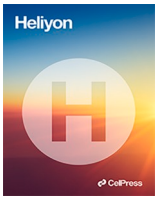
Heliyon 10 (2024) e32805

Contents lists available at ScienceDirect

Heliyon

journal homepage: www.cell.com/heliyon



Research article

Assessing microplastic contamination in soda beverages: A Multi-city, Multi-container laser Direct infrared spectroscopy study

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ARTICLE INFO

Keywords:
Microplastic
Soda beverage
Laser direct infrared spectroscopy
Principal component analysis

ABSTRACT

Microplastics are tiny plastic particles, typically smaller than 5 mm in diameter, that result from the degradation of larger plastic products. Minuscule pollutants are increasingly being found in our food supply, especially in beverages, raising substantial health concerns. Ingested microplastics can release hazardous chemicals and act as carriers of pathogens, leading to adverse health effects upon chronic exposure. Despite the numerous studies on microplastic contamination, few have assessed the influence of geographic location and container type on the presence of microplastics in beverages. Our comprehensive study bridges this research gap by collecting a particular soda beverage from Atlanta, Chicago, Los Angeles, and Washington D.C. and examining three different types of beverage containers: aluminum, glass, and plastic. Using direct laser infrared spectroscopy, we identified the types and quantified the numbers of microplastics. Our statistical analysis, which incorporated principal component analysis, investigated the distribution of microplastics in beverage samples, focusing on the impacts of geographic location and container material. Notably, our analysis revealed that the microplastic profiles were distinguishable in some cities, although not all. Conversely, no distinguishability was revealed between the different container types. This study sheds light on the complex patterns of microplastic contamination according to geographical location and packaging. Our findings contribute to a broader effort to understand and address the widespread challenges of microplastics, with implications for public health and ecosystem preservation.

1. Introduction

Soda beverages, which originated in the 18th century, have become a staple for global consumption, with billions of servings enjoyed daily [1]. Although precise statistics on the most recent consumption are lacking, data from 2011 to 2014 show that 63 % and 49 % of American youth and adults, respectively, consumed sugar-sweetened beverages on a daily base, with sodas being the predominant type in this category [2]. Soda beverage contamination can arise from various sources including impurities in water, tainted syrups or flavors, and manufacturing equipment [3,4]. Internationally, research has been conducted on soda beverages to identify the presence of emerging contaminants, such as heavy metals [5], bacteria [6], and organic compounds [7].

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https://doi.org/10.1016/j.heliyon.2024.e32805
Received 2 April 2024; Received in revised form 3 June 2024; Accepted 10 June 2024
Available online 10 June 2024

Microplastics (MPs), defined as tiny plastic particles ranging in size from 1 μm to less than 5 mm, pose substantial potential risks to both the environment and health [8]. These particles can transport invasive species and pathogens, disrupt ecosystems, and affect key elements of the food web such as plankton. MPs also act as carriers of environmental contaminants such as polychlorinated biphenyls, polycyclic aromatic hydrocarbons, and polybrominated diphenyl ethers, which can be ingested by animals [9–11]. As vectors of heavy metals and persistent organic pollutants, MPs present a threat to a wide range of organisms. Human exposure to MPs through the consumption of contaminated seafood, water, and other foods raises concerns regarding the introduction of harmful pathogens and the release of toxic compounds, potentially leading to inflammatory responses or hormonal imbalances [12–14].

Despite their ubiquitous presence in everyday consumables, including soda, bottled water, and tea [15–17], research on MP contamination in sodas has been scarce, with a particular gap in understanding the effects of geographical location and packaging type variables. Our study aimed to bridge this knowledge gap by utilizing laser-direct infrared (LDIR) spectroscopy to identify and quantify MPs in soda. LDIR technology offers comprehensive characterization of each MP particle by simultaneously collecting visual images and infrared (IR) spectra, allowing for in-depth analysis. This approach provides detailed geometric and chemical information for each particle, enabling a more nuanced understanding of MPs.

We examined a particular soda beverage from four major US cities—Atlanta, Chicago, Los Angeles, and Washington, DC—and analyzed the beverages contained in three different packaging materials: aluminum, glass, and plastic. This study provides critical insights into the dynamics of MP contamination, contributing to more sustainable practices in the beverage industry and guiding potential regulatory measures to curb MP pollution.

2. Materials and methods

2.1. Sample preparation

For this study, a specific soda beverage was selected; the brand remained confidential to avoid potential bias or misinterpretation. Beverage samples were collected from retailers in four major US cities: Atlanta (A), Chicago (C), Los Angeles (L), and Washington, DC (W). The samples contained three types of packaging materials: aluminum (A), glass (G), and plastic (P). At least three samples from each city and packaging material combination were examined and labeled numerically as 1, 2, and 3. The labeling convention used was a combination of letters and numbers, following the "city-material-sample number" format, such as WP for a Washington DC-plastic combination or CA2 for the second sample from Chicago in an aluminum container.

Prior to filtering, the soda samples were left unsealed for 60 min to allow the release of carbon dioxide. The filtration setup consisted of a detachable syringe filter (Ks-Tek, 25 mm) in combination with a polytetrafluoroethylene (PTFE) membrane filter paper (Deschem, 5.0 μm pore size, 25 mm diameter), and a 50-mL glass syringe (Tomopal) equipped with a metal Luer lock. This assembly creates an effective, straightforward, and efficient vacuum filtration system.

Each soda container underwent three water rinses after filtration using 20 mL of MP-free water per rinse, and the wash was filtered to capture any remaining MPs. This procedure is also essential for removing water-soluble contaminants from filter paper. Following this, the filter paper was subjected to three ethanol rinses using 10 mL of pure ethanol (99.5 %, Sigma Aldrich) each time, to cleanse the solids and eliminate water traces.

The filter paper with MPs was then transferred to a 50 mL glass bottle containing 20 mL of Pure Ethanol (99.5 %, Sigma Aldrich) and sealed with an aluminum cap. Each sample was agitated at 3000 rpm for 5 min using a vortex mixer (Four E's Scientific Laboratory) to ensure even distribution of the MPs in ethanol. The filter paper was removed from the bottle and the samples were left to evaporate until the volume was reduced to approximately 1 mL.

This concentrated solution was carefully added dropwise onto a MirrIR low-E glass microscope slide (Kevley Technologies), targeting a 12-mm diameter area on the slide designated for testing. To hasten the evaporation of the remaining ethanol, the slides were heated to a temperature of approximately 100 °C. Once dry, the IR-glass slides bearing MPs were prepared for in-depth analysis using an LDIR analyzer.

2.2. LDIR MP analysis

The Agilent 8700 LDIR imaging system was used for the detection and analysis of MPs ranging from 20 to 500 μm in size. This state-of-the-art system is equipped with a Quantum Cascade Laser that allows rapid and precise scanning across various infrared wavelengths. The LDIR system not only produces detailed visual representations of the particles, but also collects comprehensive spectral information for each one, within the range of 975–1800 cm^{-1}. The spectral data obtained for each MP particle were then compared against a sophisticated library contained in Agilent Clarity software (Version 1.5.58, Agilent Technologies, USA). This robust database enables precise identification of each particle by matching its unique spectral fingerprint against the library, ensuring high-quality analytical results.

2.3. FTIR analysis

The core aim of this study was to scrutinize the presence of MPs in selected beverages. However, it is critical to acknowledge the possibility that additional MPs may be inadvertently introduced during the storage or handling of samples. These extraneous MPs can come into contact with the samples via storage containers or processing equipment, making it difficult to differentiate them from the MPs originally contained in the beverage. To maintain the integrity of our findings, it was imperative to meticulously document all

potential sources of contamination. This diligent approach to identify and record possible external MP sources is the cornerstone of our methodology, ensuring that our analysis is transparent and that readers are fully informed of all variables that could influence the presence of MPs in the samples.

Our analysis yielded substantial insights into the materials constituting various components of the packaging.

1. The filtration paper membrane was fabricated from PTFE, which is nonreactive.
2. The core material of the filter was polypropylene, which was selected because of its durability and chemical resistance.
3. The plastic soda bottles were constructed using polyethylene terephthalate, which is recognized for its strength and clarity.

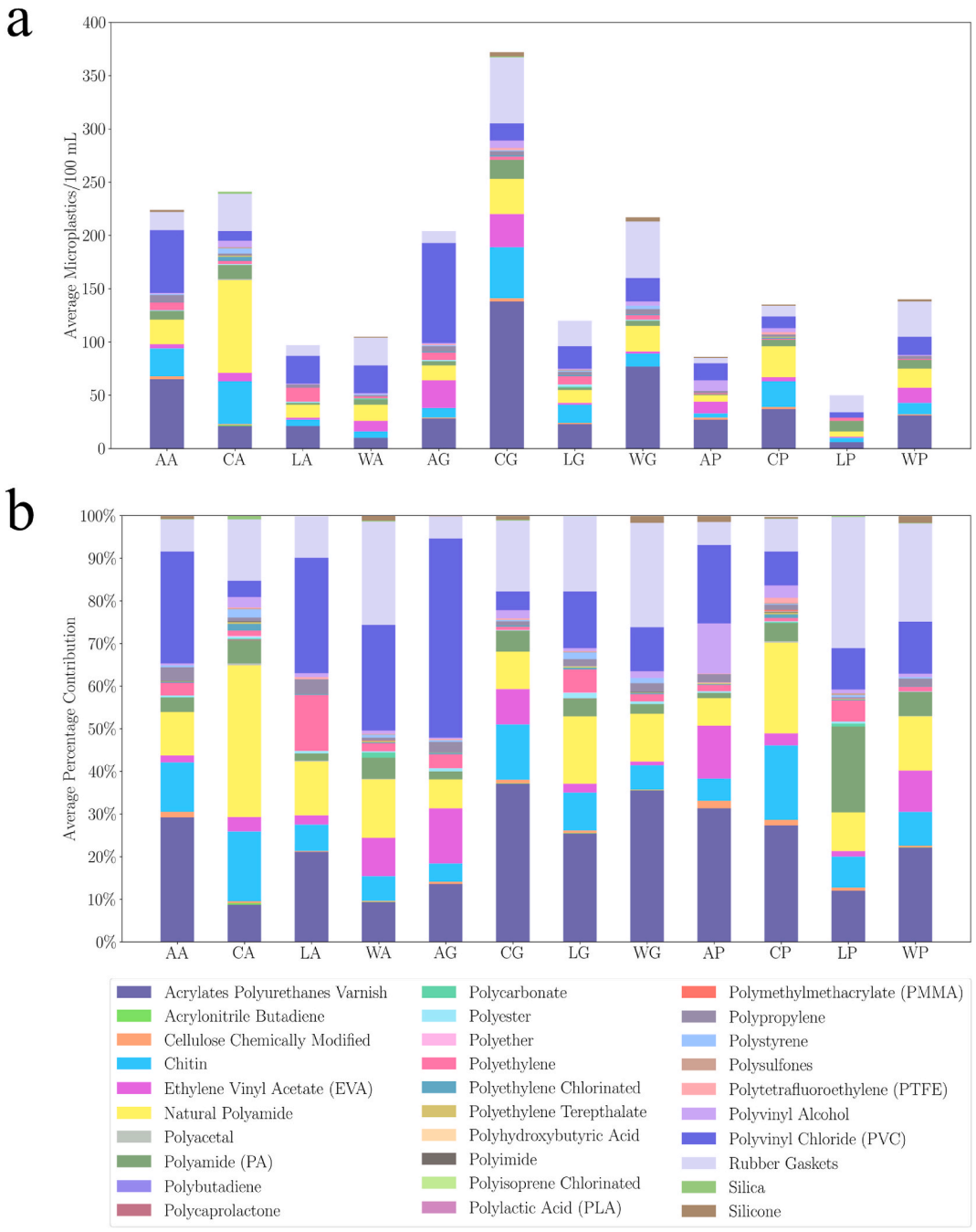

Fig. 1. **a.** Average count of microplastics per 100 mL found in each type of soda beverage. **b.** Percentage representation of microplastic types in each soda beverage. Each soda beverage is designated with a two-letter code: the first letter signifies the city of purchase—A for Atlanta, C for Chicago, L for Los Angeles, and W for Washington DC; the second letter denotes the packaging material—A for aluminum, G for glass, and P for plastic.

4. The caps used to seal the plastic containers were predominantly composed of polyethylene, which is noted for its flexibility and resilience.
5. The padding within the caps of the glass bottles was derived from polyethylene to ensure a tight seal.
6. The internal coating of the aluminum cans was discerned as a polymethyl methacrylate-type material, providing a protective barrier between the beverage and the metal.

2.4. Quality control/quality assurance

Comprehensive quality control protocols were implemented to minimize the risk of MP contamination. LDIR analysis confirmed

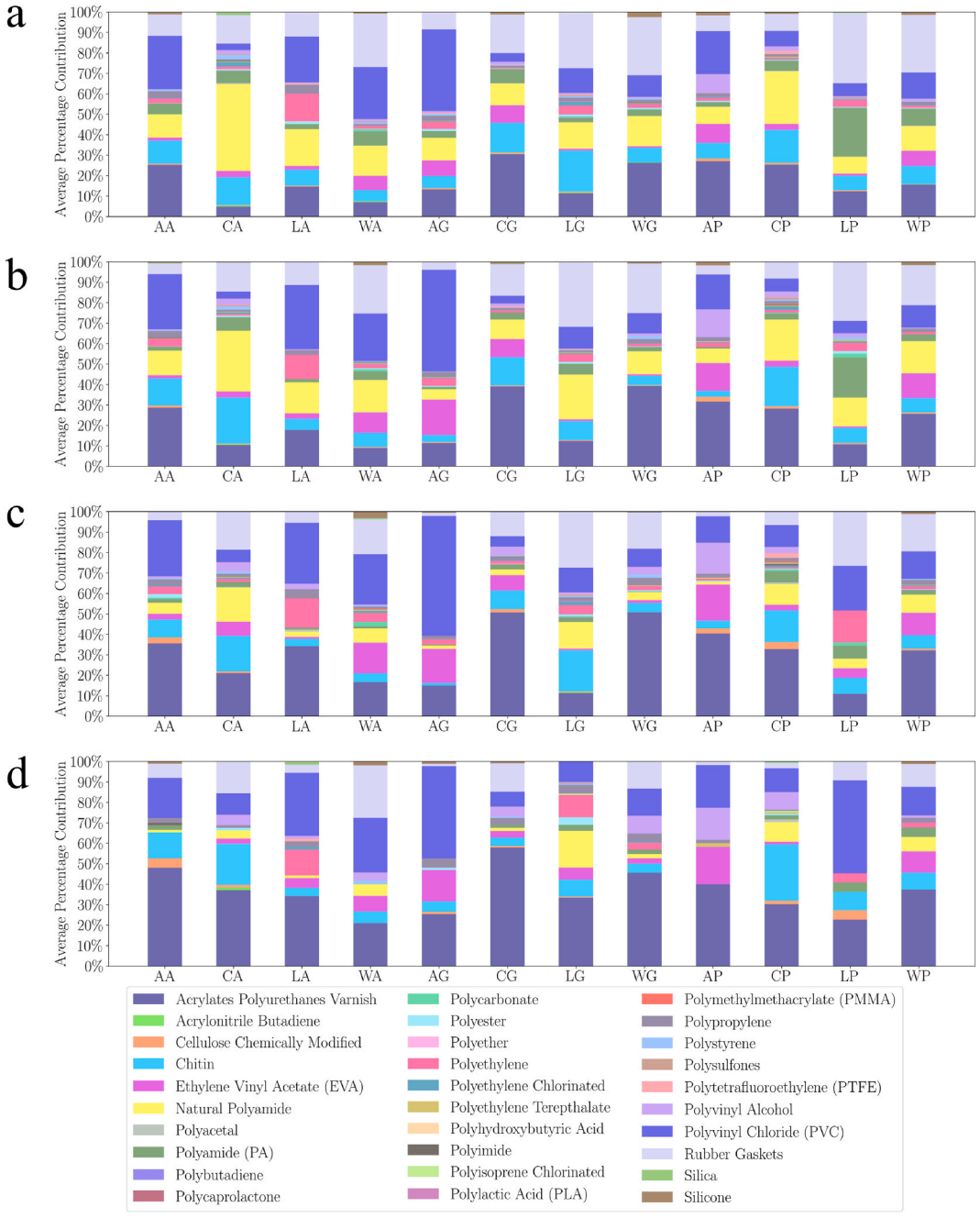

Fig. 2. Percentage representation of microplastic types, with sizes within the **a.** 20–30 μm, **b.** 30–50 μm, **c.** 50–100 μm, **d.** 100–500 μm range, in each soda beverage.

that the water and ethanol used in the processes were devoid of MPs, ensuring their suitability for the procedures. Nonplastic equipment, such as glass syringes, glass bottles, metal caps, and metal tweezers, have been used to reduce potential plastic-derived contaminants. All instruments were cleaned meticulously with MP-free water and ethanol to maintain sample integrity. The preparation of IR glass slides included a verification step under a microscope to ensure cleanliness before use. During sample handling, the operators adhered to rigorous standards of hygiene and safety by wearing nitrile gloves and pure cotton lab coats to minimize the introduction of contaminants. The effectiveness of these contamination prevention strategies was verified using blank tests, which reinforced the credibility of the findings.

2.5. Data processing

Data were collected using Agilent Clarity analysis software (version 1.5.58, Agilent Technologies). The subsequent extraction and quantification of MP data were performed using Excel® 365 (Version 2308 Build 16731.20234, Microsoft, USA). Principal component analysis (PCA) was carried out using Solo™ software (version 8.3, Eigenvector Research, USA), which provided the necessary statistical tools for in-depth data interpretation and pattern recognition.

3. Results and discussion

3.1. Overview of MPs in soda beverages

Fig. 1a shows the average number of MPs/100 mL of each soda beverage sample. MPs were identified in all 12 analyzed samples. On average, the concentration of MPs of all the samples was 166 ± 62 MPs/100 mL. The minimum recorded concentration was 51 ± 11 MPs/100 mL (LP), while the maximum was 371 ± 111 MPs/100 mL (CG). Fig. 1b shows the percentages of MP types in each soda beverage. In the combined data from the 12 soda beverage samples, polyurethane acrylate varnish was the most predominant type of MP, comprising 24.2 % of the total MPs. Following were polyvinyl chloride (PVC) at 16.2 %, rubber gaskets at 15.0 %, natural polyamide at 13.9 %, and chitin at 10.5 %. Smaller percentages were observed for ethylene vinyl acetate at 5.6 %, polyamide at 4.1 %, polyethylene at 2.6 %, polyvinyl alcohol at 1.8 %, and polypropylene at 1.8 %. Other MP varieties combined made up the remaining 4.3 %, with each individual type contributing <0.5 % of the total MP tally. The least prevalent among the MPs, polybutadiene, polyhydroxybutyric acid, and chlorinated polysoprene, each accounting for a fraction under 0.01 % of the total detected. On average, MPs within the 20–30 μm size range constituted 55.4 % of the total count, while 27.6 % of MPs were within the 30–50 μm range. MPs within 50–100 μm accounted for 11.0 % and a smaller proportion of 6 % were >100 μm–500 μm. Although smaller MPs are more predominant, their cumulative volume may be substantial less than that of larger MPs because the volume of an object increases with cube size.

Fig. 2a–d delineate the distribution of MP types across these four size ranges in each soda beverage sample. The predominant types of MPs across the different size ranges were consistent with those identified in the cumulative data analysis. A summary of the MP statistics for bottled water is provided in the Supporting Information.

MPs can infiltrate soda beverages via various routes. In this study, we explored the most prevalent MPs and their potential origins. The prevalence of polyurethane acrylate in soda production is likely due to its widespread application as a coating and sealing material, integrated into various stages and pieces of equipment in soda manufacturing. Although it is plausible that polyurethane acrylate is linked to the production process, particular equipment, or specific operational steps, a more detailed investigation is required to accurately identify its source in soda production. Similarly, the presence of rubber gaskets in soda can be traced back to their use in manufacturing equipment and machinery. Commonly used in soda production for sealing valves, pipes, and containers, gaskets are also potential sources of contamination. PVC most likely originates from the production environment, given its frequent application in pipelines, containers, and equipment parts in soda manufacturing facilities. Polyamides in soda may also be related to manufacturing processes or equipment. In contrast, chitin, a natural substance, is more likely to originate from the raw materials used in soda production and serves as an inherent component of the ingredients.

3.2. MPs in soda beverages from different cities

All three samples from Los Angeles (LA, LG, and LP) consistently exhibited the lowest MP concentrations across all container types, with an average presence of 89 ± 26 MPs/100 mL. This level is substantially lower, by 58 %, compared with the average MP count in soda beverages from Washington DC (WA, WG, and WP), which indicated a concentration of 155 ± 77 MPs/100 mL. This significant discrepancy suggests that Los Angeles employs more effective practices for MP filtration or containment than those observed in other surveyed cities. Samples obtained from Washington, DC, displayed a consistently moderate presence of MPs across all types of containers. This indicated a uniform and moderate degree of effectiveness in the management and containment of MPs within a city's infrastructure. Soda beverages purchased from Atlanta revealed an average MP concentration of 173 ± 64/100 mL. The data indicated a higher MP count in glass bottles (AG) and aluminum cans (AA), whereas a lower count was observed in plastic bottles (AP). These discrepancies may reflect the differential efficacy of MP containment and control among the various packaging materials used in Atlanta. The MP levels in Chicago's soda beverage samples are notably high, especially in glass bottles (CG) and aluminum cans (CA), with an average count of 250 ± 83 MPs/100 mL. This elevated count suggests that the methods of MP filtration or containment in Chicago may be less effective than those in other regions.

3.3. MPs in soda beverages with different packaging

Soda beverages packaged in plastic bottles displayed consistently low MP concentrations, averaging 104 ± 30 MPs/100 mL. Aluminum cans from Los Angeles and Washington DC mirrored the low MP levels found in plastic bottles, while those from Atlanta and Chicago exhibited higher concentrations, yielding an overall higher average for aluminum cans at 168 ± 73 MPs/100 mL. Conversely, soda beverages in glass bottles demonstrated uniformly high MP concentrations across all cities, with an average of 228 ± 84 MPs/100 mL. This disparity can be attributed to several factors: 1. glass and aluminum containers undergo frequent recycling, which can introduce MPs through wear and tear; plastic bottles may not undergo abrasive recycling; 2. aluminum cans are typically lined with a plastic layer to prevent the beverage from interacting with metals that can break down and release MPs; glass bottles may have similar coatings that contribute to MP content; and 3. the complex sealing mechanisms of aluminum and glass containers, which can include various types of caps and tabs, may be more susceptible to degradation, leading to MP release. Plastic bottles with their simpler twist-off caps may impart less mechanical stress during opening and sealing, potentially reducing MP release. Notably, these explanations for the observed disparities in MP concentrations among the different container types are currently speculative and need to be substantiated by rigorous research to confirm or refute these hypotheses.

The research findings revealed that plastic bottles have the lowest levels of MPs, which may seem counterintuitive given their material composition. This aligns with the understanding that MPs are fragments of plastic breakdown. All samples analyzed in our study were within their shelf life, which is much shorter than that required for substantial plastic degradation. This suggests that plastic bottles do not substantially increase MP levels over a relatively short period. While it is tempting to speculate on the impact of older plastic bottles on the presence of MPs, confirming this hypotheses requires further research.

3.4. PCA on MP data

Beyond conducting statistical analyses on the MP distribution in soda beverages, we can also treat the unique distribution within each beverage as a "fingerprint" vector. Employing data mining techniques allows us to explore the intricate patterns and distributions within these "fingerprints." Our dataset included 30 different MPs. Directly analyzing the original data can be challenging and may obscure the underlying patterns. To address this, we used PCA, a method that reduces high-dimensional data into a more manageable, lower-dimensional space, while preserving as much valuable information as possible. PCA achieves this by transforming the original data into a new set of uncorrelated variables called principal components. The first principal component captured the maximum variance in the data, and the subsequent components captured decreasing amounts of variance. This allowed PCA to highlight the most substantial patterns or relationships in the data. PCA can help eliminate noise and redundancy by reducing the dimensionality of the data, making it easier to visualize and analyze the data and detect underlying structures. PCA is widely used in various fields, including

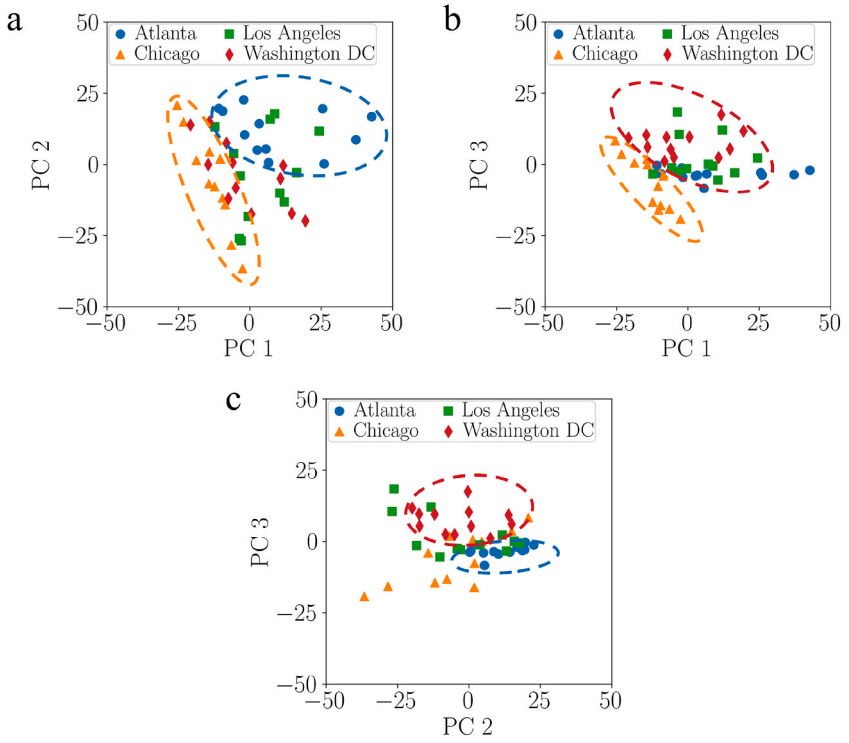

Fig. 3. Principal component analysis of microplastic distribution in soda beverages from different cities.

image processing, genetics, finance, and natural language processing, to obtain insights and simplify complex datasets. Here, instead of directly using the data from 30 distinct MPs, we further refine our analysis by categorizing these MPs into four specific size ranges: 20–30 μm, 30–50 μm, 50–100 μm, and 100–500 μm. This enhances the complexity of the "fingerprint" for PCA, effectively increasing the vector count. Utilizing these four size categories instead of a single size range expanded the number of data points from 30 to 120 per sample. This allows the option of using selected size ranges in PCA, such as choosing the smallest and largest sizes for better differentiation. This methodology has the potential to yield a more effective differentiation and precise analysis of the MP profiles of the samples.

After applying PCA to the "fingerprint" vector of each sample, the first three major principal components were selected for analysis. The selection was based on the ability to retain most of the original information. The variance explained by these components was substantial; the first accounted for 37.11 %, the second for 35.68 %, and the third for 11.06 %, summing up to 83.85 %. This high percentage of explained variance suggests that the three components are sufficiently representative of the underlying trends in the data. We had the flexibility to select any two-component combination of the three principal components for our plots. Initially, these principal components were used to identify the city-specific characteristics of the samples. In Fig. 3a–c, the data points are organized based on the purchase locations of sodas. Fig. 3a shows plots of the first and second principal components (PC1 and PC2) and that the samples from Atlanta and Chicago formed distinct clusters, each delineated by an ellipse. The distinct PCA boundaries suggest the possibility of classifying soda samples from specific city pairs, although not universally across all cities. Similarly, Fig. 3b, using the first and third principal components (PC1 and PC3), reveals two distinct, non-overlapping clusters for the samples from Chicago and Washington, DC. Fig. 3c, created using the second and third principal components (PC2 and PC3, respectively), displays separate clusters for the Atlanta and Washington, DC samples. However, in these visualizations, the data points representing Los Angeles were scattered and did not form distinct clusters. Subsequently, we used this methodology to analyze the characteristics of the container types in the samples. Fig. 4a–c shows the data points based on the container type. In all three figures, the samples for each container type are spread over a large area without the formation of clear, distinct clusters.

Our results indicate that soda samples can be more effectively classified by geographic location than by container type, highlighting the substantial influence of local environmental factors on MP composition. This pattern may stem from the soda-canning process, particularly if the beverages are produced in local factories. Although containers and syrup may be sourced from various locations, water, which is a critical component, is typically drawn from local sources. Therefore, the quality and treatment protocols of these local water sources are likely to substantially affect the MP content of the final product.

4. Conclusion

Our study confirmed that LDIR is effective in detecting up to 30 different types of MPs, both synthetic and natural, in soda. We used

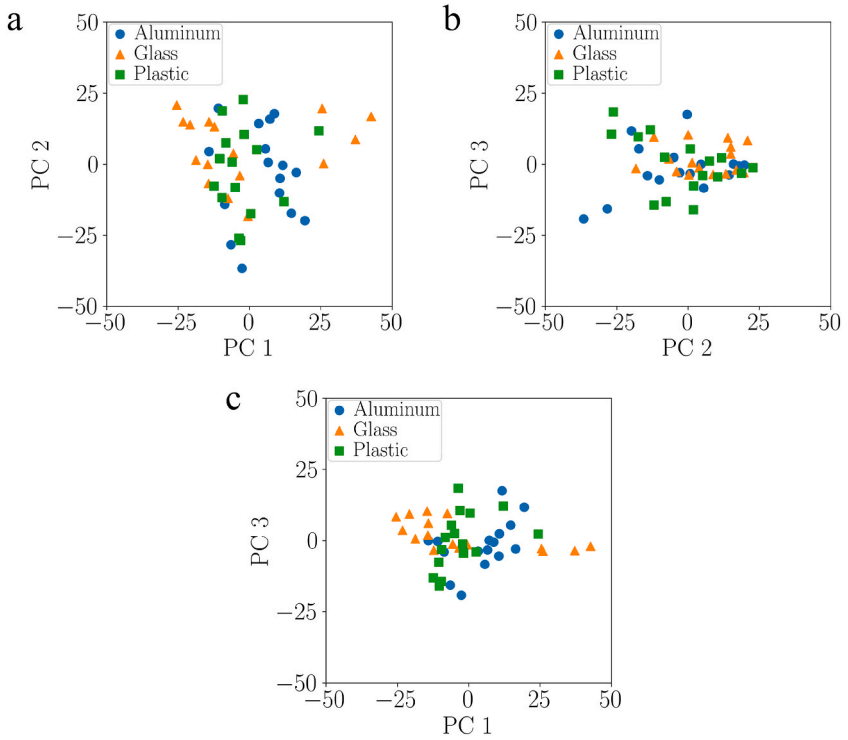

Fig. 4. Principal component analysis of microplastic distribution in soda beverages with different container types.

LDIR to examine the MPs in sodas sourced from four distinct US cities packaged in three different container types. The detected MPs provided valuable information about the soda production process and the quality of the raw materials used. Our analysis revealed variations in MP levels across different cities, with Los Angeles showing the lowest MP levels, indicating potentially stricter MP control in its manufacturing processes. Moreover, when comparing MP levels in different container types, we observed that plastic bottles, within their shelf life, tended to have similar or lower MP levels than that of glass and aluminum containers. This indicates that plastic bottles did not significantly contribute to an increase in MP levels during the intended usage period. In addition, the unique MP distribution patterns observed in different cities and container types serve as distinct "fingerprints that are invaluable for data analysis. Through PCA, we isolated three major principal components, demonstrating their utility in highlighting city-specific characteristics in the samples, although they were less effective in differentiating between container types. This suggests that the MP profile is more closely linked to the city of origin than to the container type, likely because of the reliance of the soda industry on local water sources. The water quality and treatment significantly influenced the MP composition of the final product. The dataset from this study offers considerable potential for further studies, such as examining temporal variations or expanding the geographical scope. Our findings make a valuable contribution to global efforts to reduce plastic pollution. These insights are crucial for developing effective strategies to reduce MP contamination, thereby aiding the broader goal of environmental conservation.

Funding statement

This research did not receive any specific grant from funding agencies in the public, commercial, or not-for-profit sectors.

Data availability statement

Data will be made available on request.

CRediT authorship contribution statement

Yuqi Wang: Writing – original draft, Visualization, Methodology, Investigation, Formal analysis, Data curation. **Yunxiang Wang:** Writing – review & editing, Validation, Resources, Project administration.

Declaration of competing interest

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

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.heliyon.2024.e32805.

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