



Quantification and Characterization of Microplastics in Five Popular Indian Toothpaste Brands - A Comprehensive Analysis

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ABSTRACT: This research has investigated the presence and composition of microplastics in five popular toothpaste brands in India. Using a novel wet peroxide digestion method, the study successfully isolated and quantified microplastics in each brand, highlighting significant variations in concentration and polymer types. Sample S4 had the highest microplastic concentration (0.248 g/g) and the largest particle size (30 micrometers), while other brands exhibited lower levels. FTIR and AFM analyses identified multiple polymer types, including PET, PP, and PTFE, with sample S4 displaying complex surface topography, indicative of increased environmental interaction and pollutant absorption. One-way ANOVA confirmed significant differences in microplastic particle sizes across the brands. The study also performed principal component analysis (PCA), showing the intricate co-occurrence of polymers across samples, suggesting synergistic ecotoxicological effects. The results raise concerns about the potential health risks of daily exposure to microplastics through oral hygiene products, as well as the broader environmental implications, such as bioaccumulation in aquatic systems. The study calls for stricter regulation of microplastics in consumer products and further investigation into biodegradable alternatives.

KEY WORDS: Aquatic ecosystem, FTIR Analysis, Microplastics, Plastic Pollution, Toothpaste.

INTRODUCTION

Plastics have been around since the 1900s and our lives have been completely transformed by them. They are cheap and have corrosion resistance and electrical insulation properties. In our daily life, plastics have become one of the essential materials, used in transport, telecommunication, household objects and products, furniture, toys, clothing, footwear and also as packaging material which helps in easy transport of food material and other goods. Microplastics (MPs) are plastic fragments of varied shapes but below 5 millimetres (mm) in size [1-3]. Cosmetic products (shampoos, glitters, nail decorations) and personal care products (toothpaste, facial scrubs, soaps) contain MPs. These products are important contributors to primary plastics that contaminate the environment [4-5].

Toothpastes are formulated to maintain oral hygiene and address various dental issues. They generally contain abrasives (that scrub the enamel to remove embedded material), humectants (chemicals that retain moisture and prevent drying out), detergents (chemicals that create foam and help in cleaning), fluoride (the key ingredient that prevents tooth decay), binders and thickeners (chemicals that give toothpaste its texture and consistency), flavours and sweeteners (chemicals that improve the taste of toothpaste), preservatives (chemicals that prevent the growth of microorganisms in the toothpaste) and therapeutic agents (additional ingredients to cure/help specific dental problems) [6]. MPs in the form of 'Microbeads' were patented in the 1960s and used profusely as abrasives in toothpastes. Microbeads can serve multiple purposes: they act as bulking agents, exfoliants, and tooth polishers, while also extending shelf life by trapping and adsorbing degradable ingredients [7].

Millions of microbeads are flushed into the sewage system everyday and end up in the aquatic environment. Domestic sewage is often dumped without treatment into nearby freshwater ecosystems such as lakes, ponds and rivers in developing countries such as India from which drinking water is drawn. As a result of the alarm raised in the early 2000s about the accumulation of these MPs in oceanic ecosystems, countries such as the United Kingdom, Canada, France, South Korea, Sweden, New Zealand and Taiwan have banned their use in personal care products. The bans were issued between 2017 and 2018 [8]. India does not have any regulation for use of these MPS to date. Since awareness is increasing, more and more studies are focusing on the isolation and characterization of these MPs from toothpastes [9-11].



The objectives of the present study were to quantify and characterize microplastic content in five popular Indian toothpaste brands (SDG 12: Responsible Consumption and Production) and to identify potential environmental risks associated with microplastic release from toothpaste (SDG 14: Life Below Water).

MATERIALS AND METHODS

Selection and Purchase of Toothpaste: Five popular brands of toothpaste were chosen. We purchased 15 samples of each from random shops around Madurai, Tamil Nadu, India. It was ensured that the toothpaste purchased was the same brand and type within the brand. We have not mentioned the names of the brands. They were labelled S1 to S5 for further analysis.

Isolation of Microplastics: The toothpaste was weighed, dissolved in distilled water, and heated continuously on a hot plate. Once dissolved the solution was passed through a Whatman No. 1 filter paper. The filter paper was dried for three hours at 50° C in an hot air oven, this was kept for further analysis. The filtrate was allowed to cool and sedimentation was done overnight¹⁰. The sediment produced was collected and subjected to further wet peroxide digestion.

Wet Peroxide digestion [12]: The sediment was added to a 30% hydrogen peroxide solution and incubated at room temperature for 10 min. The solution was then heated to 75°C on a hot plate with a magnetic stirrer. Once gas bubbles were observed, the beaker was removed from the hot plate and placed in a fume hood until the boiling subsided. If it was felt that the reaction was about to overflow the beaker, distilled water was added to slow down the reaction. The mixture was then reheated at 75°C. Since natural organic material was still visible 30% hydrogen peroxide solution was added and the previous steps repeated till all the organic material dissolved. Sodium Chloride (NaCl) was added to increase the density of the aqueous solution (~5 M NaCl). The mixture was heated till the salt dissolved. This was then subjected to density separation.

Density separation [12] (with minor modifications): The entire reaction mixture was transferred into a separation funnel and solids were allowed to settle overnight. The floating solids were collected by blotting with a Whatman No. 1 filter paper. The settled solids were visually inspected for plastics, when they were observed they were removed with a forceps. The residues obtained after the above process were sent for further analysis.

Characterization of MP s:

- Fourier transform infrared spectroscopy (FTIR):** The material collected on the filter papers were stored in glass containers and were analysed by FTIR (Shimadzu - I Raffinity 1) at a range of 500 to 4500 cm⁻¹ and resolution 4.0. The resulting graphs were analysed to find bond patterns / peaks. The peaks were analysed based on existing reference values [13-15]. This confirmed the presence of plastic polymers.
- Atomic force microscopy (AFM):** The plastic particles were subjected to AFM (Nanosurf - Easy scan 2) analysis to confirm their size and to study their surface topology [10].

Data Analysis:

The data obtained was subjected to the following analysis for the reasons cited using Microsoft Excel (2019) and RStudio.

- One-way ANOVA to compare microplastic concentrations across brands
- Correlation analysis between microplastic concentration and particle size
- Principal Component Analysis (PCA) for polymer type composition across brands

Writing Assistance: <https://app.grammarly.com/> - The Grammarly web application was used to check for spelling and grammatical error in this paper.

RESULTS

Extraction / Isolation Methods

The novel wet peroxide digestion methodology demonstrated significant efficacy in microplastic isolation from toothpaste matrices. The procedure exhibited superior organic matter elimination through iterative hydrogen peroxide treatment, enabling enhanced visualization and separation of microplastic particles. This method's adaptability to varying organic content levels across different toothpaste formulations indicates robust analytical reliability. The protocol demonstrated consistent reproducibility across all samples, with minimal interference from organic constituents, representing a methodological advancement over traditional environmental sample processing techniques.



Quantitative Assessment of Microplastics

Analysis of five commercial toothpaste brands revealed distinct variations in microplastic concentrations. Sample S4 exhibited the highest concentration (0.248 g/g), while S2 and S5 showed intermediate levels (0.157 g/g and 0.158 g/g, respectively). The lowest concentrations were observed in S1 (0.101 g/g) and S3 (0.092 g/g). Notably, S4 demonstrated a significant differential between filtration residue (15.437 g) and post-digestion residue (12.437 g), indicating substantial organic matter content in the original formulation. (Table No: 1, 2 and 3)

Table No. 1: Mean concentration of Microplastics (g/g) of toothpaste samples

Brand	Initial weight of sample (g)	Mean residues obtained after filtration (g)	Mean residues after WP digestion (g)	Mean concentration of MPs (g/g)
S1	50	5.998	5.085	0.101
S2	50	8.054	7.854	0.157
S3	50	5.057	4.637	0.092
S4	50	15.437	12.437	0.248
S5	50	8.244	7.944	0.158

Table No. 2: Types of plastics present in toothpaste samples.

Type of Plastic	S1	S2	S3	S4	S5
Common Plastics					
Polyethylene glycol (PEG)	/		/	/	/
Polypropylene (PP)		/	/		/
Polytetrafluoroethylene (PTFE)	/	/		/	
Polyethylene Terephthalate (PET)	/			/	
Polyurethane (PU)				/	/
Uncommon Plastics					
Nitrile		/			
Polyethylene (PE)			/		
Polystyrene (PS)				/	
Ethylene vinyl acetate (EVA)					/
Poly methyl methacrylate (PMMA)					/

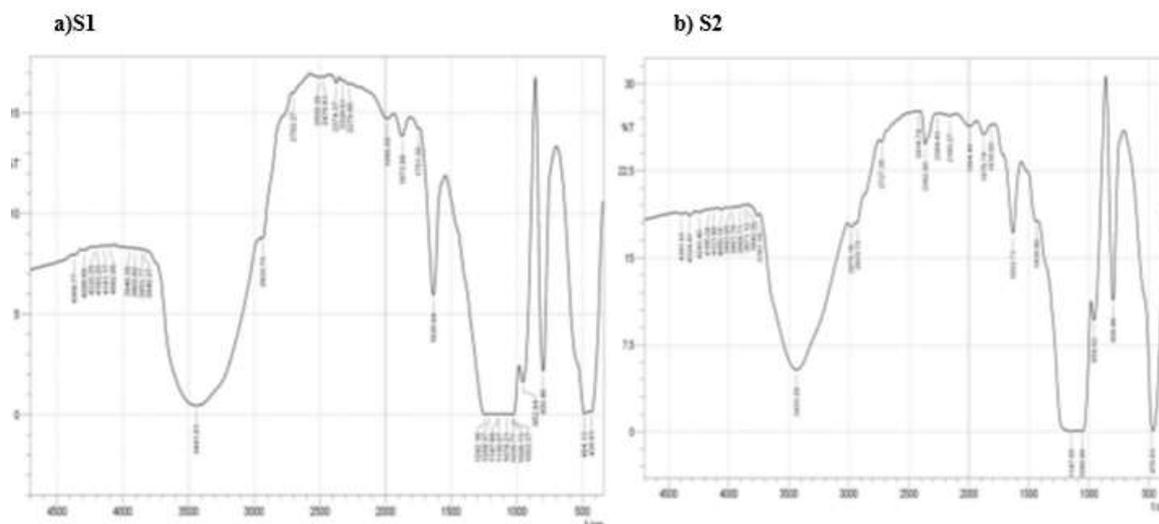
Table 3: Concentration Vs Size of Microplastics in toothpaste samples

Brand	Mean Concentration of MP in mg / gm	Mean size of particles in micrometers
S1	10.1	1.5
S2	15.7	1.6
S3	9.2	1.6
S4	24.8	30
S5	15.8	1.5

FTIR and AFM Analysis (Table No: 2 Figure No.: 1 and 2)

Fourier Transform Infrared Spectroscopy (FTIR) analysis identified diverse polymer compositions across the samples. The spectra revealed the presence of polytetrafluoroethylene (PTFE), polyethylene terephthalate (PET), and polyethylene glycol (PEG) in S1; polypropylene (PP), PTFE, and nitrile-based polymers in S2; polyethylene (PE), PP, and PEG in S3; PET, polystyrene (PS), PTFE, and polyurethane (PU) in S4; and PP, ethylene vinyl acetate (EVA), polymethyl methacrylate (PMMA), and PU in S5.

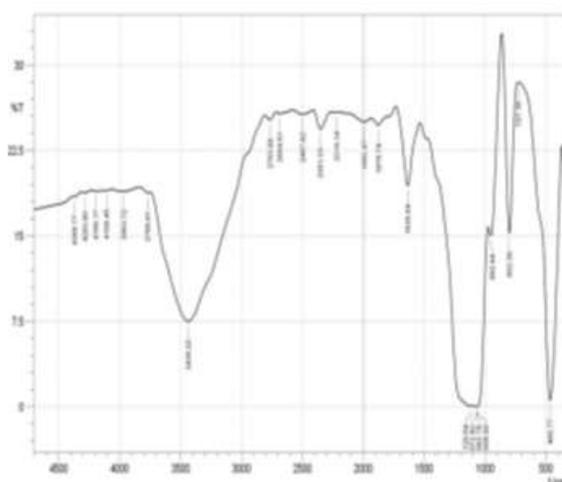
Figure 1: FTIR ANALYSIS OF ALL SAMPLES (S1 TO S5)



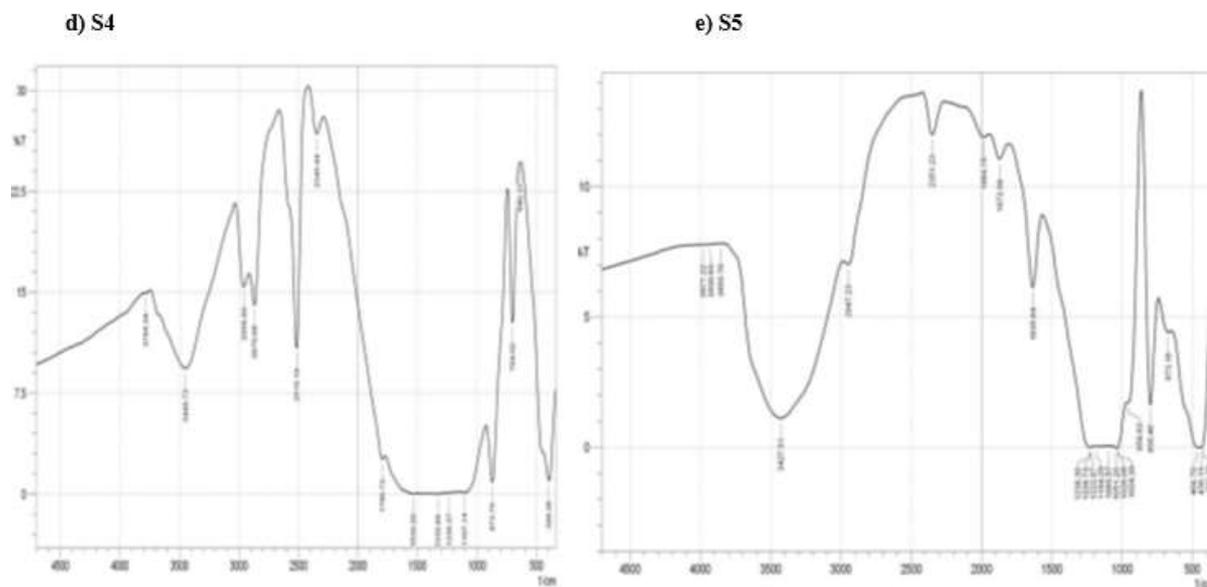
Observed Peak	Reference Peak	Chemical Bond	Type of Plastic
1147.65	1147	CF2 Stretching	Polytetrafluoroethylene (PTFE)
1242.16	1241	C-O Stretching	Polyethylene Terephthalate (PET)
3441.01	3444	O-H Stretching	Polyethylene glycol (PEG)

Observed Peak	Reference Peak	Chemical Bond	Type of Plastic
800.46	808	CH3 rocking, C-C stretching CH2 rocking, C-CH3 stretching CH2 rocking, C-C stretching	Polypropylene (PP)
1147.65	1147	CF2 Stretching	Polytetrafluoroethylene (PTFE)
1438.9	1440	CH2 Bending	Nitrile

c) S3



Observed Peak	Reference Peak	Chemical Bond	Type of Plastic
727.16	730	CH2 rocking	Polyethylene (PE)
802.39	808	CH3 rocking, C-C Stretching, CH2 rocking, -CH3 Stretching CH2 rocking, C-C Stretching	Polypropylene (PP)
1072.42	1071	C-O Stretching	Polyethylene glycol (PEG)



Observed Peak	Reference Peak	Chemical Bond	Type of Plastic
719.45	720	CH out of plane bending	Polyethylene Terephthalate (PET)
873.75	874	C-H bending	Polyethylene glycol (PEG)
1026.13	1027	C-O stretching	Polystyrene
1147.65	1147	C-O stretching	Polytetrafluoroethylene (PTFE)
1539.2	1531	C-N stretching	Polyurethane (PU)

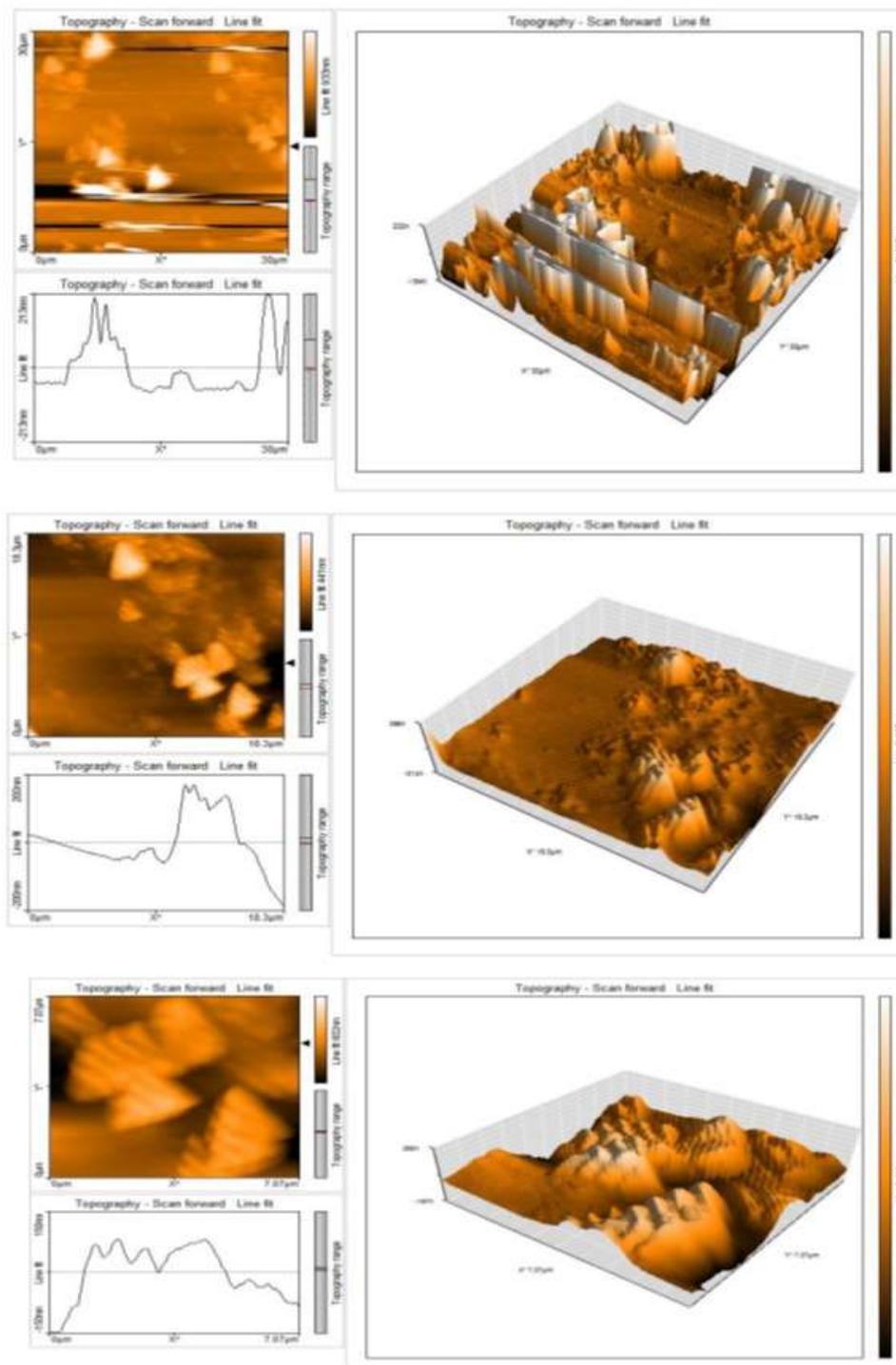
Observed Peak	Reference Peak	Chemical Bond	Type of Plastic
800.46	808	CH3 rocking, C-C stretching CH2 rocking, C-CH3 stretching CH2 rocking, C-C stretching	Polypropylene (PP)
1024.2	1020	C-O stretching	Ethylene vinyl acetate (EVA)
1184.29	1189	CH3 rocking	Poly methyl methacrylate (PMMA)
1222.87	1223	C(=O)O stretching	Polyurethane
1238.5	1245	C-N stretching	Polyethylene glycol (PEG)

Figure No. 2: MPs of S4 visible under a Dissection Microscope (10x)



AFM analysis (Figure No. 3) of S4 particles revealed complex surface topography with heterogeneous morphological features. The particles exhibited an average diameter of 30 micrometers, characterized by irregular conformations and elevated surface area to volume ratios. The surface structure analysis indicated potential for enhanced environmental interaction and pollutant adsorption capacity.

Figure No. 3: Topology of Particles for S4 using Atomic force Microscopy (AFM)



Statistical Analysis

One-way ANOVA demonstrated significant inter-brand variation in particle sizes ($F = 14919.37$, $p < 0.001$). The extremely low p-value ($9.1845E-102$) and high F-statistic, substantially exceeding the critical value (2.5027), provided strong evidence against homogeneity of group means. The minimal within-group mean square (0.039) indicated consistent particle size distributions within individual brands.

Correlation analysis between microplastic concentration and particle size revealed no significant relationship in four of the five samples (S1, S2, S3, and S5), which exhibited similar average particle sizes (1.5-1.6 micrometers) despite varying concentrations. S4 demonstrated unique characteristics with both maximum concentration and particle size.

Principal Component Analysis of polymer type composition required four principal components to account for 95% of the variance, indicating complex polymer distribution patterns. The high value ranges within each principal component suggested distinct plastic composition profiles across brands, with potential implications for synergistic ecotoxicological effects (Figure No. 4).

Figure No. 4: Biplot representing the Principal Component Analysis (PCA)

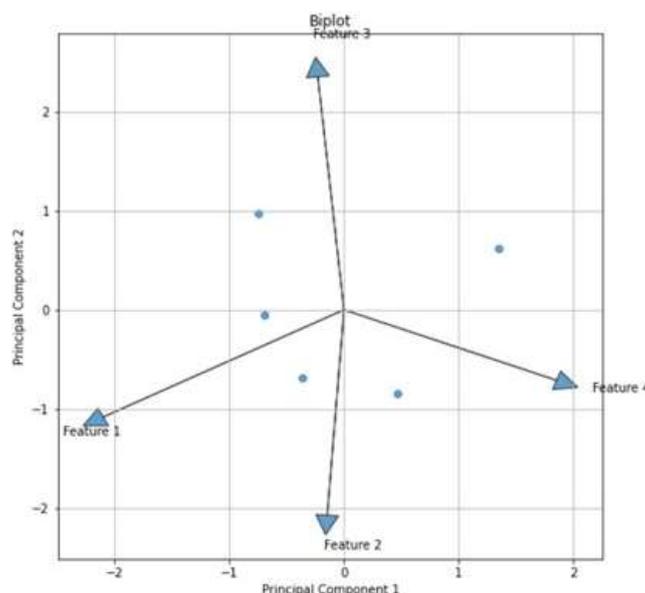


Figure 4: Biplot of the first two principal components (PC1 and PC2) from PCA of microplastic compositions in toothpaste samples. Sample points are represented by blue dots, while red arrows indicate the loadings of original variables (plastic types). The length and direction of the arrows show the contribution of each plastic type to the principal components. This visualization reveals distinct clustering of samples and highlights the relative importance of different plastic types in differentiating toothpaste brands.

DISCUSSION

Contemporary consumer behaviour demonstrates limited awareness of product composition and environmental consequences, particularly in oral care products. Consumer selection of toothpaste is predominantly driven by marketing appeal and brand popularity, with minimal consideration of ingredient implications or ecological impact.

Microplastics represent a critical environmental and health concern, particularly within aquatic ecosystems. These microscopic polymer fragments pose multifaceted risks through direct physiological disruption and chemical toxicity. Their ingestion by aquatic organisms initiates a complex bioaccumulation process, characterized by progressive tissue accumulation of hazardous chemical compounds, including heavy metals and persistent organic pollutants. The ecological ramifications extend beyond individual organism health, potentially compromising entire ecosystem functioning through habitat disruption and biodiversity reduction. Personal care products, utilized daily by global populations, serve as significant vectors for microplastic introduction into environmental systems.



This research investigates microplastic content in commercially available toothpaste brands, addressing a critical knowledge gap in understanding potential environmental and health risks associated with routine consumer products [20]. The methodological approach aims to provide empirical insights into microplastic characteristics, distribution, and potential ecological implications. The implementation of wet peroxide digestion methodology represents a significant advancement in microplastic isolation from complex consumer matrices. This technique demonstrates superior efficacy in organic matter elimination and microplastic visualization compared to conventional environmental sample protocols, facilitating enhanced accuracy in subsequent spectroscopic analyses.

The observed microplastic concentrations raise significant toxicological concerns regarding chronic oral exposure. The presence of multiple polymer types, particularly in S4, suggests potential synergistic effects in environmental systems. The complex surface topography observed via AFM indicates enhanced potential for contaminant adsorption and biological interaction, consistent with previous studies on environmental persistence of microplastics.

These findings align with international research documenting microplastic content ranging from 0.4% to 7.24% in oral care products. The variation in polymer composition among samples suggests divergent manufacturing protocols, potentially reflecting different functional requirements or cost considerations. The presence of specific polymers, notably PTFE and PU, warrants particular attention due to their environmental persistence and potential degradation products [21-22].

The statistical analyses reveal significant heterogeneity in microplastic characteristics across brands, suggesting the need for standardized manufacturing guidelines. The complex polymer distributions indicated by PCA underscore the potential for multiple environmental interaction pathways and varied toxicological impacts.

These results emphasize the necessity for enhanced regulatory frameworks governing microplastic inclusion in personal care products. Future research directions should prioritize investigation of chronic exposure effects, environmental fate modelling, and development of biodegradable alternatives. Additionally, comprehensive evaluation of polymer-specific environmental impacts and bioaccumulation patterns is warranted.

CONCLUSION

The research conclusively demonstrates the significant presence of microplastics in various toothpaste brands, with notable differences in both particle concentration and polymer types. The detection of plastics like PET, PP, and PTFE, especially in high concentrations within certain brands, raises concerns regarding both human health and environmental contamination. The identified large, irregular microplastic particles, particularly in sample S4, pose heightened risks of bioaccumulation and ecological disruption, emphasizing the urgency of addressing microplastic pollution in consumer products. This study highlights the necessity for more stringent regulations on the use of microplastics in personal care products, given their persistence and toxicity.

Future research should investigate the long-term ecological impacts of these microplastics, particularly their interaction with environmental pollutants, such as heavy metals and microbes. Additionally, further studies are required to assess the potential health effects on humans through chronic exposure, especially focusing on the retention and breakdown of microplastics in human tissues. Exploring alternative biodegradable polymers for use in consumer goods could offer a pathway to mitigate the ecological and health risks posed by synthetic microplastics.

To mitigate this issue, the first recommendation is to develop and implement eco-friendly alternatives to microplastics in toothpaste formulations. Secondly, wastewater treatment plants should be upgraded to better capture microplastics before they enter aquatic systems. Lastly, public awareness campaigns should educate consumers on the environmental risks associated with microplastic-containing products, encouraging the demand for safer, biodegradable options.

AUTHOR CONTRIBUTIONS

MSM: Study Design, Data compilation and analysis, writing and editing of the paper.

VA: Collection of samples, Running of experiments and collection of data

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- The Names of the brands remain undisclosed in the present paper but are available with the author for reference.

CONFLICT OF INTEREST

The Authors declare NO conflict of interest.

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