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The Occurrence of Haloacetic Acids and Dalapon in Bottled Waters and an Assessment of Their Health Risk

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Abstract: While disinfection ensures the destruction of pathogenic microorganisms, the disinfectant substances used react with some organic and inorganic substances in water, causing the formation of disinfection by-products. Some disinfection by-products have been classified as carcinogenic by the Environmental Protection Agency (EPA). Haloacetic acids are one of the disinfection by-product groups that have been detected in drinking water and are carcinogenic. It is commonly believed that bottled water, which is being increasingly consumed worldwide, does not contain environmental pollutants. For this reason, research on bottled water is limited. In this study, the amount of 9 haloacetic acids and dalapon were investigated in 28 different branded bottled water samples collected from a market. As a result of the study, the total haloacetic acid concentrations were found to be from 2.13 to 7.56 µg/L, and the dalapon concentration was < detection limit-12.47 µg/L. At least three different haloacetic acids were detected in each sample analyzed. It was determined that the threshold values given by the EPA and the World Health Organization (WHO) were not exceeded. It has been observed that bottled waters, which are generally considered to be of higher quality than mains water, contain haloacetic acids and dalapon at low concentrations. A health risk assessment was performed for dalapon and trichloroacetic acid (TCAA). A low non-carcinogenic risk and tolerable carcinogenic risk were determined. Care should be taken to counter the negative health effects of HAAs and dalapon.

Keywords: bottled water; disinfection by-products; haloacetic acids; dalapon

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1. Introduction

With an increasing population and an increasing release of pollutants, water pollution has become a major problem worldwide. Pollutants that impair the quality of drinking water can be physical, chemical, and biological [1]. Access to safe drinking water is very important for protecting human health. Disinfection is a standard treatment for drinking water. Disinfection of drinking water is accomplished to remove pathogenic microorganisms, and residual disinfectant must remain in water distribution networks to prevent the regrowth of pathogenic microorganisms [2]. An undesirable consequence of disinfection is the formation of disinfection by-products (DBPs). Disinfectants may react with natural organic and inorganic substances and cause the formation of DBPs. More than 700 DBPs have been identified in waters [3]. Commonly used disinfectants include chlorine, chloramines, chlorine dioxide, ozone, and ultraviolet radiation [1,4]. Chlorine-based (chlorine, chlorine dioxide, and chloramines) have a high disinfectant effect, low cost, and easy applicability and they persist in the distribution network [5]. Ozone and ultraviolet radiations are very good disinfectants, but they are not permanent in the distribution network and their cost is higher than chlorine-based disinfectants. Haloacetic acids (HAAs) are one of the most commonly seen disinfection by-product groups after trihalomethanes (THMs). The precursor to the formation of HAAs and THMs is natural organic matter. Natural organic substances are formed as a result of physical, chemical, and biological activities in water resources [6]. The foremost HAAs include monochloroacetic acid (MCAA), dichloroacetic acid (DCAA), trichloroacetic acid (TCAA), monobromoacetic acid (MBAA), dibromoacetic acid (DBAA), tribromoacetic acid (TBAA), bromochloroacetic acid (BCAA), bromodichloroacetic acid (BDCAA), and chlorodibromoacetic acid (CDBAA) [7,8].

The toxic effects of HAAs, such as carcinogens, and reproductive and developmental effects, have been observed in laboratory animals [6]. The Environmental Protection Agency (EPA) integrated risk information system (IRIS) classified DCAAs and TCAAs as possible carcinogens. In some experimental studies, it was reported that the DCAA is hepatoxic, which means that it has been found to cause cell accumulation in liver glycogen and creates neurotoxicity in rodents. Adverse reproductive effects in mammals have been observed for DCAA and DBAA. In experiments on rats, it was observed that DBAA and DCAA can impair the fertility of adult male rats [9]. HAAs are commonly detected in drinking water. Xue et al. [10] detected a total 5-HAA concentration of 0.19 µg/L in tap water and below the detection limit in bottled water. Wu et al. [11] determined the total concentrations of HAAs in groundwater and surface waters to be 17.8 µg/L and 190 µg/L, respectively. Huang and Rohrer [12] established concentrations of HAAs in the range from <0.1 to 20.2 µg/L in tap water. Previous studies indicated that HAAs are detected at µg/L levels in drinking water. The EPA has set a maximum contaminant level for 5-HAA (MCAA, DCAA, TCAA, MBAA, DBAA) at 60 µg/L in drinking water [7]. Dehghani et al. [13] detected a total 5-HAA concentration ranging from 148 to 3488 µg/L indoor swimming pools. 5-HAA in swimming pools has also been detected in higher concentrations than in drinking water. The World Health Organization (WHO) set the maximum contamination level at 20 µg/L, 50 µg/L, and 100 µg/L for MCAAs, DCAAs, and TCAAs, respectively, in drinking water. A limit value of 200 µg/L has been set for dalapon in drinking water by the EPA [7]. Worldwide consumption of bottled drinking water is rising. Disliking the taste of tap water and thinking that bottled water contains fewer pollutants are the principal reasons for this situation [14]. Since bottled water is thought to be both pure and safe, and perceived as delicious, consumption of bottled water is increasing despite its high price compared to mains water [15]. There are some studies examining the quality of bottled waters. For example, Shams et al. [16] observed a trend in the microbial quality in bottled water of increasing heterotrophic and pathogenic bacteria numbers after bottling. Cidu et al. [15] compared the quality of bottled water and tap water. It was determined that 1 out of every 37 examples of bottled water exceeded the limit values for arsenic. They concluded that bottled water does not always deliver better quality than tap water. It has been determined in different studies that toxic natural radio elements and trace elements exceed the limit values in some bottled waters [17–19]. Bottled water is subjected to treatment processes, such as filtration, reverse osmosis, and ozonation. Ozone can react with natural organic matter or halides in spring waters to form HAAs and bromates [14].

This study focused on monitoring the presence of HAAs and dalapon in bottled water offered for sale in Konya, Türkiye. HAAs (MCAA, MBAA, DCAA, TCAA, BCAA, DBAA, BDCAA, DBAA, and BDCAA) and dalapon were examined in 28 bottled water samples. In addition, the health risk assessment induced by HAAs and dalapon was evaluated.

2. Materials and Methods

2.1. Chemicals and Bottled Water Samples

The EPA Method 552.2 HAAs standard, including MCAA, MBAA, DCAA, TCAA, BCAA, DBAA, BDCAA, CDBAA, TBAA, and dalapon was obtained from Absolute Standards (Hamden, CT, USA). Methyl tert butyl ether (MTBE), methanol, sodium chloride (NaCl), sodium sulfate ($NaSO_4$), sodium bicarbonate ($NaHCO_3$), and sulfuric acid (H_2SO_4) were purchased from Merck (Darmstadt, Germany). Ultra-pure water was obtained from a Milli-Q Plus water purifier (Burlington, MA, USA). Physico and chemical properties of HAAs and dalapon are given in Table S1. Bottled water samples were taken from the bottled water offered to the public from markets in Konya, Türkiye. Twenty-eight bottled water samples were taken, including 24 natural spring waters (SW) and four natural mineral waters (MW). Natural spring waters and natural mineral waters are all different brands. Each sample represents a different brand. In the city of Konya, mostly natural spring water is consumed as bottled water. Natural mineral waters of the four brands that are sold the most in the markets were preferred. All bottled water samples examined were plastic-packaged bottled waters.

2.2. *Analytical Procedure*

Analyses of the samples were carried out according to EPA method 552.2. After the sample preparation stage was performed by liquid–liquid extraction and derivatization, the detection of HAAs was performed using gas chromatography equipped with a μ-electron capture detector (GC-μ-ECD). EPA method 552.2 is a method capable of detecting HAAs and dalapon in drinking water. Liquid–liquid extractions, derivatization procedures and the use of gas chromatography (GC) are defined in this method. For the extraction of HAAs and dalapon, a 35 mL sample was placed in a 40 mL EPA vial. A 2 mL amount of concentrated H_2SO_4 was added and shaken to adjust the pH to be <0.5. A further 16 g of sodium sulfate was added, and the vial was shaken for 5 min until it was all dissolved. A 4 mL amount of MTBE was added, and the capped vial was shaken for 2 min. The vial was left for 5 min to achieve phase separation. Then, 3 mL of MTBE was transferred to a 10 mL vial, and 1 mL of 10% H_2SO_4 in methanol was added. The capped vial was heated in a water bath at 50 °C for 2 h. After the sample had cooled, 4 mL of the saturated sodium bicarbonate solution was added and shaken for 2 min. A 1 mL amount of the MTBE was transferred into amber vials for GC analysis. A recovery study was carried out to determine the effect of different analyte concentrations on the extraction method applied to the samples. To achieve this, HAAs and dalapon at concentrations of 0.1 ng/μL and 1.0 ng/μL were spiked into 35 mL of distilled water, and the extraction procedure was carried out.

The analysis of HAAs and dalapon was carried out using an Agilent Technologies 6890N gas chromatograph equipped with a μ-electron capture detector. Chromatographic separation was carried out using an HP-5 5% phenylmethyl siloxane fused silica capillary column (30 m length, 0.32 mm i.d., and 0.25 μm film thickness). The injection volume, injection port temperature and detector temperature were set to 1 μL, 175 °C, and 300 °C, respectively. The temperature program for the analysis was as follows: start at 35 °C, hold for 9 min, increase at a rate of 1 °C/min to 40 °C, hold for 3 min, and increase at a rate of 6 °C/min to 220 °C, then hold for 10 min. The carrier gas was helium (99.999% purity), and the complementary gas was nitrogen (99.999% purity). A chromatogram for a 1 ng/μL of HAA and dalapon standard has been depicted in Figure 1. Analytical parameters for HAAs and dalapon are shown in Table S2.

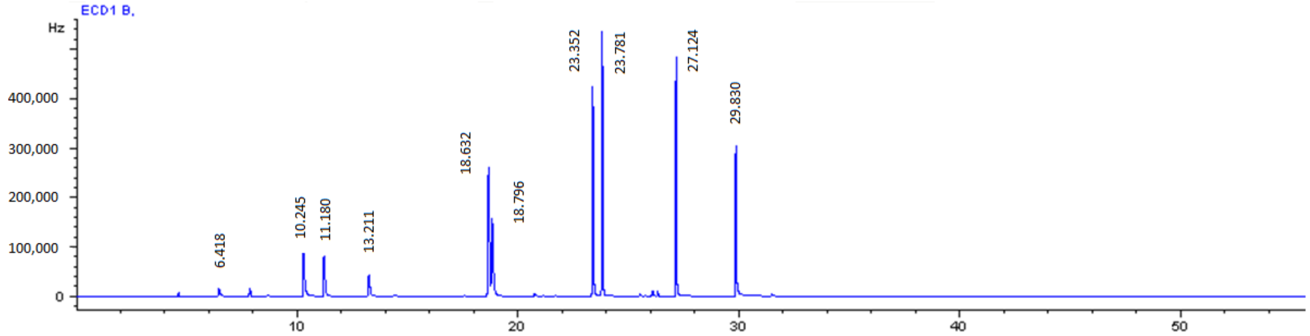

Figure 1. An HAAs and dalapon compounds chromatogram (peak order: MCAA, MBAA, DCAA, dalapon, TCAA, BCAA, DBAA, BDCAA, CDBAA, and TBAA).

2.3. *Risk Assessment*

Risk assessment procedures were adapted from Radwan et al. [20], Djam et al. [21], and Gan et al. [22]. In this study, non-carcinogenic risk and carcinogenic risk were evaluated.

The ELRC (excess lifetime cancer risk) or carcinogenic risk was calculated by Equation (2). To determine the non-carcinogenic risk, the HQ value was calculated and evaluated with Equation (3), where ELRC is the excess lifetime cancer risk; CDI is the chronic daily intake; SF is the slope factor; HQ is the hazard quotient; and RfD is the reference dose. CDI was assessed by Equation (1). Here, C is the concentration of HAAs and dalapon, IR is the ingestion rate, EF is the exposure frequency, ED is the exposure duration, BW is the body weight, and AT is the average time.

$$\mathrm{CDI} = (\mathrm{C} \times \mathrm{IR} \times \mathrm{EF} \times \mathrm{ED})/(\mathrm{BW} \times \mathrm{AT}) \quad (1)$$

$$\mathrm{ELRC} = \mathrm{CDI} \times \mathrm{SF} \quad (2)$$

$$\mathrm{HQ} = \mathrm{CDI}/\mathrm{RfD} \quad (3)$$

The terms SF for carcinogenic risk and RfD for non-carcinogenic risk were used. The values of the parameters used in the health risk assessment are summarized in Table 1. The SF and RfD values were taken from IRIS. The RfD values were available for only DCAA, TCAA, and dalapon. The SF values were available for only DCAA and TCAA. Since DCAA was shown to be below the detection limit, a risk assessment could not be made. In addition, the risk assessment was made according to the average age and weight of males, females, and children.

Table 1. Input parameters value for HAAs and dalapon health risk assessment.

Parameters	Value	Reference
Rfd (mg/kg/day)	0.004 (DCAA) 0.020 (DCAA) 0.003 (Dalapon)	[23]
Sf (mg/kg/day)	0.05 (DCAA) 0.07 (DCAA)	[23]
C	Measured	In this study
IR (L/day)	2 (Male, female) 1 (Children)	[24] [21]
EF (days/year)	365	[25]
ED (year)	75.9 (Male) 81.3 (Female) 5–17 (Avagere 10) (Children)	[26] [27]
AT (days)	Lifetime×365 (carcinogenic risk) ED×365 (non-carcinogenic risk)	[20] [25]
BW	75.8 (Male) 69.9 (Female) 32.7 (Children)	[26] [27]

3. Results

3.1. Physicochemical Properties of Bottle Waters

The physicochemical properties of natural spring waters and natural mineral waters are given in Table S3 and Table S4, respectively. In Türkiye, limit values for drinking water are determined according to the regulation on water intended for human consumption and the regulation on natural mineral waters. In Tables S3 and S4, the limit values mentioned in these regulations are given. Aluminum, ammonium, chloride, conductivity, pH, iron, manganese, oxidizability, sulfate, sodium, colony number, coliform bacteria, and the total organic carbon (TOC) values of natural spring waters do not exceed the limit values given in the regulation on water intended for human consumption. The criteria for color, odor, taste, and turbidity parameters are to meet the standard phrasing: "acceptable by consumers and there is no abnormal change" stated in the regulation. A limit value is given for fluoride in natural mineral waters in the regulation on natural mineral waters. The values found in the samples do not exceed this limit value. In addition, in this regulation, natural mineral waters are classified as having sodium, fluoride, bicarbonate, calcium, magnesium, and ferrous ingredients. The natural mineral waters are also in the water class suitable for a sodium diet.

3.2. Concentrations of HAAs and Dalapon in Bottled Water Samples

The concentrations of HAAs and dalapon in bottled water are summarized in Table 2. Even though MBAA and DCAA were not detected in any of the twenty-eight samples, CDBAA and TBAA were detected in all investigated samples. Dalapon and MCAA were detected in all but one of the samples. TCAA was detected in seven samples and BCAA was detected in four samples. BDCAA was detected in all but two of the samples. At least three HAAs were detected in each of the bottled water samples examined. The maximum detected concentration of dalapon was 12.48 µg/L. The highest concentrations of dalapon were detected in the bottled water samples. Dalapon was followed by MCAA (maximum concentration, 2.78 µg/L), BCAA (maximum concentration, 2.58 µg/L), TBAA (maximum concentration, 1.77 µg/L), CDBAA (maximum concentration, 1.02 µg/L), BDCAA (maximum concentration, 0.78 µg/L), and DBAA (maximum concentration, 0.77 µg/L). Dalapon, which is commonly used in lawns, drainage ditches, railway lines, and industrial areas, is a herbicide used to eliminate unwanted weeds [7]. Detection of high concentrations is also dependent on these uses. Simon et al. [28] studied the bromination and chlorination of amino acids. DBAA produced by bromination was found to be up to three times higher than DCAA produced by chlorination. In this study, DBAA was detected at higher concentrations than DCAA in similar compounds.

Figure 2 illustrates the concentrations of total HAAs and total 5-HAAs (MCAA, DCAA, TCAA, MBAA, and DBAA). Total HAA concentrations ranged from 2.13 to 7.56 µg/L. The total 5-HAA concentrations varied between <dl-4.47 µg/L. The total 5-HAA concentration was determined at the highest rate in sample 7-SW. MBAA and DCAA, which are among the compounds that make up the 5-HAA group determined by the EPA, were not detected in the bottled water samples. The total 5-HAA concentrations did not exceed the limit value (60 µg/L) given by the EPA. Additionally, MCAA, DCAA, and TCAA did not exceed the limit value (20 µg/L, 50 µg/L, and 100 µg/L, respectively) given by the WHO.

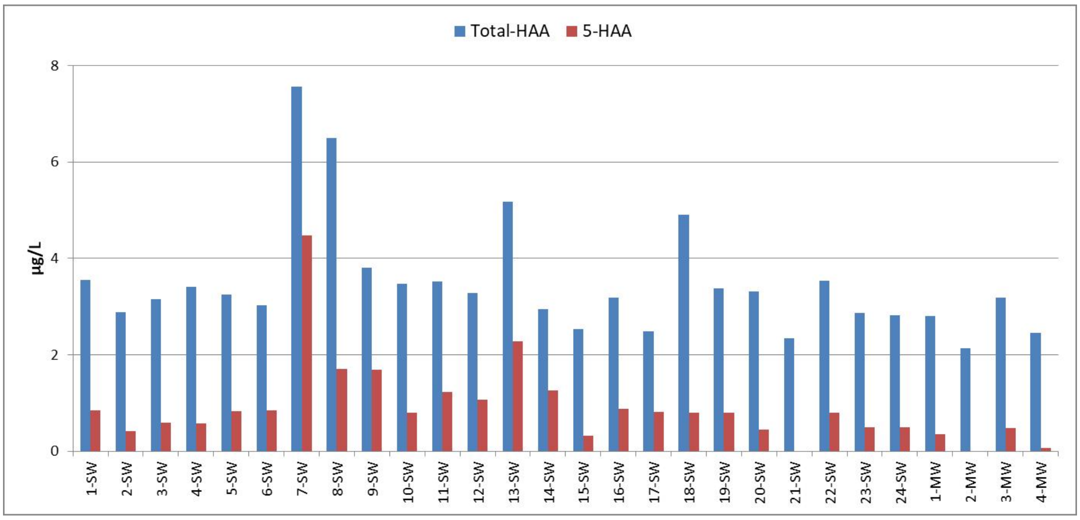

Figure 2. Total HAA and Total 5-HAA concentrations.

Table 2. Concentrations of HAAs and dalapon in bottled water (μg/L).

Sample No	MCAA	MBAA	DCAA	TCAA	BCAA	BDCAA	DBAA	CDBAA	TBAA	Dalapon
1-SW	0.85	<dl	<dl	<dl	<dl	0.08	<dl	1.00	1.62	11.76
2-SW	0.41	<dl	<dl	<dl	<dl	0.03	<dl	1.00	1.45	6.16
3-SW	0.59	<dl	<dl	<dl	<dl	0.03	<dl	1.00	1.54	1.16
4-SW	0.57	<dl	<dl	<dl	<dl	0.07	<dl	1.00	1.77	12.41
5-SW	0.83	<dl	<dl	<dl	<dl	0.09	<dl	1.00	1.32	12.48
6-SW	0.85	<dl	<dl	<dl	<dl	0.04	<dl	1.00	1.14	12.25
7-SW	2.78	<dl	<dl	0.93	0.10	0.78	0.77	0.98	1.23	<dl
8-SW	1.05	<dl	<dl	<dl	2.58	0.06	0.65	0.98	1.18	6.21
9-SW	0.85	<dl	<dl	0.14	<dl	<dl	0.70	1.00	1.11	6.73
10-SW	0.80	<dl	<dl	<dl	<dl	0.03	<dl	1.00	1.65	10.62
11-SW	0.30	<dl	<dl	0.25	<dl	0.06	0.68	1.00	1.22	9.32
12-SW	0.38	<dl	<dl	0.01	<dl	0.04	0.66	1.00	1.18	11.37
13-SW	0.29	<dl	<dl	1.32	0.57	0.06	0.65	1.02	1.25	11.37
14-SW	0.21	<dl	<dl	0.66	<dl	0.05	0.38	0.51	1.14	11.03
15-SW	<dl	<dl	<dl	<dl	<dl	0.03	0.33	1.00	1.18	11.35
16-SW	0.21	<dl	<dl	<dl	<dl	0.07	0.67	1.00	1.24	11.76
17-SW	0.03	<dl	<dl	0.11	<dl	0.04	0.67	1.00	0.64	11.84
18-SW	0.47	<dl	<dl	<dl	1.63	0.17	0.33	1.00	1.30	12.09
19-SW	0.47	<dl	<dl	<dl	<dl	0.10	0.33	1.00	1.47	7.22
20-SW	0.44	<dl	<dl	<dl	<dl	0.34	<dl	1.00	1.54	8.49
21-SW	0.00	<dl	<dl	<dl	<dl	<dl	<dl	0.98	1.36	8.17
22-SW	0.47	<dl	<dl	<dl	<dl	0.36	0.33	1.00	1.38	9.31
23-SW	0.16	<dl	<dl	<dl	<dl	0.12	0.33	1.00	1.26	8.90
24-SW	0.18	<dl	<dl	<dl	<dl	0.08	0.32	1.00	1.25	8.41
1-MW	0.35	<dl	<dl	<dl	<dl	0.09	<dl	1.00	1.37	9.23
2-MW	0.00	<dl	<dl	<dl	<dl	0.03	<dl	0.98	1.13	7.68
3-MW	0.15	<dl	<dl	<dl	<dl	0.09	0.33	1.00	1.62	8.66
4-MW	0.07	<dl	<dl	<dl	<dl	0.08	<dl	1.00	1.31	7.76

Note: SW: Spring water; MW: Mineral water; <dl: below detection limit.

Table 3 shows the concentrations of HAAs seen in published literature studies alongside those in this study. In the literature studies, the maximum concentration detected in bottled water was 1 μg/L for the BDCAA compound. However, the BDCAA compound was determined to be 2.58 μg/L in this study. The difference in concentrations detected in this study and literature studies may be due to different treatment methods, different water sources, and different analysis methods. The dalapon compound, which was detected at the highest concentrations in this study, was not detected in the literature studies. In this study, MBAA and DCAA, which we detected as <dl in all samples, were determined to be a maximum 0.76 μg/L in bottled waters tested in the literature. In the literature, it has been stated that bottled water is disinfected with ozone, and that ozone can form bromates and HAAs. The reaction of ozone with bromide can form the secondary oxidant HOBr. HOBr can react with natural organic materials to form brominated HAAs [29]. Liu and Mou [30] found that the DCAA concentration in bottled water decreased over time. In some studies, conducted in drinking water distribution networks, it has been stated that THMs increase in distribution networks and become stable over time, while the concentrations of HAAs decrease [31]. This may be due to the decrease in residual chlorine over time. Considering that bottled water is also stored after bottling, the initial concentrations of HAAs may have decreased. In this study, a significant correlation was not found between HAA and dalapon concentrations and the physicochemical properties of the bottled waters.

Table 3. HAAs and dalapon concentrations in bottled water in published literature studies and this study (µg/L).

Country	MCAA	MBAA	DCAA	TCAA	BCAA	BDCAA	DBAA	CDBAA	TBAA	Dalapon	Reference
USA	<0.05	<0.01	<0.01	<1.00	<0.02	<0.01	<0.02	<0.01	<0.01	nd	[10]
Saudi Arabia	0.71–0.75	0.57–0.76	0.53–0.65	0.58–0.74	0.55–0.82	nd	0.59–1.58	nd	nd	nd	[32]
Greece	nd	nd	nd	0.1–1.5	1.6–2.2	1	0.6–0.9	nd	nd	nd	[33]
China	nd	nd	0.4–0.6	nd	nd	nd	nd	nd	nd	nd	[30]
USA	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	[7]
USA	<0.1	<0.1	<0.1	nd	nd	nd	nd	nd	nd	nd	[12]
Turkey	<dl-2.77	<dl	<dl	<dl-1.32	<dl-2.58	<dl-0.78	<dl-0.76	0.51–1.02	0.64–1.77	<dl-12.4	This study

Note: nd: not detected, <dl: below detection limit.

3.3. Risk Assessment

The maximum HQ values for dalapon and TCAA were determined to be 0.12 and 0.0019 in females, 0.11 and 0.0017 in males, and 0.12 and 0.020 in children, respectively. If HQ is more than 1, it may negatively affect human health. If HQ is less than 1, low non-cancer health risks may occur to human health [34]. The non-carcinogenic risk was identified as low for dalapon and TCAA. The non-cancer risk from TCAA was lower than dalapon, and the non-carcinogenic risk was found to be higher in females and children. Figure 3 shows the result of the carcinogenic risk assessment for the TCAA compound. A value of ELRC $< 10^{-6}$ is considered an acceptable risk; $10^{-6} <$ ELRC $< 10^{-4}$ is considered a tolerable risk and a value of ELRC $> 10^{-4}$ is considered an unacceptable risk [34,35]. When Figure 3 is examined, an acceptable risk was determined for the 9-SW, 11-SW, 12-SW, and 17-SW samples for males and females. In samples 7-SW, 13-SW, and 14-SW, the risk level exceeded 10−6 and a tolerable risk was determined for males and females. In all samples, the risk is acceptable risk for children. In addition, the level of carcinogenic risk is higher in females than in males. Zhao et al. [36] found an acceptable level of carcinogenic and non-carcinogenic risks for HAAs in drinking water. Gan et al. [22] detected above 10^{-6} cancer risks for DCAA and TCAA in drinking water.

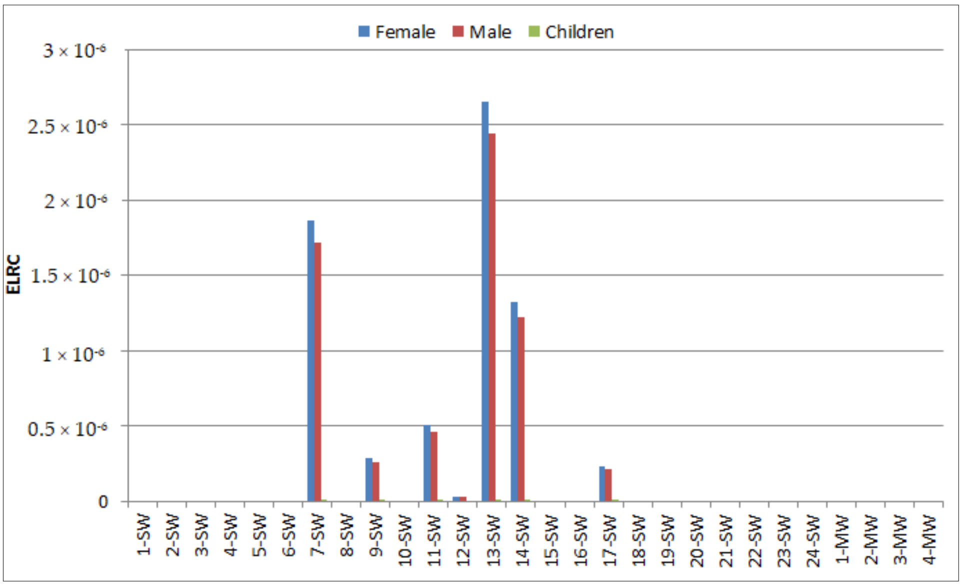

Figure 3. Carcinogenic risk assessment for TCAA.

4. Conclusions

The concentrations of 9-HAAs and dalapon in bottled water purchased from the markets were investigated. At least three of the HAAs and dalapon were detected in all samples. Total HAAs, 5-HAAs, and dalapon concentrations were 2.14–7.56 µg/L, <dl-4.48 µg/L, <dl-12.48 µg/L, respectively. In the regulations in force in Türkiye, there are no limit values for HAAs and dalapon in drinking water. When the results were compared

with the limit values in EPA and WHO, these limits were not exceeded. However, there are no limit values for all target analytes. Carcinogenic and non-carcinogenic health risks have been evaluated for dalapon and TCAA. The non-carcinogenic risk was determined to be low. The carcinogenic risk was determined to be a tolerable risk. However, it should not be forgotten that all pollutants are found in water together and they can show potential synergistic effects. HAAs have negative effects on human health, while dalapon has been seen to cause kidney changes in some people. It is crucial for public health to monitor these compounds in tap water, bottled water, and freshwater offered for public consumption and to determine the maximum concentrations permissible by the regulations. In the regulation on water intended for human consumption, which is in force in Türkiye, only limit values for THMs are determined for disinfection by-products. It will be important for the protection of human health that pollutants, such as HAA and dalapon, are included in the parameters to be monitored in drinking water.

Supplementary Materials: The following supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/w15101810/s1, Table S1: Physico-chemical properties of the investigated HAAs and dalapon; Table S2: Analytical parameters for HAA and dalapon; Table S3: Physicochemical properties of natural spring water; Table S4: Physicochemical properties of natural mineral water samples.

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Data Availability Statement: The data presented in this study are available on request from the corresponding author.

Conflicts of Interest: The author declares no conflict of interest.

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