Water Research 242 (2023) 120243

Contents lists available at ScienceDirect

Water Research

journal homepage: www.elsevier.com/locate/watres

Plastic bottles for chilled carbonated beverages as a source of microplastics and nanoplastics

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ARTICLE INFO

Keywords:
Microplastics
Nanoplastics
Pressure
Bubbles
Freeze-thaw cycles

ABSTRACT

Carbonated beverages are characterized by low temperatures, multiple microbubbles, high pressure, and an acidic environment, creating ideal conditions for releasing contaminants from plastic bottles. However, the release patterns of microplastics (MPs) and nanoplastics (NPs) are poorly understood. We investigated the effects of plastic type, CO_2 filling volume, temperature, sugar content, and additive on the leakage of MPs/NPs and heavy metals. Our results showed that polypropylene bottles released greater MPs (234±9.66 particles/L) and NPs (9.21±0.73 × 10^7 particles/L) than polyethylene and polyethylene terephthalate bottles. However, subjecting the plastic bottles to 3 repeated inflation treatments resulted in 91.65-93.18% removal of MPs/NPs. The release of MPs/NPs increased with increasing CO_2 filling volume, driven by the synergistic effect of CO_2 bubbles and pressure. After 4 freeze-thaw cycles, the release of MPs and NPs significantly increased, reaching 450±38.65 MPs and 2.91±0.10 × 10^8 NPs per liter, respectively. The presence of sugar leads to an elevation in MPs release compared to sucrose-free carbonated water, while the addition of additives to carbonated water exhibits negligible effects on MPs release. Interestingly, actual carbonated beverages demonstrated higher MPs concentrations (260.52±27.18-281.38±61.33 particles/L) than those observed in our well-controlled experimental setup. Our study highlights the non-negligible risk of MPs/NPs in carbonated beverages at low temperatures and suggests strategies to mitigate human ingestion of MPs/NPs, such as selecting appropriate plastic materials, high-pressure carbonated water pretreatment, and minimizing freeze-thaw cycles. Our findings provide insights for further study of the release patterns of the contaminants in natural environments with bubbles, pressure, low temperature, and freeze-thaw conditions.

1. Introduction

When walking down a hot summer street, the oppressive heat seems endless, as if the earth is thirsty and crying out for water. Even breathing feels hot, and sweat drips down the neck and back. But a refreshing glass of ice-cold carbonated beverage can offer a welcome respite. The cold liquid bursts with a dessert-like sweetness and a rush of cooling bubbles that erupted in the mouth, delivering exhilaration. However, while enjoying these drinks, have you ever considered the potential release of microplastics (MPs) and other pollutants, which could pose a health risk?

MPs, defined as plastic particles smaller than 5 mm in diameter (Weithmann et al., 2018), pose potential threats to human health when ingested. *In vitro* experiments have demonstrated MPs' ability to infiltrate human cell membranes, inducing the generation of reactive oxygen species, triggering inflammatory responses, and eliciting neurotoxic effects (Huang et al., 2021). These findings emphasize the urgent need for a comprehensive investigation into the release patterns of MPs originating from carbonated beverages.

Numerous studies have documented that high temperatures can cause plastic polymers to degrade and fracture, leading to the release of MPs into ordinary food and beverages (Astner et al., 2019; Ranjan et al., 2021). Nevertheless, it is critical to note that low temperatures can also alter the physical characteristics of plastics (Astner et al., 2019). For example, plastic becomes fragile when frozen (Fu et al., 2015), presenting a comparable possibility of MPs being released. Unfortunately,

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https://doi.org/10.1016/j.watres.2023.120243
Received 3 March 2023; Received in revised form 10 June 2023; Accepted 16 June 2023
Available online 17 June 2023

few studies have examined the pattern of MPs' release at low temperatures.

Carbon dioxide (CO_2) exhibits higher solubility at lower temperatures (Liger-Belair, 2019). The shaking when drinking carbonated beverages releases large amounts of CO_2 microbubbles. Despite their small size, these bubbles may generate destructive forces when they collapse. Submerged objects such as ship rudders or propellers are susceptible to severe corrosion or mechanical damage from cavitation erosion caused by bubble rupture (Kadivar et al., 2021). Moreover, MPs exhibit greater affinity with bubbles than with ice and water (Vega-Martínez et al., 2020), enabling their transportation from surfaces to solutions via bubbles. Consequently, the release of MPs should also be considered within the context of bubbles.

It is noteworthy that the release of CO_2 from carbonated beverages causes an indirect increase in pressure in plastic bottles. Approximately 0.2 MPa of pressure exists inside a chilled carbonated beverage bottle (Kuntzleman and Sturgis, 2020); however, higher gas pressure (0.5 MPa) can cause severe cracks in the container's physical structure (Xie et al., 2017). Observations have revealed that a crack could be formed under low stress for a long time and slowly grow until brittle failure (Juan et al., 2020). These results indicate that the inner walls of plastic bottles are susceptible to leaking significant quantities of MPs into the carbonated beverages due to indirect pressure increases by the release of CO_2. Yet, this potential pressure effect has been overlooked in past studies. Considering that high concentrations of CO_2 released from the carbonated beverages not only scour the plastic bottle's inner wall but also increase the pressure inside, it is particularly important to examine the combined effect and their interaction on the MPs' release.

Dissolved CO_2 in carbonated beverages also creates a low pH environment. Prior research has shown that heavy metal leaching from plastic additives is strongly pH-dependent, with the maximum amounts of the examined elements detected in an acidic environment (Krol et al., 2020; Saria et al., 2006). The mobility of heavy metals was also accelerated on acidic occasions (pH≅ 2) (Saria et al., 2006). Therefore, the acidity of carbonated beverages may affect the concentration of heavy metals released from plastic bottles.

In addition to MPs, the overlooked issue of nanoplastic (NPs) release from plastic bottles deserves attention. NPs, denoting plastic particles with minute dimensions (<1 μm) (Gigault et al., 2021), are ingested by individuals through a variety of consumer products. Remarkably, a single cup of beverage has been found to contain an astonishing quantity of 3.1 billion NPs (Hernandez et al., 2019), while disposable food containers have been observed to harbor approximately 1 million NPs (Liu et al., 2022). Importantly, NPs possess a higher potential to harm human health compared to MPs (Rist et al., 2018), as they can effectively permeate biological membranes (Wu et al., 2019). Research findings indicate that exposure of human hematopoietic cells to NPs can instigate cellular oxidative stress and induce primary DNA damage (Rubio et al., 2020). Therefore, it is imperative to consider the release of NPs alongside MPs.

The quantification of MPs and NPs includes the utilization of both mass-based methodologies, which determine particle quantities based on their mass, and number-based methodologies, which assess particle abundance. However, to acquire comprehensive information regarding the size, morphology, concentration, and composition of MPs/NPs, a synergistic integration of multiple techniques is indispensable, as elucidated in the accompanying Text S1.

This study evaluated the impact of plastic types, CO_2 filling volumes, storage temperature, freeze-thaw cycles, sugar content, and additives on the release of MPs/NPs and heavy metals from plastic bottles containing carbonated beverages. The investigation included an assessment of the individual and combined effects of pressure and CO_2 bubbles on the release of MPs/NPs induced by CO_2 filling. We aimed to assess the environmental risks from the release of contaminants such as MPs/NPs and heavy metals in carbonated beverages and to investigate the characteristics and patterns of MPs/NPs release in the unique carbonated beverage environment, providing important insights to reduce their risk to human health.

2. Materials and methods

2.1. Collection of plastic bottles

Beverage storage bottles with a total volume of 500 mL made of polyethylene (PE), polypropylene (PP), and polyethylene terephthalate (PET) were procured from Suzhou Qiaojing Trading Ltd. We chose these bottle types based on their widespread use in beverage packaging (Changwichan and Gheewala, 2020; Poortinga and Whitaker, 2018). The chemical composition of the bottles was confirmed using micro-Fourier transform infrared spectroscopy (μ-FTIR) spectrometer (Nicolet iN10, Thermo Fisher Scientific, USA), and the corresponding spectra are shown in Fig. S1.

2.2. Different treatment of plastic bottles

The pristine plastic bottles were subjected to a rinsing procedure with ultrapure water, following the guidelines outlined in the general specification for rinsing-filling-capping machines of plastic bottles (General Administration of Quality Supervision, 2011), to replicate the pre-treatment of plastic bottles prior to their incorporation in an authentic production process. Subsequently, the bottles were carefully air-dried in an inverted position to prevent the ingress of airborne contaminants. The detailed treatment procedures are delineated in Fig. 1a.

To investigate the impact of plastic bottle types on MPs/NPs' release, 450 mL of ultrapure water at 4°C was poured into PE, PP, and PET bottles, and 3.6 g/L of CO_2 was injected into them using a soda machine (COCOSODA, Dongguan Linuo Electric Co., China) to simulate carbonated beverages (Liger-Belair, 2019; Liger-Belair et al., 2015). A filter membrane (MEC membrane, 25 mm diameter, 0.22 μm pore size, BKMAN) was installed between the plastic tube and the glass tube to prevent MPs from the CO_2 and plastic tube entering the bottle during inflation (Fig. 1a). The plastic bottles were refrigerated at 4°C for 12 h to mimic chilled beverage storage and then agitated in a shaker at 110 rpm for 15 min to replicate consumer behavior while consuming drinks. The period of 15 min was chosen because most consumers prefer to enjoy their drinks within 15 min of receiving their drinks (Ranjan et al., 2021). Moreover, we conducted repetitive CO_2 inflation experiments (1-3 times) using the same plastic bottles to examine the release pattern of MPs.

We also examined the effect of CO_2 filling volume (0 g/L, 3.6 g/L, 5.4 g/L, 7.2 g/L) and storage temperature (-18°C, 4°C, 25°C) on the release of MPs/NPs from PET bottles under the conditions described above. Since carbonated beverages may freeze again after being thawed according to most people's consumption habits, experiments were conducted with 1-4 freeze-thaw cycles at -18°C for 8 h and 25°C for 16 h.

The effect of bubbles or pressure on the release of MPs/NPs from plastic bottles was further investigated using a custom setting, as shown in Fig. 1b. There were two hoses inserted into the bottle's cap, one connecting to a valve that controlled the inhalation of CO_2 and the other to the atmosphere directly or to a pressure gauge. Two treatments were implemented. For the first one, the PET bottle contained 450 mL ultrapure water, and the right hose was connected to the atmosphere directly to allow the pressure inside and outside the bottle to be balanced, while the left valve was opened to inject 3.6-7.2 g/L CO_2. This treatment simulated the impact of CO_2 bubbles in the absence of pressure. For the second one, the PET bottle was empty, and the right hose was connected to a pressure gauge (YN-60 1MPa, Yueqing Brady Instrument Factory), while the left hose injected the same volume of CO_2 as the above. Then 450mL ultrapure water was poured into the plastic bottle after refrigeration at 4°C for 12 h. This treatment replicated the impact of pressure without CO_2 bubbles.

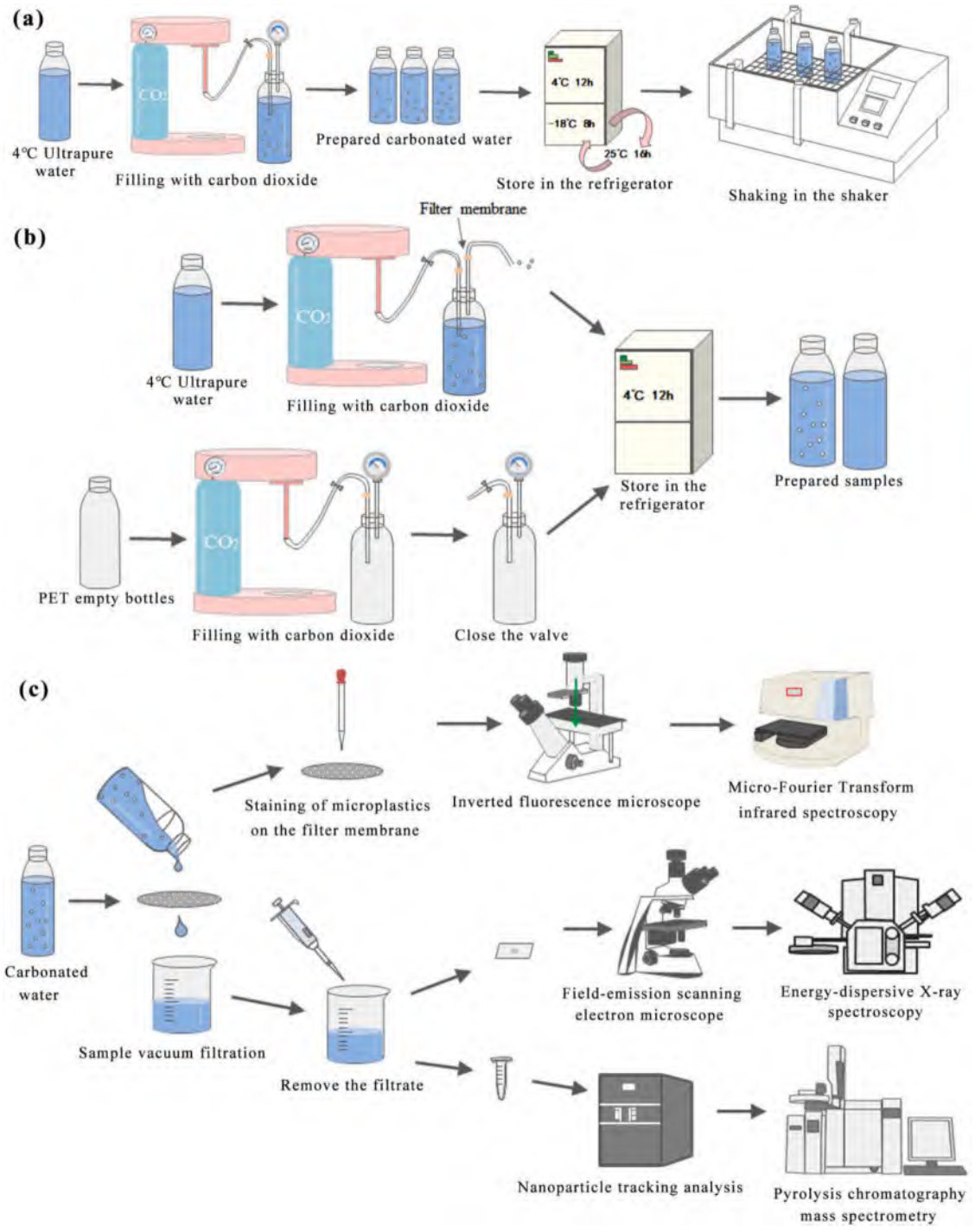

Fig. 1. Diagram showing (a) the preparation of various treatments of carbonated water, (b) the methodology used to determine the effect of bubbles or pressure on the release of MPs, and (c) the steps involved in the extraction, identification, and quantification of MPs.

To assess the influence of constituents (food additives, and sugar) in carbonated beverages (General Administration of Quality Supervision, 2008) on the release of MPs, we examined several frequently used food additives, namely sucralose, acesulfame, sunset yellow, and carmine. The concentrations of these additives were determined based on the maximum allowable levels specified in the relevant standard (Commission, 2022), with concentrations set at 0.25 g/L, 0.3 g/L, 0.1 g/l, and 0.05 g/l, respectively. The effect of sugar on MPs release from plastic bottles was investigated by adding sucrose (reagent grade, product no. V900116, Sigma-Aldrich) at concentrations of 0, 30, 60, and 90 g/L, representing typical sugar contents in carbonated beverages (Wierzejska, 2022). To eliminate any potential confounding effects originating from MPs present in the sucrose itself, the sugar-laden water underwent exclusive filtration prior to the experiment (see Text S2).

Finally, the quantities of MPs present in two distinct commercial brands of carbonated beverages were meticulously determined, aiming to evaluate the potential risks associated with MPs release in real-world beverages.

A triplicate of all treatments was performed, and the water was then analyzed to determine the number of MPs/NPs and heavy metals that might have leached into the liquid.

2.3. MPs extraction, identification and quantification

In this study, particles of two sizes, MPs (1 μm-5 mm) and NPs (< 1 μm) were considered. To identify MPs, we implemented Nile Red staining in conjunction with μ-FTIR spectrometer (Nicolet iN10, Thermo Fisher Scientific, USA), while the analysis of NPs involved the utilization of field-emission scanning electron microscope (FESEM, ZEISS Sigma 300, Germany) combined with energy-dispersive X-ray spectroscopy (EDS) mapping, as well as nanoparticle tracking analysis (NTA) (NanoSight NS300, Malvern Panalytical, England) in conjunction with pyrolysis gas chromatography/mass spectrometry (Py-GC/MS) (PY-3030D, Frontier Lab, Japan /7890B-5977A, Agilent, USA).

To ensure the accuracy of the methodologies for detecting MPs and NPs, we conducted validation experiments using two positive control samples. These samples consisted of predetermined quantities of blue 30 μm polystyrene (PS) beads (product no. 84135, Sigma-Aldrich) and red 200 nm uniform PS microspheres (product no. 43302, Sigma-Aldrich). The recovery rates for the 30 μm PS beads, as determined by Nile Red staining and μ-FTIR analysis, were found to be 89.4±3.51% and 92.6 ±4.47%, respectively, with a percentage difference of 3.46±4.29% observed between the groups. Additionally, the combination of FESEM and EDS mapping confirmed the absence of any dust contamination in the samples, thereby yielding a recovery rate of 81.5±6.97% for the 200 nm PS microspheres. Similarly, NTA analysis yielded a recovery rate of 85.6±6.82% for the 200 nm PS microspheres, while Py-GC/MS analysis confirmed the composition of the sample as PS. The percentage difference between the FESEM and NTA groups was determined to be 4.91 ±6.73%. These positive control experiments demonstrated acceptable levels of quality (80-110%) throughout the experimental procedures (Bannick et al., 2019).

The detailed extraction, identification, and quantification procedures are presented in Fig. 1c. An entire bottle of water was vacuum filtered through a polycarbonate track-etched filter membrane (PCTE, 25 mm diameter, Whatman) with a pore size of 1 μm to minimize detection errors of MPs. Furthermore, in cases where the sample contains sugar, it was rinsed multiple times with deionized water to eliminate any influence from the sugar component. To address the possibility of additives particles exhibiting spectra similar to those of MPs, a washing step employing 30 mL of ethanol (high-performance liquid chromatography grade, product no. 64175, Sigma-Aldrich) was conducted to mitigate the interference from such particles, as reported by Li et al. (2022). Then Nile Red staining was used to identify the MPs retained on the membranes using a method modified from Erni-Cassola et al. (2017). A higher Nile Red solution of 10 μg/mL, compared to Erni-Cassola et al. (2017) was prepared by dissolving Nile Red (technical grade, product no. N3013, Sigma-Aldrich) in methanol to improve the hue. After adding approximately 0.05 mL of Nile Red solution to each filter, the filters were kept dark at 60°C with coverslips for 30 min. The stained filters were viewed using an inverted fluorescence microscope (Nikon, Ti2-E, 4x, Japan) under green light (460-550 nm). The fluorescent images were used to quantify the particles using the Image J software, and triplicates were analyzed for each treatment. Following Nile Red, a comprehensive analysis was conducted on 1/8th of the filter area using the μ-FTIR spectrometer in visual mode. FTIR spectra were recorded in transmittance as described in our previous study (Luo et al., 2023). The spectra thus obtained were compared to those in a standard IR library (KnowItAll IR library), and those displaying a similarity of more than 85% were identified as MPs (Fig. S2). Results from Nile Red and μ-FTIR were compared. If the percentage difference was below 3.46%, the average value became the final count for MPs. Otherwise, the experiment was repeated.

The number of NPs present in the leachates was estimated using a FESEM (ZEISS Sigma 300, Germany), following the protocol described by Hernandez et al. (2019). To achieve a homogeneous dispersion of NPs, the filtrate was supplemented with 10 mg/L of sodium dodecylbenzene sulfonate (SDBS) (technical grade, product no. 25155-30-0, Sigma-Aldrich). Furthermore, to prevent the formation of coffee rings during the drying process, the leachates were carefully deposited onto silicon wafers in 10 μL aliquots. The drops were incrementally added until a final volume of 200 μL was reached, ensuring the uniform dispersion of NPs on the silicon substrate (Fig. S3). Subsequently, SEM imaging was performed after the samples were coated with aurum. A magnification (50000 ×) was taken to view the NPs (Fig. S4a) and then counted by Image J software. Considering the relatively simple composition of our experimental samples, devoid of complex organic constituents, the potential influence stemming from dustfall is the primary factor to be considered. The EDS mapping was performed on the same samples to differentiate between NPs and any potential dust contaminants based on elemental composition. Inclusion of samples containing carbon and oxygen, while lacking magnesium (Mg) and calcium (Ca), was done for NPs enumeration purposes (Fig. S4b and S4c).

Additionally, the quantification of NPs in the leachates was also conducted using the NTA technique. Specifically, 10 mL of the filtrate was carefully pipetted, and the addition of 10 mg/L SDBS ensured the uniform dispersion of NPs. NTA measurements were then performed to count the NPs. Subsequently, the chemical composition of the particles in the filtrate was determined by utilizing Py-GC/MS equipment (Fig. S5). The quantification results obtained from FESEM and NTA techniques were compared. If the percentage difference between the two methods was less than 4.91%, the average value was used as the final count of MPs. Otherwise, the experiment was repeated for improved accuracy and reliability.

Given the inherent challenges associated with maintaining stringent quality control measures (refer to Text S3), the quantification of NPs in the leachate resulting from additive treatment and in actual carbonated beverages was not determined.

2.4. Analysis

A scanning electron microscope (SEM, Tescan Mira Lms, Czech) was utilized to observe the morphological characteristics of the inner surfaces of plastic bottles. Additionally, 3D images of these surfaces were captured using atomic force microscopy (AFM, Bruker Dimension Icon, Germany). The analysis of heavy metals was described in Text S4.

2.5. Quality assurance and quality control

To prevent contamination from MPs and NPs, rigorous measures were implemented throughout our experimental procedures. For a comprehensive overview of these measures, please refer to Text S5.

2.6. Statistical analysis

Utilizing SPSS 27.0, statistical analysis was conducted to examine the correlation between various heavy metal concentrations in the leachate, the detail can be seen in Text S6.

3. Results and discussion

3.1. The effect of plastic bottle types on the release of MPs and NPs

The results presented in Fig. 2a reveal that the number of MPs released from different plastic bottle types varied significantly after a single inflation experiment ($F=1165.36$, $p < 0.001$). PP bottles had the highest number of MPs released (234±9.66 particles/L), followed by PET (161±14.94 particles/L), and the lowest in PE (68±6.20 particles/L). A higher release of MPs from PP compared to PET was also observed by Mortula et al. (2021). The relatively low release of MPs from PE bottles may be attributed to the highly stable secondary carbon in the carbon chain of PE material (Gewert et al., 2015).

Similarly, NPs followed the same pattern as MPs, with PP bottles exhibiting the highest number of NPs released ($9.21\pm0.73 \times 10^7$

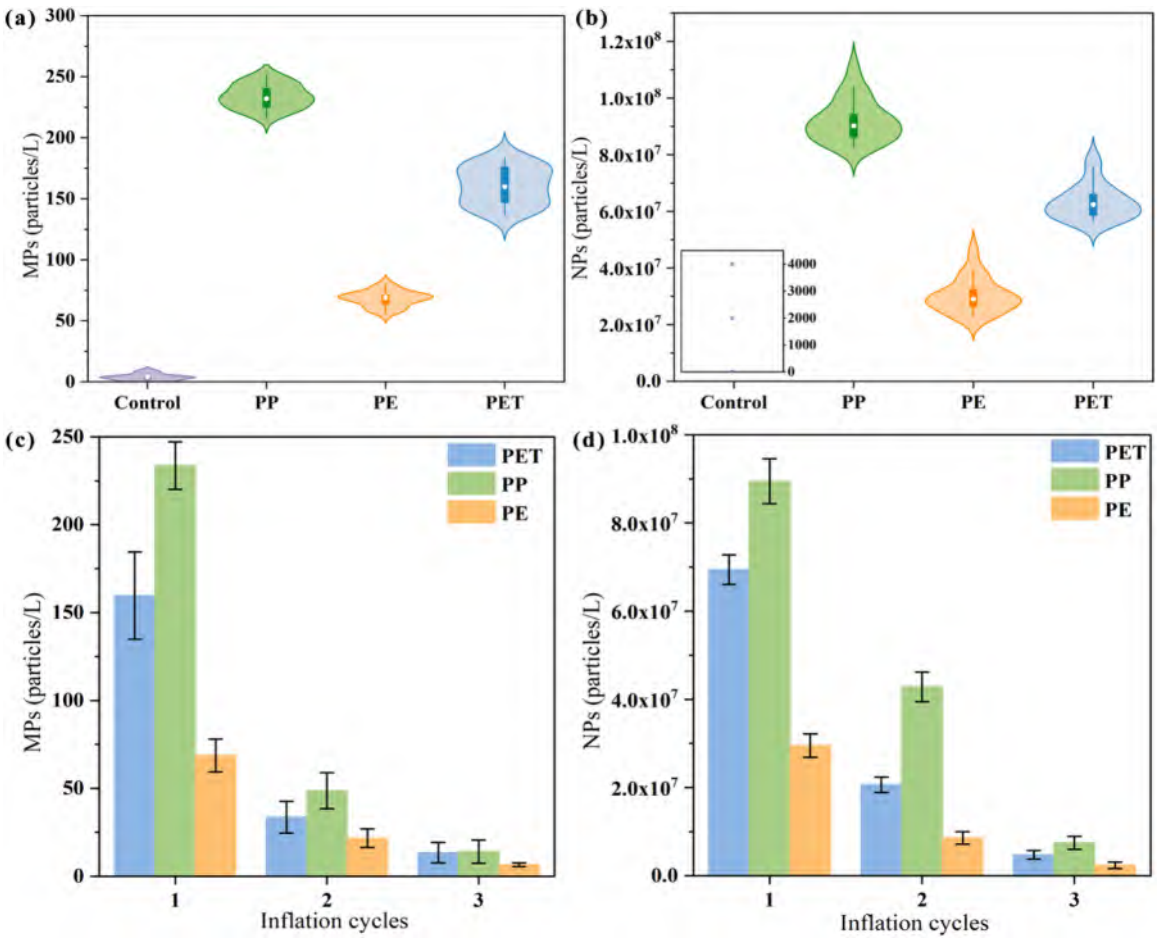

Fig. 2. Number of particles released from different types of plastic bottles after inflation experiments. (a) MPs and (b) NPs after one inflation (4°C, 3.6g/L CO_2, n=21), and (c) MPs and (d) NPs after 1-3 times inflation (4°C, 3.6g/L CO_2, n=21).

particles/L), PET bottles showing an intermediate release (6.32±0.60 × 10^7 particles/L), and PE bottles having the least NPs released (3.02 ±0.59 × 10^7 particles/L) (F=464.62, P < 0.001) (Fig. 2b). Therefore, it is essential for beverage manufacturers to carefully choose the plastic material for their bottles to reduce the health risks posed by MPs, particularly NPs. Notably, the number of NPs was approximately six orders greater than that of MPs, probably due to the further dissociation of MPs (Mitrano et al., 2021; Sharma et al., 2022). These findings highlight that the consumption of carbonated beverages leads to a greater intake of NPs than MPs. It should be noted, however, that most publications do not consider NPs (Table S2), despite the fact that they are capable of causing greater environmental harm and human toxicity (Dong et al., 2022; Gaylarde et al., 2021; Yin et al., 2021).

Fig. 2c and 2d depicts the amounts of MPs and NPs released after additional inflation tests. Of particular interest was that a second inflation treatment resulted in significant reductions of MPs (78.9-79.2-68.4%) and NPs (70.3-52.1-71.0%) in PET, PP, and PE bottles (Fig. 2c and 2d). These reductions are much higher than those reported by Zhou et al. (2023) for washing plastic cups, suggesting that carbonated water is more effective than ultrapure water in removing MPs/NPs from plastic bottles. Moreover, after 3 repetitions of the inflation test, the removal of MPs/NPs from all plastic bottles reached 91.65-93.18%. These findings indicate that high-pressure carbonated water treatment (similar to repeated inflation experiments) before use can effectively minimize the health risks associated with their ingestion.

To identify the cause of this reduction in MPs/NPs release, SEM was used to characterize the inner surfaces of the bottles before and after three repeated inflation experiments (Fig. 3). Images taken at high resolution revealed that the original PP and PET plastic bottles had a large number of particles attached to the inner wall (Fig. 3a and 3e), while the original PE bottles had uneven folds and films (Fig. 3c). This difference might be related to the various plastic bottle production methods. PET and PP plastic bottles are both made by injecting molten masterbatch into molds under pressure, whereas PE plastic bottles are created by blowing plastic blanks and adhering to the interior of the mold and then demolding them after cooling (Du et al., 2020). In this case, the plastic particles may break apart into finer granules during the pressurization process and adhere to the surface of the plastic bottles, resulting in that pristine bottle being the significant source of MPs and NPs. Despite being rinsed with water before use, a substantial number of MPs and NPs were still released (Fig. 2a and 2b). Fortunately, after three inflation treatments, the inner walls of the bottles became smooth and clean (Fig. 3b, 3d, and 3f), and the removal of MPs/NPs reached 91.65-93.18%. (Fig. 2c and 2d).

3.2. *The effect of CO_2 filling volume on the release of MPs and NPs*

The CO_2 filling volume also affected the release of MPs/NPs. As observed in Fig. 4, increasing CO_2 filling volume significantly enhanced their release (MPs: F=339.16, P < 0.001; NPs: F=2205.33, P < 0.001). A possible explanation for this variation was that bubbles could generate stress waves that travel through solid materials when they rupture (Haosheng and Shihan, 2009), resulting in plastic particles adhering to the bottle's inner surface falling out. Considering the high affinity of bubbles for plastic (Liger-Belair, 2019; Zhang et al., 2022), these bubbles will adhere to the MPs/NPs on the inner wall and then be transferred to

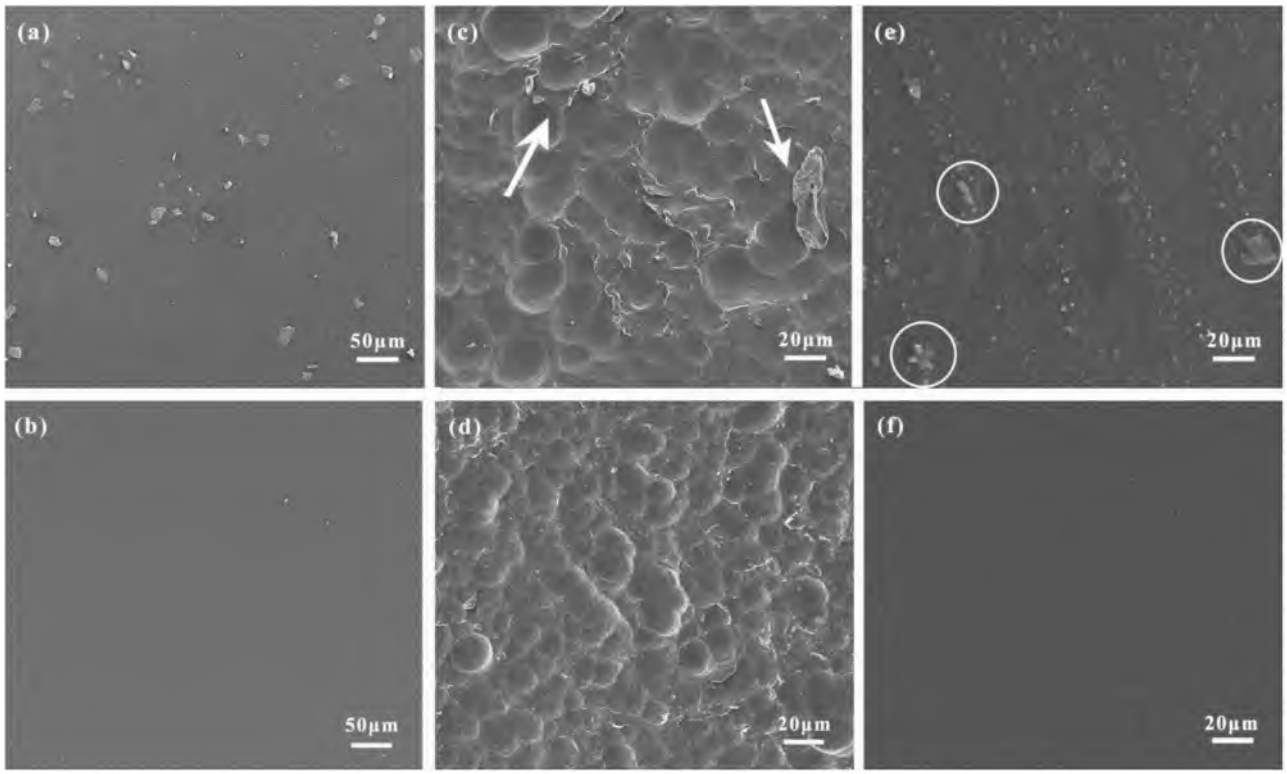

Fig. 3. SEM images of the inner surface of pristine bottles: (a) PP (c) PE, and (e) PET, and after 3 times inflation experiments: (b) PP, (d) PE, and (f) PET.

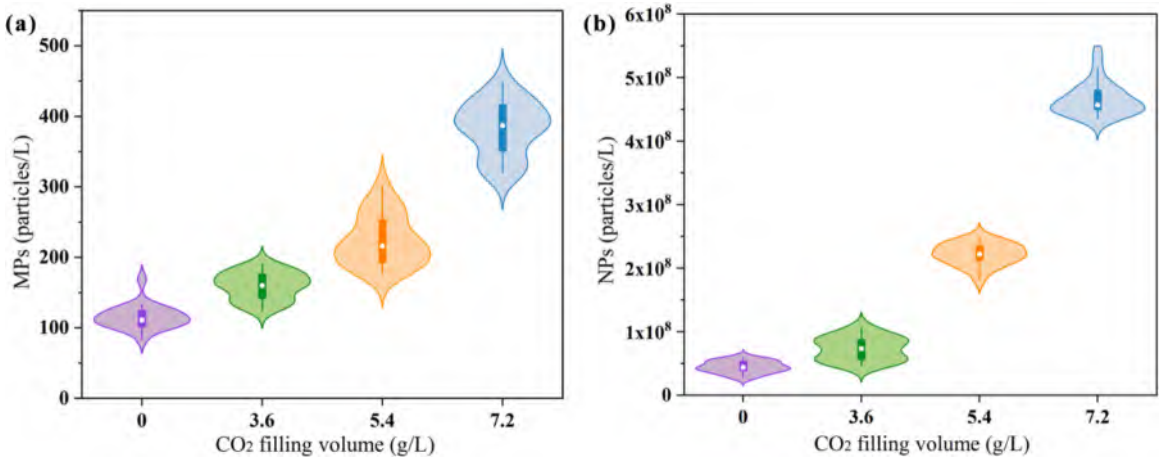

Fig. 4. Number of particles released from different CO_2 filling volume after inflation experiments. (a) MPs and (b) NPs after one inflation (4°C, 3.6g/L CO_2, n=21).

the liquid (Kim et al., 2020; Masry et al., 2021). Obviously, the increased filling volume caused a tremendous amount destructive CO_2 to be released, leading to higher MPs/NPs numbers. Additionally, another part of the bubbles would burst on the surface of the attached MPs, producing finer NPs. This might explain why the number of NPs increased with increasing inflation. The destruction of the bubbles continues until they are destroyed (Xu et al., 2021). In a solution, the bubbles interact, combine to form more giant bubbles, disperse, and finally cease to form. However, the bubbles on the walls continued to grow for a long time (Vega-Martínez et al., 2020), resulting in the continuous release of MPs/NPs while consumers drink carbonated beverages.

In addition to the cavitation caused and disturbance by bubbles on the bottle's inner surfaces, a pressure effect was also involved in drinking carbonated beverages. The pressure inside the bottle presented a significant increase with increasing CO_2 filling volume ($F=640.43$, $p < 0.001$) (Fig. 5a). At a filling amount of 7.2 g/L; the pressure reached 0.281 MPa, equivalent to 2.8 atmospheres (Fig. 5a). It was thus clear that the pressure of CO_2 on the inner walls of PET bottles cannot be ignored.

To quantify the influence of bubbles and pressure on the release of MPs, we conducted two separate treatments, and the results are presented in Fig. 5b. In the pressure-only experiment, 142±8.34 particles/L MPs and $4.94\pm0.22 \times 10^7$ NPs were observed, while in the bubble-only experiment, 118±9.66 particles/L MPs and $4.27\pm0.39 \times 10^7$ particles/L NPs were detected (Fig. 5b). Our findings indicated that bubbles and pressure, as indirect factors induced by CO_2 injection, significantly contribute to the release of MPs/NPs. However, to the best of our knowledge, the individual effects of these factors on MPs and NPs release have not been evaluated in previous studies. Therefore, a

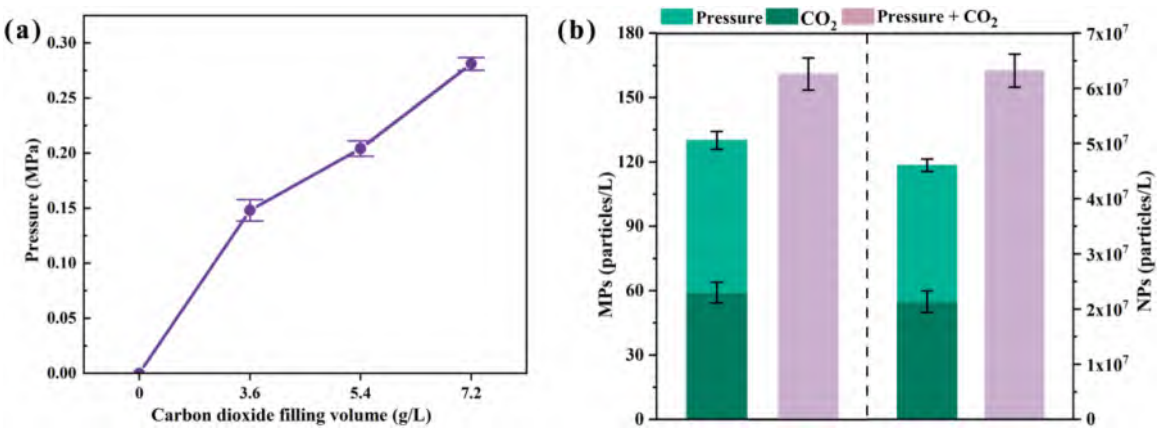

Fig. 5. The effect of different carbon dioxide filling volumes on the release of MPs/NPs was investigated. (a) The magnitude of pressure varies when plastic bottles are filled with different volumes of carbon dioxide. To investigate the effect of pressure and bubbles on the release of MPs/NPs, (b) the number of MPs/NPs released under single and combined factors was counted.

comprehensive investigation on the effects of bubbles and pressure on MPs release in other occasions is necessary in the future, given their ubiquity in the environment. For instance, in shallow-water substrates, the absence or reduction of dissolved oxygen concentration can lead to the activation of anaerobic processes, which generate deoxidized gases such as methane, hydrogen sulfide, and ammonia (Golosov et al., 2012; Joye et al., 2004). This process can result in the generation of bubbles, leading to the weathering of plastics in sediments and the production of MPs/NPs. On the other hand, water pressure increases at a rate of 0.1 MPa per 10 m depth as seawater depth increases, reaching up to approximately 20 MPa at a depth of 2000 m (Kobayashi et al., 2021). This significant pressure can cause the decomposition of plastic products and result in the release of MPs/NPs in the deep sea.

It should be noted that the combined effect of pressure and bubbles was greater than that of pressure or bubbles alone, as indicated by the significantly higher release of MPs and NPs (MPs: $F=69.44$, $p<0.001$; NPs: $F=116.43$, $p<0.001$) (Fig. 5b). These results suggest that bubbles and pressure have a synergistic effect on the release of MPs/NPs from plastic bottles. The significance of this finding extends to environmental settings, particularly in anaerobic zones and natural gas seeps in the deep-sea where high-pressure and bubble conditions may lead to substantial release of MPs/NPs, posing adverse impacts on marine

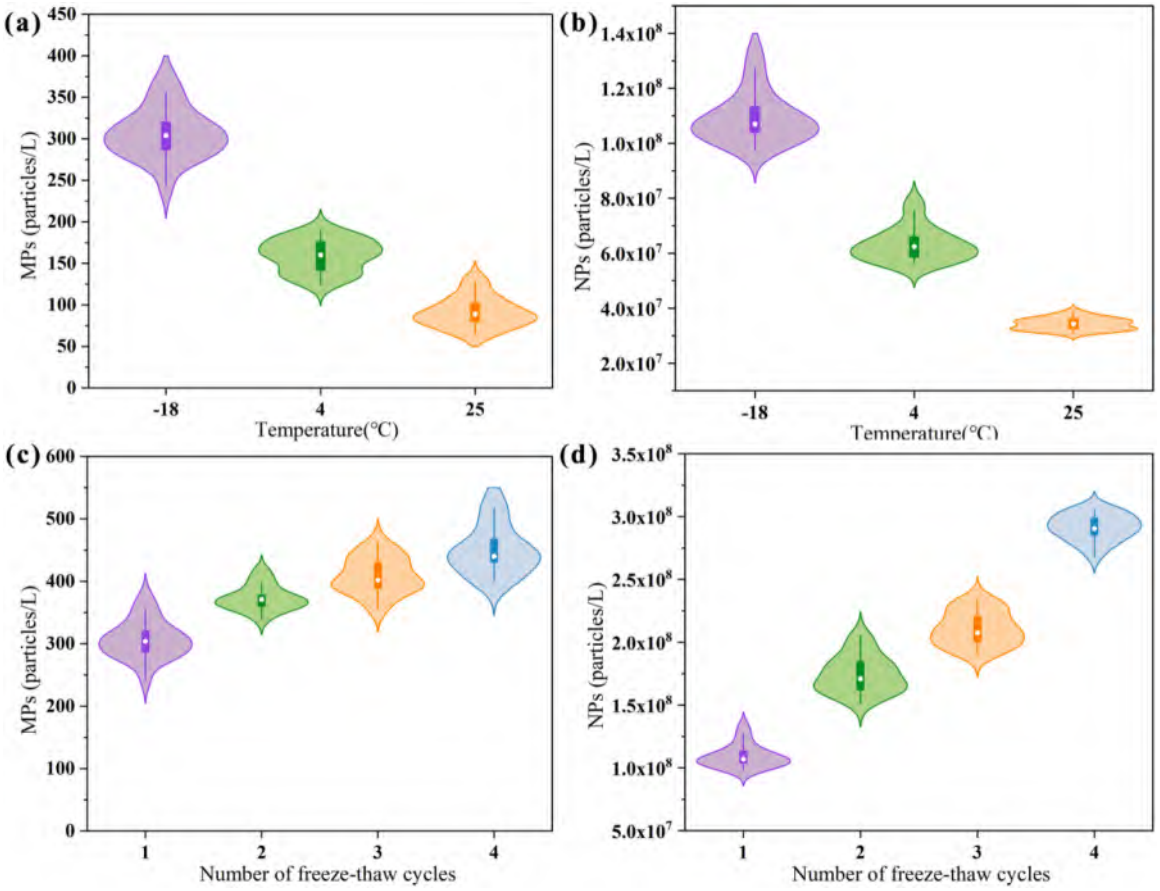

Fig. 6. Released MPs (a) and NPs (b) at varying temperatures and subjected to 1-4 freeze-thaw cycles (c and d) (PET bottles, 4°C, n=21).

ecosystems and ultimately on human health.

Additionally, the presence of CO_2 in carbonated beverages confers an acidic nature that trigger heavy metal leaching (Fig. S6a), with the concentrations of Cr, Ni, Cu, Zn, Cd, Pb, and Mn significantly increasing as pH decreases. Of note, the concentration of Zn was the highest (125.94 ppb), likely due to its extensive use as a plasticizer, stabilizer, and fungicide in plastic additives (Chubarenko, 2022; Kobayashi et al., 2021). Moreover, a noteworthy correlation was found among heavy metal leaching (Fig. S6b), highlighting that the investigation of heavy metal leaching from plastic products should not be restricted to a single type.

3.3. The effect of temperature on the release of MPs and NPs

The effect of storage temperature on MPs/NPs release was investigated. The results revealed a significant positive correlation between decreasing temperature and increased release of MPs/NPs (MPs: $F=456.05$, $p < 0.001$; NPs: $F=700.69$, $p < 0.001$). Specifically, the release of MPs was 307 ± 30.39, 159 ± 18.98, and 91 ± 17.74 particles/L at -18°C, 4°C, and 25°C, respectively (Fig. 6a). The release of NPs was similar, with values of $1.09 \pm 0.09 \times 10^8$, $6.32 \pm 0.60 \times 10^7$, and $3.43 \pm 0.21 \times 10^7$ particles/L at the respective temperatures (Fig. 6b). In contrast, Zhou et al. (2023) found that a low temperature of 5°C did not promote the release of MPs compared to room temperature, which was inconsistent with our findings. This discrepancy might be due to the indirect effect of pressure generated by CO_2 in our experiments. The pressure inside the bottle at 4°C was higher than at 25°C (Fig. S7), which likely contributed to the elevated release of MPs/NPs.

At a temperature of -18°C, carbonated beverages were observed to freeze and experienced a 40% increase in volume (Fig. S8). This expansion potentially resulted in plastic cracking and delamination (Chubarenko, 2022). Additionally, the low temperature and ice-rich environment restricted molecular mobility, causing the plastic to become more brittle, and increasing the likelihood of plastic fragmentation and MPs/NPs release (Fig. 7b) (Zheng et al., 2020). The pressure inside the bottle prior to melting was recorded as zero, however, after complete melting the pressure reached 0.162 MPa, which is likely due to CO_2 bubble release. Therefore, the combined influence of CO_2 bubbles, pressure, and the ice-rich environment contributed to the elevated release of MPs/NPs at -18°C, making it the condition with the highest release rate. The results of SEM and AFM tests confirmed that the freeze-thaw treatment led to the rupture of the plastic bottles. The inner surface of the original bottles was relatively clean and smooth, with most features less than 3 nm in roughness (Fig. 7a), whereas the surface subjected to one freeze-thaw cycle showed the formation of fissures with roughness around 6.5 nm and a length of about a dozen micrometers (Fig. 7b). This was in agreement with previous findings that reported a rougher surface texture of high-density polyethylene bags after a "cold" storage treatment (Hee et al., 2022).

It is evident that plastic containers release a large quantity of MPs/NPs when subjected to high temperatures, with concentrations ranging from 78-16.1 $\times 10^{11}$ particles/L (Table S2). Our investigation report that the levels of MPs/NPs released in low temperatures range from 68-4.66 $\times 10^8$ particles/L, a magnitude that should not be overlooked. These results underscore the need for further research into the effects of low temperature on the release of MPs, as the current literature regarding this topic is limited (Table S2).

Common consumption practices can lead to the repeated freezing and thawing of carbonated beverages. To study the impact of these conditions, we performed experiments with 1 to 4 freeze-thaw cycles. Our results, shown in Fig. 6c and 6d, indicated a significant increase in the release of MPs and NPs from plastic bottles as the number of freeze-thaw cycles increased (MPs: $F=81.92$, $P<0.001$; NPs: $F=797.40$, $P<0.001$). After 4 cycles, the release of MPs and NPs reached 450 ± 38.65 and $2.91 \pm 0.10 \times 10^8$ NPs per liter, respectively. Additionally, more pronounced cracks were observed on the inner wall of the bottles with a mean roughness of 12.7 nm and a root mean square roughness of 15.6 nm (Fig. 7b and 7c). Therefore, consumers are advised to reduce the number of freeze-thaw cycles when consuming such carbonated beverages to avoid excessive intake of MPs and NPs.

In conclusion, repeated freeze-thaw processes accelerated the aging and degradation of plastic, leading to an increase in MPs/NPs abundance and a decrease in particle sizes. The expansion of ice crystals during freezing causes friction against the plastic and subsequently disrupts the polymer molecular chemistry, which is a potential mechanism for the subsequent release of MPs/NPs during thawing (Koutnik

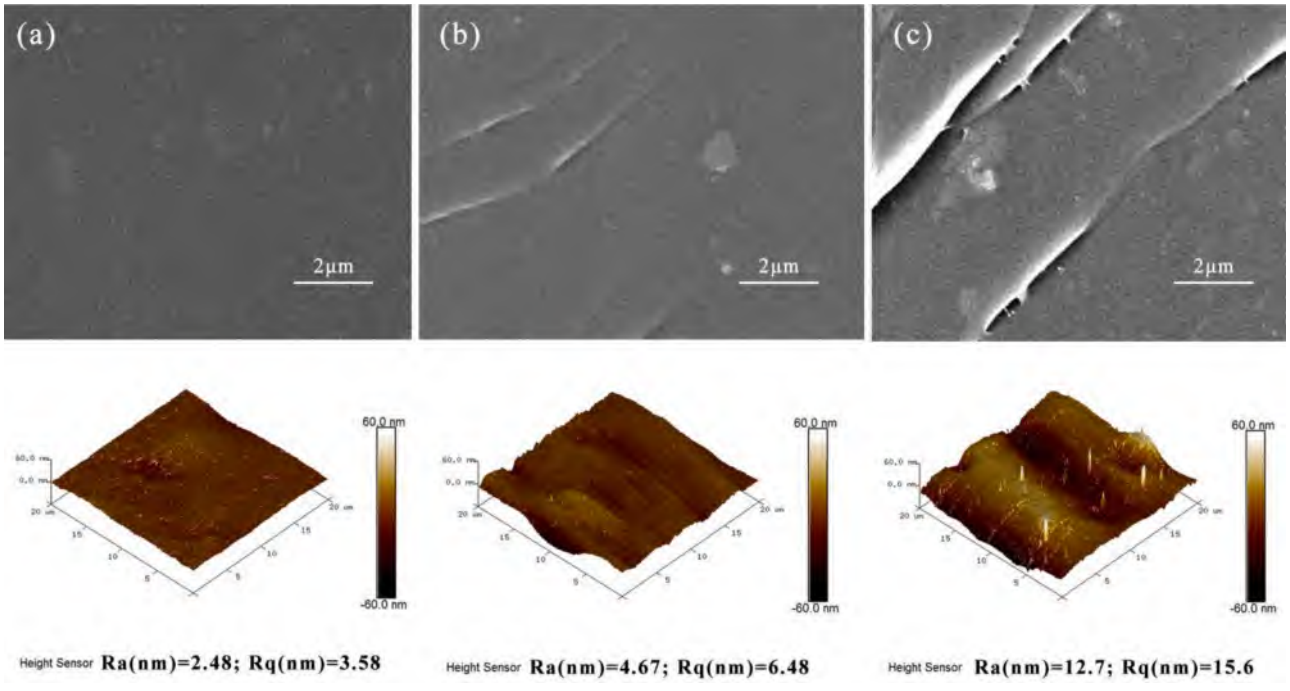

Fig. 7. SEM and AFM 3D microscopic of the inner surface of PET bottles subjected to 0 (a), 1(b), and 4(c) freeze-thaw cycles.

et al., 2022). The freeze-thaw cycle is a very common phenomenon at high latitudes, high altitudes and in some temperate regions, with approximately 55% of the total land area in the Northern Hemisphere experiencing annual freeze-thaw (Wang et al., 2017). However, the duration time, intensity, and frequency of freeze-thaw cycles are likely to increase with global climate change. Given that freeze-thaw cycles are widespread on Earth's terrestrial surface, this presents a significant risk to ecosystem security and human health by contributing to the release of MPs into the environment.

3.4. The effect of sugar and food additives on the release of MPs

The addition of sugar to carbonated water yielded a considerable release of MPs in the range of 179 ± 14.18-218±12.32 particles/L (Fig. 8a). Moreover, an upward trend in MPs release was observed with increasing sugar content, although the trend exhibited a moderate degree of significance. The presence of sugar (90 g/L) led to a rise of approximately 36.57% in MPs release compared to carbonated water without sugar. The incorporation of sugar enhanced the viscosity of the solution, impeding bubble coalescence (Zhou et al., 2021) and promoting the entrapment of MPs on the inner surfaces of plastic bottles, ultimately facilitating their release into the solution (Henry and Craig, 2009).

The introduction of additives into carbonated water yielded a range of MPs concentrations from 157.81±12.58 to 164.24±13.73 particles/L (Fig. 8b). Importantly, no statistically significant differences were observed in the MPs content between carbonated water samples with various additives and those without additives (MPs: F=0.825, P=0.512). It is noteworthy that the relatively low dosage of additives, compared to CO_2 and sugar, limited their influence on the release of MPs.

In the final phase of our study, we selected two brands of carbonated beverages to investigate the release of MPs in these genuine market products. The outcome unraveled notable quantities of MPs, with concentrations measuring 281.38 ± 61.33 and 260. 52± 27.18 particles/L, respectively (Fig. 8c). These values considerably surpass the maximum releases observed in our well-controlled experimental setup, exhibiting an augmentation ranging from 19.58% to 78.30%. The actual composition of carbonated beverages is notably more intricate and might harbor substantial concentrations of MPs, as corroborated by our preliminary experiments, where sugar water at a concentration of 90 g/L exhibited levels of 36 ± 1.48 particles/L, consistent with the findings reported by Afrin et al. (2022). Moreover, the handling and storage of actual carbonated beverages, involving repeated shaking during transportation and recurrent freeze-thaw cycles, may potentiate the liberation of MPs within the actual beverages. Unfortunately, due to the challenges associated with ensuring stringent quality control measures (see Text S3), data on NPs in actual carbonated beverages were not assessed. However, drawing from the evidence furnished in this study, it is justifiable to infer that the presence of NPs within actual carbonated beverages warrants careful consideration and cannot be deemed negligible.

4. Conclusion

Our study reported that the levels of MPs/NPs released in iced carbonated beverage bottles range from 68-4.66 × 10^8 particles/L, potentially posing health risks to humans. PP bottles released more MPs/NPs than PET and polyethylene PE bottles. Following 3 repetitions of the inflation test, the removal of MPs/NPs from all plastic bottles reached 91.65-93.18%. These results suggest that beverage manufacturers could mitigate the ingestion of MPs/NPs in chilled carbonated beverage bottles by choosing appropriate plastic materials or by adopting techniques such as high-pressure carbonated water pretreatment.

Our study also revealed, for the first time, that indirect variables such as CO_2 bubbles and pressure, caused by CO_2 filling, significantly increased the release of MPs and NPs from plastic bottles. Furthermore, the combined effect of these variables was greater than their individual effects, providing cues for further investigation of MPs/NPs release behavior in natural environments characterized by high pressure and bubbles, such as the bottom of deep water.

In addition, our research found that repeated freeze-thaw cycles (from -18°C to 25°C) considerably amplified the release of MPs and NPs, attributable to the combined impact of CO_2 bubbles, pressure, and the ice-rich environment. Thus, it is recommended that consumers limit the number of freeze-thaw cycles when consuming carbonated beverages to minimize their ingestion of MPs and NPs. Moreover, considering the widespread occurrence of freeze-thaw cycles on Earth's terrestrial surface, this phenomenon may accelerate the aging and degradation of plastics in the environment, thereby posing potential hazards to ecosystem security.

The influence of sugar on the release of MPs is significant and should not be underestimated. Conversely, the presence of additives in carbonated water has a negligible impact on the release of MPs due to their low concentrations. Importantly, our study highlights that actual beverages exhibit a higher release of MPs compared to our experimental results, emphasizing the need for further investigation and control of MPs release in real-world scenarios.

In conclusion, our study enhanced the understanding of the risks associated with the release of MPs and NPs from carbonated beverage bottles, and offered strategies for reducing the intake of MPs in carbonated beverages. Furthermore, our study could inspire researchers to explore further the release patterns and effects of natural environments, such as air bubbles, pressure, low temperature, freeze-thaw, on the release of MPs and NPs.

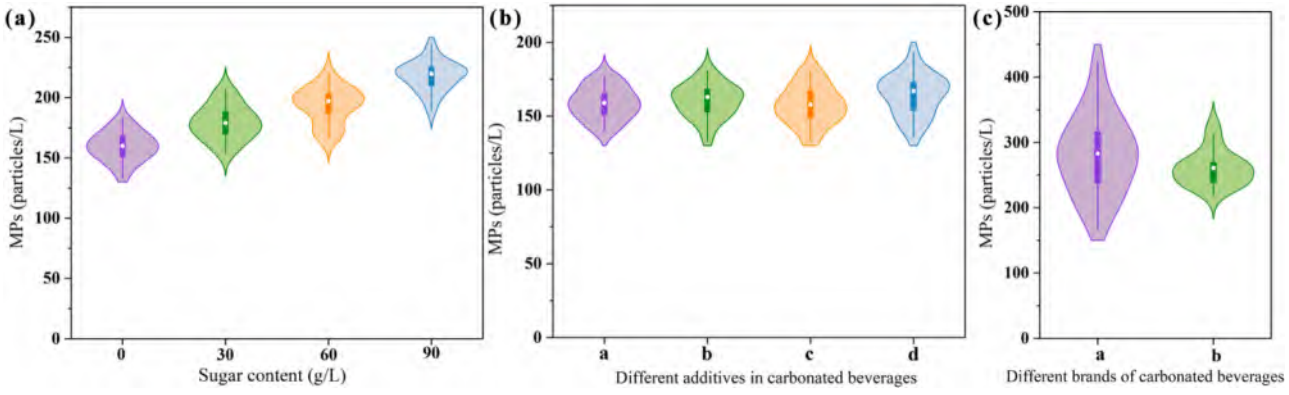

Fig. 8. Number of MPs in (a) carbonated waters containing sugar, (b) carbonated waters containing food additives, "a-d" represent sucralose, acesulfame, sunset yellow and carmine, respectively (PET bottles, 4°C, n=21) and (c) actual carbonated beverages (n=21, "a" stands for the brand 1 beverages; "b" stands for the brand 2 beverages).

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.

Acknowledgements

We are grateful for the financial support of Science and Technology Project of Changsha City (KQ2107018), Natural Science Foundation of China (51908561), Natural Science Foundation of Hunan Province of China (2021JJ31162), Educational Commission of Hunan Province of China (22A0172, 22B1084), Hunan Students' platform for innovation and entrepreneurship training program (S202210538077).

Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.watres.2023.120243.

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