Evaluation of Bis (2-ethylhexyl) Phthalate (DEHP) in the PET Bottled Mineral Water of Different Brands and Impact of Heat by GC–MS/MS

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Received: 23 March 2022 / Accepted: 18 May 2022 / Published online: 7 June 2022
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Abstract
Phthalates are one of the ubiquitous contaminants in the environment due to their extensive use in the last few years. They are easily released because they are not chemically bonded to polymers. They migrate into the food during food packing while in water, they migrate during water filling or storage and bottle manufacturing. They are toxic to human health and known as carcinogen/endocrine disruptors. A total of sixty PET (polyethylene terephthalate) bottled mineral water samples of different six brands were purchased from the local market of Noida, India. These bottles were of two different batch numbers of each brand. Two bottles of each brand with a different batch number were analyzed immediately after purchase while the other eight bottles were analyzed after 2 and 6 months when they were stored in sunlight (~45 °C) and —20 °C. The aim of the present study was to determine the migration of DEHP and its impact on storage conditions of PET bottled mineral water in retail stores or homes. Gas chromatography-mass spectrometry (GC–MS/MS) was used for the estimation of DEHP in these samples. The author observed that the migration of DEHP was dependent on high temperature and storage time. DEHP was present only in those samples, which were stored in sunlight for 2 & 6 months and at —20 °C for 6 months. While found below the detection limit in those samples which were analyzed immediately after purchase and stored at —20 °C for 2 months.

Keywords Bis (2-ethylhexyl) phthalate (DEHP) · PET (polyethylene terephthalate) · Bottled water · Gas chromatography-mass spectrometry (GC–MS/MS)

1 Introduction
Phthalates (an ester of ortho-phthalic acid) are a group of aromatic chemicals that contain a phenyl ring with two attached acetate groups. They are colorless, odorless liquids which do not evaporate easily [1]. They are manufactured chemicals used to make plastics soft and more flexible. They can be removed easily from plastic resources because the covalent bond is weak between the phthalate and their parent things [2–7] after that they migrate and pollute water, soil, air, beverage, and food products [8–10]. Kouhpayeh et al. [11] stated that phthalate esters are the priority pollutants which were categorized by the US Environmental Protection Agency (USEPA) and several other agencies. According to Singh and Li [12], phthalates are plasticizers and softening additives in the manufacture of plastic goods.

Bis (2-ethylhexyl) phthalate is known as diethyl hexyl phthalate (DEHP) and it is the main compound of PET bottled materials which migrates from the PET bottle to the mineral water [13]. It is the most important congener and is about 50% of the world’s production of phthalates [14]. It is also known as a rodent liver carcinogen [15]. It can enter into the environment through releases from factories that make or use it. It can also enter into the environment through air, water, and household items containing it. A large amount of plastic that contains DEHP is buried in landfill sites. It has been found in groundwater near waste disposal facilities. It usually attaches strongly to the soil while dissolves very slowly into underground water or surface water. The level of DEHP in indoor air could be higher than the outdoor air when coatings and flooring were installed recently in a room.

Phthalates are present in plastic products such as toothbrushes, wall coverings, furniture upholstery, toys, paints, drugs, adhesives, inks, packaging film and sheets, sheathing for wire and cable, medical tubing and blood storage bags, etc. [16–18]. The use of phthalates in plastic products, cosmetics, and personal care products makes the presence
of phthalate in water, wastewater, and soil [19, 20]. They migrate in a low amount into the food, drinking water, and beverages from the packaging or manufacturing processes or bottling materials and this is the most probable route of exposure of phthalate. They can enter into the body through food, drinking water, air, skin contact, and medicines. Blood transfusion and kidney dialysis are the other routes of phthalate exposure. Phthalate effluent from the wastewater treatment plant is the main source of phthalate in the aquatic environment [21, 22].

Phthalate may cause developmental toxicity and reproductive effects in animals. They have carcinogenic and teratogenic effects [23, 24]. They exhibit low toxicity with short-term exposure while exhibiting high toxicity with long-term exposure. Short-term exposure may cause nausea, and mild gastrointestinal disturbances while long-term exposure may cause cancer, damage to the liver and reproductive system [25]. In children, Santana et al. [26] reported higher metabolites of phthalates due to the daily intake of phthalates. Due to the exposure of phthalate obesity and insulin resistance may occur in children [27–30]. The USEPA and EU (European Union) have banned phthalate in toys and children’s articles [31]. Several researchers studied the toxic effect of phthalate on human health and stated that the migration of phthalates from PET bottles has been a matter of concern globally. Exposure to phthalates may cause several diseases in adults like low gestational age of foetus, delayed appearance of pubarche in girls, endometriosis, undescended testes, reduced anogenital distance, decrease in sperm count or its quality, increase in sterility and testicular cancer, impairs cholesterol metabolism in hepatic stellate cells, maternal glucose disorders, infant cryptorchidism, cardiovascular (CVD) and urological disease [12, 32–35]. They are known as hormonally active agents (HAAs) or endocrine-disrupting chemicals (EDCs) because they affect the hormonal system or the endocrine system of the body [36]. Over the past few decades, phthalate is responsible for the endocrine-mediated adverse effects which were observed in humans and wildlife [37].

Due to the size, strength, ease of transport, and low cost of production the most widespread water bottling material is PET [38]. Bolgar et al. [39] stated that the plastic material used to manufacture bottles consists of some additives such as catalysts, accelerators, stabilizers, coupling agents, and antioxidants. PET consists of terephthalate subunits and ethylene glycolate, linked via ester bonds [40]. In the last few decades, the consumption of PET bottled mineral water has increased and has effectively replaced tap water in several developed countries [41–43]. Li et al. [44] stated that China was the largest consumer of PET bottled water with a market of 14.7 billion dollars in 2013. As per the study of IBWA [45], the utilization of bottled water has increased in India from 4553.3 to 6492.7 million gallons in the period 2014 to 2019. PET bottled water has emerged as drinking water, which preserves the original purity of natural mineral water. In many areas where there is a lack of portable public water, PET bottled water is the only water available for human consumption [46].

The packaging of the bottle acts as a gas barrier to avoid interaction with the adjacent environment [47]. However, the presence of oxygen at higher temperatures promotes thermo-oxidative and thermo-mechanical reactions which enhance the migration of phthalate from PET bottled to the water [48–50]. In the presence of oxygen, DEHP can be broken down into carbon dioxide and other simple chemicals in water and soil with the help of microorganisms [51]. The concentration of DEHP in PET bottled mineral water may vary with pH [52], storage time [53, 54], storage temperature [55, 56] and exposure to sunlight [57]. Suits and Hsuan [58] described PET degradation by thermal-oxidation, hydrolysis, and photo-oxidation, which are initiated in the presence of UV light (Fig. 1).
Carbon dioxide, carbon monoxide, glycerol, 1,4-dioxane, p-carboxy benzoic acid, and esters are formed after the thermal oxidation of PET. p-carboxy benzoic acid and glycerol are formed after the hydrolysis of PET. Carbon dioxide, carbon monoxide, anhydride, esters, and p-carboxy benzoic acid are formed after the photo-oxidation of PET. Ros-Chumillas et al. [59] studied the shelf life of PET bottled water and stated that the shelf life of multilayer PET bottles can reach 300 days at 4 °C and 250 days at 25 °C while it was 180 days at 4 °C and 160 days at 25 °C for simple monolayer PET bottles.

The purpose of this study was to estimate the DEHP migration in the PET bottled mineral water which was stored at different temperatures for a different time. GC–MS/MS was used for the estimation of DEHP. To the best of information, this study is the first study to estimate the migration of DEHP under normal conditions, freezing conditions (−20 °C), and sunlight (~45 °C) in the period of July 2018 to December 2018. Besides the estimation of DEHP other emerging pollutants like microplastics should be analysed in the future. These particles are ubiquitous and may be harmful to living organisms [60]. The size of these particles is ranging from 1 to 5 mm in length and can be classified as primary microplastics and secondary microplastics.

## 2 Materials and Methods

### 2.1 Sampling

The author purchased a total of sixty PET bottled mineral water samples of different six brands (Aquafina, Bisleri, Catch, Divyajal, Kinley, and Railneer) from the local market of Noida, India. The author purchased ten water bottles of each brand with two different batch numbers. All PET bottled water samples were contained in their original sealed containers. The sampling details are shown in Table 1. The first two water bottles of each brand with a different batch number were analyzed immediately after purchase. The other four water bottles of each brand with a different batch number were stored in the refrigerator (at ~20 °C) for 2 months and 6 months, respectively. While another four water bottles of each brand with a different batch number were stored in sunlight (at ~45 °C) for the same time.

### 2.2 Equipment and Reagents

GC–MS/MS (Model No.: 7000C, Agilent), HP-5MS column (30 m length × 0.25 mm width × 0.25 μm film thickness), rotary evaporator (Model No.: R 300, Buchi), UV–Visible spectrophotometer (Model No.: 1800, Shimadzu), pH meter (Model No.: LMPH-10, Labman), TDS meter (Model No.: S-945, Systonic), conductivity meter (Model No.: S-941, Systonic), and electronic weighing machine (0.01 mg to 220 g, Sartorius) were used for this study.

DEHP standard was purchased from Sigma-Aldrich (CAS No.: 117–81-7). Dichloromethane (CAS No.: 75–09-2) and hexane (CAS No.: 92112–69-1) were procured from Fisher Scientific. Hydrochloric acid (HCl) (CAS No.: 7647–01-0), sodium chloride (NaCl) (CAS No.: 7647–14-5), sodium hydroxide (NaOH) (CAS No.: 1310–73-2), and potassium chloride (KCl) (CAS No.: 7447–40-7) were purchased from Thermo Fisher Scientific India Pvt. Ltd. Methyl anthranilate (CAS No.: 134–20-3), silver nitrate (AgNO3) (CAS No.: 7761–88-8), and potassium chromate (K2CrO4) (CAS No.: 7789–00-6) were purchased from Merck. Sulfanilic acid (CAS No.: 121–57-3) was procured from TCI Chemicals India Pvt. Ltd. Silica gel of mesh size (100/200) (CAS No.: 112926–00-8), and sodium sulphate (CAS No.: 7757–82-6) were purchased from Loba Chemie Pvt. Ltd. Prior to use, sodium sulfate was baked at the 400 °C for 4 h and silica gel was triggered at the 130 °C for 16 h. Double distilled water was used throughout the study.

### 2.3 Chemical/Instrumental Analysis

Parameters such as odour, taste, pH, TDS (Total Dissolved Solids), conductivity, and chloride were analyzed by the Indian Standard (IS): 14,543 [61]. Nitrate was analyzed by the method of Narayana and Sunil [62]. DEHP was analyzed by the method of Bhardwaj and Sharma [1]. Odour and taste were analyzed by observation. pH, TDS, conductivity, nitrate, and DEHP were analyzed by their instruments while chloride was analyzed by the titrimetric method.

### Table 1 Identification of Samples with Batch No. and Year of Packaging

| S. no | Brand name | Batch no          | Packing year |
|-------|------------|-------------------|--------------|
| 1     | Aquafina   | 7476D12J18        | 2018         |
|       |            | 7476D18J18        |              |
| 2     | Bisleri    | 297               |              |
| 3     | Catch      | 100W008           |              |
|       |            | 100W154           |              |
| 4     | Divyajal   | UP0330133         |              |
|       |            | UP0330134         |              |
| 5     | Kinley     | H30E8E11          |              |
|       |            | H30E8E12          |              |
| 6     | Railneer   | 2310              |              |
|       |            | 2311              |              |
2.3.1 pH

The pH was measured by using a pH meter. The instrument was calibrated with the buffer solutions of pH 7, 4, and 9.2. The electrode of the instrument was immersed in the sample and held for some time to achieve a stabilized reading. After each analysis, the electrode was cleaned with distilled water to avoid contamination between samples.

2.3.2 TDS (mg/l)

TDS was measured by using a TDS meter. The instrument was calibrated with a standard solution of NaCl (1000 mg/l). The electrode of the instrument was immersed in the sample and held for some time to achieve a stabilized reading. After each analysis, the electrode was cleaned with distilled water to avoid contamination between samples.

2.3.3 Conductivity (µS/cm)

Conductivity was measured by using a conductivity meter. The instrument was calibrated with a standard solution of KCl (750 mg/l). The electrode of the instrument was immersed in the sample and held for some time to achieve a stabilized reading. After each analysis, the electrode was cleaned with distilled water to avoid contamination between samples.

2.3.4 Chloride (mg/l)

Chloride was analyzed by the titrimetric method. 20 ml of each sample was taken into a 250 ml conical flask. 1 ml of K₂CrO₄ indicator was added then the sample was titrated with 0.0282 N AgNO₃ solution till the colour changes from yellow to brick red. The consumed volume of AgNO₃ was noted. A blank solution was prepared and analyzed in the same manner. Chloride was calculated by the formula given below:

\[
\text{Chloride (mg/l)} = \frac{(V_s - V_B) \times N \times 35.45 \times 1000}{\text{Volume of sample taken}}
\]

2.3.5 Nitrate (NO₃) (mg/l)

Nitrate was analyzed by the spectrophotometric method. The standard solutions of different concentrations (0.26–10.7 mg/l) were prepared. 10 ml of each sample was taken into a 250 ml conical flask. 1 ml of 0.5% sulfanilic acid and 1 ml of 2 M HCl were added then shake the solution for 5 min to allow the diazotization reaction. 1 ml of 0.5% methyl anthranilate and 2 ml of 2 M NaOH were added to form an azo dye. The volume made up to 10 ml with distilled water. The absorbance was taken of the red colour dye at 493 nm.

\[
\text{Nitrate (mg/l)} = \text{Reading of the instrument (mg/l)} \times \text{dilution factor}
\]

2.3.6 DEHP (µg/l)

2.3.6.1 Standards and Sample Preparation (Extraction and Cleanup) DEHP was extracted from the PET bottled mineral water samples by using the method USEPA-3510B [63] with slight modification. The standard solutions of different concentrations (0.1–50.0 µg/l) were prepared from the stock solution (1000 µg/l) in hexane. All solutions were kept in the dark at 4 °C until analysis. The bottled water sample was homogenized manually and then half liter of the sample was taken into a glass separatory funnel of one liter. 60 ml of dichloromethane was added into the separatory funnel and then the sample was shaken for 1–2 min. After releasing the pressure, the funnel was kept for 10 min to separate the organic layer from the aqueous phase. The organic layer of dichloromethane was filtered through the Whatman No.-41 filter paper in 250 ml of the RB (round bottom) flask. This procedure was done two more times and the solvent extract was collected in the same flask. Then the collected solvent was evaporated up to 2 ml at 45 °C by using the rotary evaporator and the final residue was ready for the cleanup process.

The final residue solution was cleaned by using the method USEPA-3630C [64]. A chromatographic glass column (250 mm length × 10 mm width) was prepared with some portion of silica gel (5 g) & sodium sulfate (2 g). The column was preconditioned with the solvent mixture (hexane: dichloromethane, 1:1, v/v). The final residue solution (2 ml) was then transferred onto the chromatographic column. Hexane (50 ml) was added onto the glass column and then eluted out into an RB flask of 250 ml. This process was repeated three more times and the eluted solvent was collected in the same flask. All the eluted solvent was concentrated at 45 °C by using a rotary evaporator and then was re-dissolved in 1 ml of hexane. The sample was ready for the estimation of DEHP by GC–MS/MS.

2.3.6.2 GC–MS/MS Analysis The final sample solution (1 µl) was injected into the GC–MS/MS. The separation was done with the help of the capillary column (HP-5MS). Helium was used as a carrier gas with a constant flow of 1 ml/min. The mode of injection was splitless and the inlet temperature was set at 280 °C with a pressure of 10.0 psi. The mass spectrometer was operated in the electron impact ionization mode with ionizing energy of 70 eV. The GC oven temperature was 160 °C, then increased at a rate of 20 °C/min up to 250 °C, again increased at a rate of 1.6 °C/min up to 270 °C which was maintained for 5 min. The total run time was
22.0 min. The detector temperature was set at 300 °C and the makeup gas was nitrogen. The ion source and transfer line temperature were set at 280 °C and 320 °C, respectively.

DEHP (µg/l) = Reading of the instrument (µg/l) × dilution factor.

2.4 Quality Assurance and Control

To minimize interferences, all glasswares were immersed in acetone for 30 min, rinsed with hexane, and then dried at 200 °C for 2 h. Each PET bottled water sample was examined three times and then the average value was taken. One blank sample with each set of water samples was processed. The calibration of the instrument was performed with a DEHP standard range from 0.1 to 50.0 µg/l and the linear correlation \((R^2)\) was > 0.99. The recovery of DEHP was found between 40 and 100%, which achieved the recovery limit of method USEPA-1699 [65]. The limit of detection (LOD) and limit of quantification (LOQ) were calculated based on the standard deviation of the response \((S_y)\) of the curve and the slope of the calibration curve \((S)\). The values of LOD & LOQ of the measured variables were calculated by the following formula: LOD = 3.3\((S_y/S)\), LOQ = 10 \((S_y/S)\), respectively. The values of LOD and LOQ of the measured variables are given in Table S2.

3 Result

Some physio-chemical parameters such as pH, TDS, conductivity, chloride, and nitrate \((NO_3^-)\) were measured with the estimation of phthalate (DEHP) in the PET bottled mineral water samples. These samples were agreeable in odour and taste in all the storage conditions. The results are shown in Tables 2, 3, 4, 5 & 6. The graphical representation of the changing of concentrations is shown in Figs. 2, 3, 4, 5, 6, 7.

3.1 Analysis After Purchase

The pH was in the range of 6.74–6.98. The TDS varied from 4.90–168.33 mg/l and the range of conductivity was found from 5.60–278.00 µS/cm. The chloride was in the range of 4.5–15.50 mg/l and nitrate was in the range of 2.78–27.12 mg/l. DEHP was found below the detection limit in all the samples.

3.2 Analysis after Two Months

The pH was in the range of 6.51–6.78 at ~45 °C and 6.73–6.96 at ~20 °C. The TDS was from 6.16–170.21 mg/l at ~45 °C and 4.88–168.10 mg/l at ~20 °C. The conductivity was in the range of 5.62–277.87 µS/cm at ~20 °C. The chloride was in the range of 4.00–15.00 mg/l at ~45 °C and 4.50–15.50 mg/l at ~20 °C. Nitrate varied from 2.98–28.86 mg/l at ~45 °C and 2.77–27.00 mg/l at ~20 °C. DEHP was found from 0.52 to 3.26 µg/l at ~45 °C and below the detection limit at ~20 °C.

3.3 Analysis After Six Months

The pH varied from 6.12–6.36 at ~45 °C and 6.57–6.81 at ~20 °C. The TDS was in the range of 8.28–180.45 mg/l at ~45 °C and 5.98–174.56 mg/l at ~20 °C. The conductivity was from 8.11–286.32 µS/cm at ~45 °C and 6.82–280.54 µS/cm at ~20 °C. The chloride was from 2.50–13.50 mg/l at ~45 °C and 4.00–15.00 mg/l at ~20 °C. Nitrate was in the range of 3.76–35.22 mg/l at ~45 °C and 3.12–28.10 mg/l at ~20 °C. DEHP was found from 1.10–5.39 µg/l at ~45 °C and 0.31–2.66 µg/l at ~20 °C.

4 Discussion

The result indicates that the value of EC, TDS, and \(NO_3^-\) is increasing in the PET bottled mineral water samples with the increase of temperature. While the value of pH and \(Cl^-\) is decreasing with the increase of temperature. The increase of TDS and EC may be due to the leaching of metals (inorganic compounds) and ions (organic compounds) from the PET bottle to the water. Hansen and Pergantis [66], and Fiket et al. [67] studied the leaching of metals from PET bottled to water and stated that it was the main reason to increase the TDS & EC in water. The decrease of chloride ions may be due to the chlorination reaction [68] and the decrease of pH is due to the oxidation of organic compounds through the photodegradation by sunlight [69]. Temperature influences on the leaching of organic and inorganic compounds from PET bottled to water [70, 71]. The author found the highest TDS, conductivity, and chloride in the Catch brand followed by Railneer, Bisleri, Divyajal, Kinley & Aquafina. The highest nitrate was found in the Railneer brand followed by Divyajal, Bisleri, Catch, Kinley, and Aquafina.

Jeddi et al. [72] analyzed PET bottled water samples immediately after purchase and stated that there was no significant relationship between the concentration of DEHP and the physicochemical properties of the water samples. While Dumitrascu [73] reported that the migration of DEHP from PET bottled to water depends on the pH. DEHP was the most abundant phthalate in the PET bottled water samples and acts as a carcinogenic agent [56, 74, 75]. Although due to analytical difficulties in achieving a low detection limit, there is a lack of data on the leaching of DEHP in PET...
**Table 2** Characteristics of the examined PET bottled water samples after purchase

| S. no | Parameters | Brands name with sample ID |
|-------|------------|-----------------------------|
|       |            | Aquafina | Bisleri | Catch | Divyajal | Kinley | Railneer |
|       |            | 7476D12J18 | 7476D18J18 | 297 | 341 | 100W008 | 100W154 | UP0330133 | UP0330134 | H30E8E11 | H30E8E12 | 2310 | 2311 |
| 1     | Odour      | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable |
| 2     | Taste      | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable |
| 3     | pH         | 6.94 | 6.98 | 6.77 | 6.74 | 6.89 | 6.90 | 6.91 | 6.96 | 6.92 | 6.90 | 6.84 | 6.80 |
| 4     | TDS (mg/l) | 5.00 | 4.90 | 85.30 | 88.45 | 165.78 | 168.33 | 68.60 | 72.40 | 22.80 | 20.40 | 129.60 | 130.90 |
| 5     | Conductivity (µS/cm) | 5.60 | 5.81 | 178.80 | 180.40 | 275.90 | 278.00 | 136.76 | 139.40 | 41.80 | 39.80 | 246.60 | 243.96 |
| 6     | Chloride (mg/l) | 4.50 | 5.00 | 7.50 | 8.00 | 15.00 | 15.50 | 7.00 | 7.50 | 5.00 | 4.50 | 9.50 | 10.00 |
| 7     | Nitrate (mg/l) | 2.78 | 3.02 | 15.56 | 16.04 | 4.65 | 4.87 | 18.43 | 18.68 | 4.22 | 4.56 | 26.87 | 27.12 |
| 8     | DEHP (µg/l) | < BDL | < BDL | < BDL | < BDL | < BDL | < BDL | < BDL | < BDL | < BDL | < BDL | < BDL | < BDL |

**Table 3** Characteristics of the examined PET bottled water samples after 2 months at ~ 45 °C

| S. no | Parameters | Brands name with sample ID |
|-------|------------|-----------------------------|
|       |            | Aquafina | Bisleri | Catch | Divyajal | Kinley | Railneer |
|       |            | 7476D12J18 | 7476D18J18 | 297 | 341 | 100W008 | 100W154 | UP0330133 | UP0330134 | H30E8E11 | H30E8E12 | 2310 | 2311 |
| 1     | Odour      | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable |
| 2     | Taste      | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable |
| 3     | pH         | 6.72 | 6.78 | 6.53 | 6.51 | 6.67 | 6.72 | 6.72 | 6.76 | 6.72 | 6.69 | 6.68 | 6.63 |
| 4     | TDS (mg/l) | 6.56 | 6.16 | 87.45 | 90.33 | 167.23 | 170.21 | 71.30 | 74.22 | 24.60 | 22.10 | 131.90 | 133.70 |
| 5     | Conductivity (µS/cm) | 6.20 | 6.76 | 181.76 | 184.80 | 278.01 | 280.56 | 139.42 | 141.98 | 43.30 | 41.65 | 248.66 | 245.21 |
| 6     | Chloride (mg/l) | 4.00 | 4.50 | 7.00 | 7.50 | 14.50 | 15.00 | 6.50 | 7.00 | 4.50 | 4.00 | 9.00 | 9.50 |
| 7     | Nitrate (mg/l) | 2.98 | 3.16 | 15.89 | 16.22 | 4.73 | 4.99 | 19.04 | 19.78 | 4.88 | 4.99 | 28.12 | 28.86 |
| 8     | DEHP (µg/l) | 0.58 | 0.52 | 1.87 | 1.67 | 2.1 | 2.5 | 0.95 | 0.98 | 0.88 | 0.76 | 3.26 | 2.97 |
### Table 4: Characteristics of the examined PET bottled water samples after 2 months at −20 °C

| S. no | Parameters | Aquafina | Bisleri | Catch | Divyajal | Kinley | Railneer |
|-------|------------|----------|----------|-------|----------|--------|----------|
|       |            | 7476D12J18 | 7476D18J18 | 297   | 341      | UP0330133 | UP0330134 | H30E8E11 | H30E8E12 | UP0330133 | UP0330134 | H30E8E11 | H30E8E12 | UP0330133 | UP0330134 | H30E8E11 | H30E8E12 |
| 1     | Odour      | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable |
| 2     | Taste      | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable |
| 3     | pH         | 6.94      | 6.96      | 6.76      | 6.73      | 6.87      | 6.90      | 6.90      | 6.95      | 6.90      | 6.90      | 6.95      | 6.91      | 6.90      | 6.83      | 6.80      |
| 4     | TDS (mg/l) | 5.00      | 4.88      | 85.28     | 88.21     | 165.33    | 168.10    | 68.40     | 72.20     | 22.50     | 20.20     | 129.41    | 130.53    | 6.83      | 6.80      |
| 5     | Conductivity (µS/cm) | 5.62   | 5.80      | 178.58    | 180.32    | 275.88    | 277.87    | 136.63    | 139.33    | 41.77     | 39.78     | 246.01    | 243.78    | 6.83      | 6.80      |
| 6     | Chloride (mg/l) | 4.50 | 5.00      | 7.50      | 8.00      | 15.00     | 15.50     | 7.00      | 7.50      | 5.00      | 4.50      | 9.50      | 10.00     | 6.83      | 6.80      |
| 7     | Nitrate (mg/l) | 2.77 | 2.98      | 15.53     | 15.93     | 4.61      | 4.80      | 18.38     | 18.59     | 4.12      | 4.51      | 26.65     | 27.00     | 6.83      | 6.80      |
| 8     | DEHP (µg/l) | < BDL    | < BDL     | < BDL     | < BDL     | < BDL     | < BDL     | < BDL     | < BDL     | < BDL     | < BDL     | < BDL     | < BDL     | 6.83      | 6.80      |

### Table 5: Characteristics of the examined PET bottled water samples after 6 months at −45 °C

| S. no | Parameters | Aquafina | Bisleri | Catch | Divyajal | Kinley | Railneer |
|-------|------------|----------|----------|-------|----------|--------|----------|
|       |            | 7476D12J18 | 7476D18J18 | 297   | 341      | UP0330133 | UP0330134 | H30E8E11 | H30E8E12 | UP0330133 | UP0330134 | H30E8E11 | H30E8E12 |
| 1     | Odour      | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable |
| 2     | Taste      | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable |
| 3     | pH         | 6.31      | 6.36      | 6.12      | 6.14      | 6.28      | 6.31      | 6.31      | 6.36      | 6.35      | 6.32      | 6.24      | 6.20      | 6.35      | 6.32      | 6.24      | 6.20      |
| 4     | TDS (mg/l) | 8.90      | 8.28      | 89.65     | 93.06     | 178.34    | 180.45    | 77.98     | 80.60     | 29.50     | 27.40     | 138.21    | 139.10    | 29.50     | 27.40     | 138.21    | 139.10    |
| 5     | Conductivity (µS/cm) | 8.11 | 8.34      | 189.32    | 191.80    | 283.56    | 286.32    | 144.98    | 147.12    | 49.02     | 47.54     | 253.80    | 250.76    | 49.02     | 47.54     | 253.80    | 250.76    |
| 6     | Chloride (mg/l) | 2.50 | 3.00      | 5.50      | 6.00      | 13.00     | 13.50     | 5.00      | 5.50      | 3.00      | 2.50      | 7.50      | 8.00      | 3.00      | 2.50      | 7.50      | 8.00      |
| 7     | Nitrate (mg/l) | 3.76 | 3.99      | 16.53     | 16.87     | 5.87      | 6.03      | 21.33     | 21.87     | 6.00      | 6.22      | 34.12     | 35.22     | 6.00      | 6.22      | 34.12     | 35.22     |
| 8     | DEHP (µg/l) | 1.31      | 1.10      | 3.67      | 3.87      | 4.76      | 4.90      | 2.16      | 2.65      | 1.73      | 1.62      | 5.25      | 5.39      | 1.73      | 1.62      | 5.25      | 5.39      |
bottled mineral water. Moreover, some studies have been done and reported the DEHP concentration from 0.35 μg/l to 8.8 μg/l in PET bottled water samples which were kept at different temperatures for different times [37, 76–78]. Casajuana and Lacorte [55] had reported the highest concentration of DEHP (<0.002–0.188 μg/l) in PET bottled water when the samples were stored in outdoors for 10 weeks at 30 °C. Schmid et al. [56], and Muhamad et al. [68] had reported the DEHP concentration (0.10–0.71 μg/l) in PET bottled water samples after 17 h of incubation in darkness/sunlight at room temperature and at 60 °C. Leivadara et al. [57] had reported the DEHP concentration (<0.002–6.8 μg/l) in bottled water samples when the samples were stored at 30 °C for 3 months. Keresztes et al. [75] had studied the leaching of DEHP from PET bottles to water and detected the DEHP concentration up to 1.7 μg/l. They concluded that recycled PET bottles contained higher DEHP than virgin PET bottles. Guart et al. [46] had reported the phthalate concentration from 0.022 to 20.5 μg/l in 12 fresh PET bottled water samples and 0.635 to 13.0 μg/l in 37 one year stored samples. Jeddi et al. [72] reported the lowest (0.32 μg/l) and highest (0.92 μg/l) concentration of DEHP at −18 °C and 40 °C respectively.

Wegelin et al. [79] reported the terephthalate monomers and dimers, which were the PET degradation products in the presence of sunlight. Monarca et al. [69] stated that phthalate esters are produced from the organic compounds after oxidation through photodegradation in sunlight. Peterson [80] has described that photolysis is the important pathway for abiotic degradation of phthalates in water. Exposure to phthalates causes developmental and reproductive toxicities in rodents [81, 82]. Plastic components migrate in low quantities into the fresh water during the bottle manufacturing or bottling process and depend on the water brand. The manufacturing process of bottles could also be a cause of plasticizers when the process of polymerization has not been completed.

After production, the presence of DEHP in PET bottled water may be due to the contamination in the water treatment facilities or bottling plant and the source of water. The effect of environmental factors such as high temperature, etc. on the PET bottled water samples from manufacture to purchase cannot be excluded [37, 75, 78]. Lin et al. [83] stated that PET degradation is temperature-dependent and it occurs more rapidly at high temperatures. In the present study, The author has reported the highest migration of DEHP from PET bottled to water samples when the samples were stored at sunlight (~45 °C) for 6 months and 2 months. While the migration of DEHP was observed below detection limits when samples were analyzed immediately after purchase and stored at −20 °C for 2 months. This study confirmed that the migration of DEHP is higher at high temperatures while lower at low temperatures. The highest migration of DEHP was

### Table 6

| S. no | Parameters | Aquafina | Bisleri | Catch | Divygal | Kinley | Railneer |
|-------|------------|----------|---------|-------|---------|--------|---------|
| 1 Odour | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable |
| 2 Taste | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable | Agreeable |
| 3 pH | 6.75 | 6.81 | 6.59 | 6.57 | 6.59 | 6.59 | 6.71 |
| 4 TDS (mg/l) | 6.50 | 5.98 | 8.87 | 90.44 | 174.56 | 71.45 | 74.62 |
| 5 Conductivity (µS/cm) | 6.82 | 7.01 | 4.50 | 4.50 | 4.50 | 4.50 | 4.50 |
| 6 Chloride (mg/l) | 3.12 | 3.33 | 15.88 | 16.03 | 15.00 | 15.00 | 15.00 |
| 7 Nitrate (mg/l) | 3.12 | 3.33 | 15.88 | 16.03 | 15.00 | 15.00 | 15.00 |
| 8 DEHP (µg/l) | 0.31 | 0.38 | 0.31 | 0.38 | 0.31 | 0.38 | 0.31 |
observed in the Railneer brand (2.97–5.39 μg/l) followed by Catch (2.10–4.90 μg/l), Bisleri (1.67–3.87 μg/l), Divya-jal (0.95–2.65 μg/l), Kinley (0.76–1.73 μg/l) and Aquafina (0.52–1.31 μg/l) at ~45 °C after 2 & 6 months. While the DEHP concentration was found below the detection limit when the samples were analyzed after purchase and after 2 months (stored at –20 °C). Some amount of DEHP was observed in the Railneer brand (2.03–2.66 μg/l) followed by Catch (1.87–1.99 μg/l), Bisleri (1.03–1.25 μg/l), Divya-jal (0.81–0.89 μg/l), Kinley (0.51–0.55 μg/l), and Aquafina (0.31–0.38 μg/l) after 6 months when the samples were stored at –20 °C.
As per the USEPA and World Health Organization (WHO) a maximum permissible limit of DEHP in drinking water is 6 μg/l–8 μg/l [84, 85]. The Food Safety and Standards Authority of India (FSSAI) and EU Plastics Regulation have specified the migration limit (1.5 mg/kg–5.0 mg/kg) for DEHP from plastic materials to food products [86, 87]. Jedd et al. [72] studied the health of children in Iran and reported that phthalate exposure through water is reduced
with increasing age because water intake is higher in children as compared to adults [88]. As per the IARC (The International Agency for Research on Cancer), only DEHP is carcinogenic to humans in all the phthalate esters [89]. The cancer risk due to DEHP exposure via water intake stored at 40 °C was greater than for the other storage conditions while the least carcinogenic risk from DEHP was observed in the bottled water stored under freezing conditions (-18 °C) [58]. Due to the DEHP concentration, the carcinogenic risk was tremendously below the accepted risk level because the DEHP in water corresponding to cancer risk in 1,000,000 was 3 μg/l [89]. Finally, the carcinogenic risk posed by the maximum concentration of DEHP in PET bottled water samples is negligible. Bolgar et al. [39] reported that DEHA can be considered an effective replacement for DEHP.

### 5 Conclusions

PET material is used worldwide as a container for bottled water; even it is used in which area where the drinking water problem occurs. In the current study, PET bottled mineral water samples were studied in different storage conditions which give an answer to public questions about the safety of bottled water. The different storage conditions such as temperature and time are the main factors which affect the migration of DEHP from PET bottles to water. DEHP was estimated in the PET bottled water samples by GC–MS/MS.

After comparing the results before and after storage, the author concludes that poor storage conditions cause an upsurge in the DEHP concentration. The migration of DEHP from the PET bottle to water depended on the pH of the water samples and increases with the decreasing pH. DEHP migration was increased with the increasing temperature and time while migration was observed low when the samples were stored at low temperatures (> 25 °C). The migration of DEHP may depend on the brand because water characteristics and bottling process vary from brand to brand. The author observed that DEHP was detected in 6 months & 2 months of stored water samples which were stored in sunlight (~ 45 °C). Some amount of DEHP was also observed in samples which were stored for six months at ~ 20 °C.

In spite of the consumption of PET bottled water was more in developing and developed countries the detected concentration of DEHP was far below the toxic levels. Therefore, the main conclusion of this study is that all common conditions of PET bottled water stored in retail supermarkets, outlets, and homes are nontoxic/safe for consumers. Further analysis of phthalates is required for the prevention of the health effects of chemical exposure considering the development of PET bottled water consumption.

### Supplementary Information

The online version contains supplementary material available at https://doi.org/10.1007/s42250-022-00385-6.

### Acknowledgements

The author is deeply grateful to Amity University, Noida for giving the platform for doing this study. The author also thank to Mr. Vishal for help in collecting the samples from the local market.

### Author contribution

Single author has done all the things.

### Funding

This study was not supported by any funding agency.

### Data availability

Not applicable.

### Code availability

Not applicable.

### Declarations

### Conflict of interest

Not applicable.

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