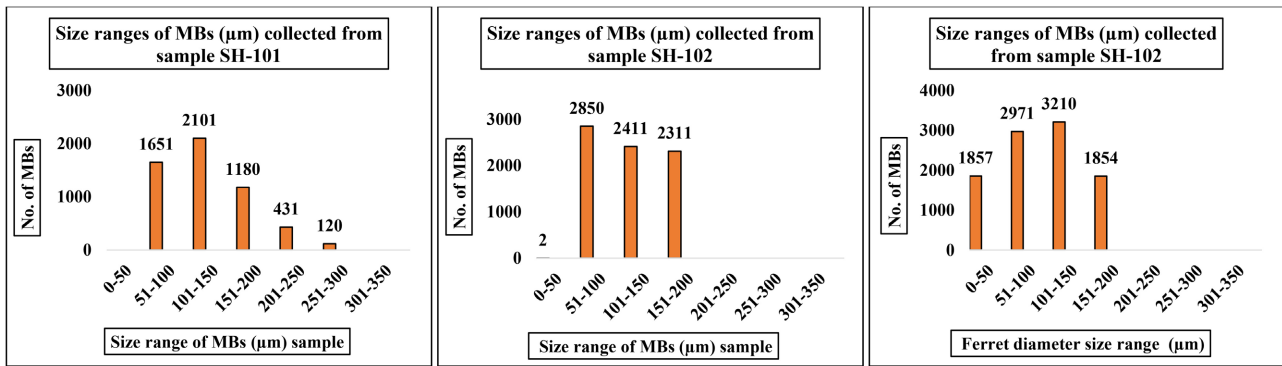












Figures S1. Size distribution graph of microbead containing products.

Figure S2. Categorization of the products according to color of the microbeads.

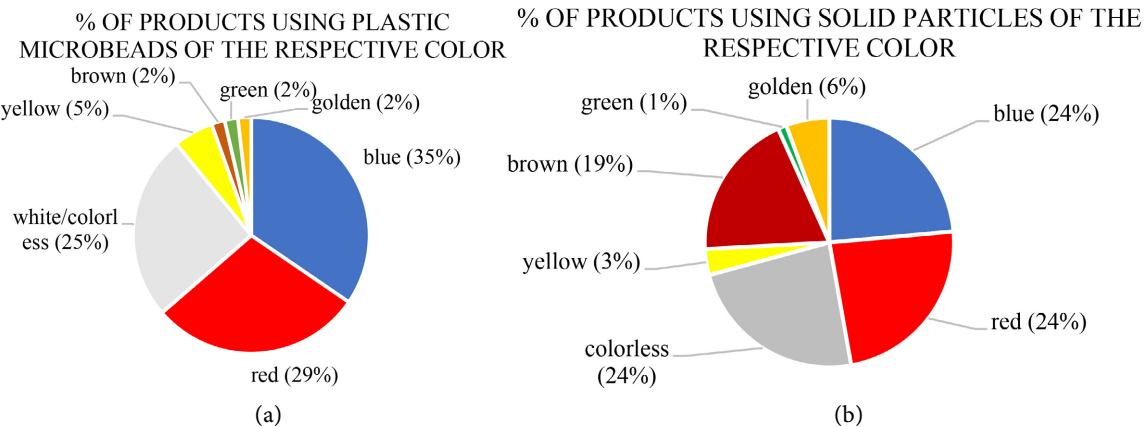
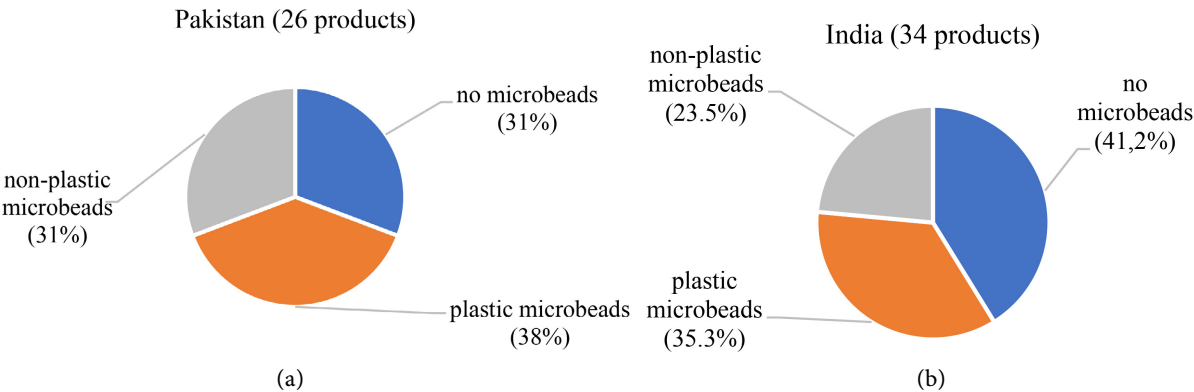


Figure S2. (a) Colors of the plastic microbeads in the examined products, (b) Colors of microbeads (plastic and non-plastic) in the examined products.

Figure S3. Categorization of the analyzed products by country as to their composition (no microbeads, plastic microbeads and non-plastic microbeads).



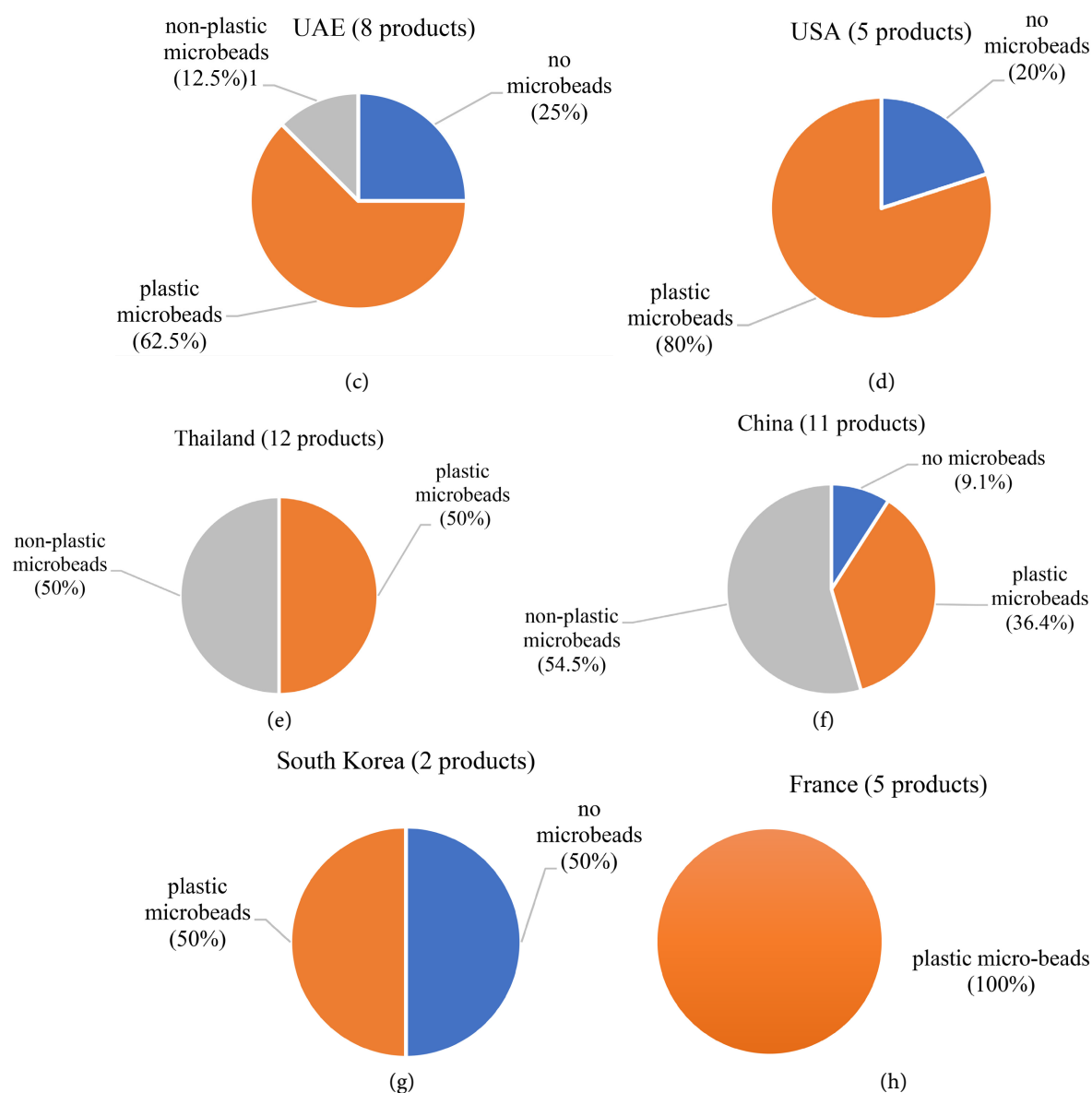


Figure S3. (a) Composition of products stemming from Pakistan, (b) Composition of products imported from India, (c) Composition of products imported from the United Arab Emirates, (d) Composition of products imported from the United States of America, (e) Composition of products imported from Thailand, (f) Composition of products imported from PR China, (g) Composition of products imported from South Korea, (h) Composition of products imported from France.