

# Concentration of Fluoride in Different Sources for Human Consumption in Kirkuk City-Northern Iraq

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**Abstract:** Throughout the world, dentists recognise that fluoride is the most effective caries-preventive agent. The major sources of exposure to fluoride are drinking water, dental products and food. In this study, the quantity of fluoride in the samples was analysed using a fluoride selective electrode. This study aimed to estimate the percentage of fluoride in seven types of toothpaste in the Iraqi market, tap water from seven different locations, eight brands of packaged drinking water and groundwater. The study found that the percentage of total fluoride (TF) was  $(1025.65 \pm 43.73 \text{ ppm})$  in Iraqi commercial brands of toothpaste and the percentage of total soluble fluoride (TSF) was  $(996.60 \pm 35.44 \text{ ppm})$  in packaged drinking water,  $(0.131 \text{ mg/L})$  in tap water and  $(0.144 \text{ mg/L})$  in groundwater. All the samples obtained from the study area were found to have a low quantity of fluoride  $(0.5 \text{ mg/l})$ . None of the samples were within the permissible World Health Organization (WHO) standard for drinking water  $(0.5\text{--}1.5 \text{ mg/l})$ .

**Keywords:** toothpaste; groundwater; drinking water; food; fluoride selective electrode

## 1. Introduction

When found in drinking water, fluoride is one of only a few chemicals that have been shown to have a vital impact on humans. Low concentrations of fluoride in drinking water have a beneficial effect on teeth. In contrast, a high concentration of fluoride in drinking water can have a number of health effects [1], such as increasing the prevalence of dental caries. A dramatic decline in the prevalence of dental caries has been shown worldwide; this reduction has been found to be due to several factors including the presence of fluoride in drinking water [2]. Moreover, fluoride is often added to consumer products, such as toothpaste and toothpowder, for the same purpose [3]. Hence, fluoridation is considered to be an efficient method for reducing dental caries at the public health level; from a societal perspective, it is also seen as a way to address the dental health needs of children with a higher prevalence of tooth decay [4].

Fluoride occurs naturally in water due to the dissolution of minerals that contain it, such as fluorite ( $\text{CaF}_2$ ), fluorapatite ( $\text{Ca}_5\text{F}(\text{PO}_4)_2$ ), cryolite ( $\text{Na}_3\text{AlF}_6$ ) and mica [3], [5]. Well, water is high in naturally occurring fluoride because the fluoride contained in the rock formations dissolves into the groundwater [6]. The incidence of dental caries [7] can be reduced by adding fluoride to community water in ranging from 0.8 ppm to 1.2 ppm, and the most common treatment procedure for achieving that is with NaF or fluorosilicates. Developed countries are not exempt from the problem of dental caries. Recently, fluoridation of water and dentifrices has been found to be the main cause for the decrease in dental caries [8]. Fluoridated toothpaste with soluble fluoride is effective in controlling dental caries. The soluble fluoride could be either in its ionic (free) form ( $\text{F}^-$  or NaF) or in its ionisable form, a monofluorophosphate (MFP) ion, both of which inhibit the caries process by lesser enamel-dentine demineralisation and promoting remineralization [9]. It has been reported that to be most effective, the concentration of soluble fluoride should be 1000 ppm or higher [10]. Consequently, recognition of the importance of using soluble

fluoride in the formulation of toothpaste has increased [11]. The use of soluble fluoride has also been considered for mouth rinses, gels and solutions for professional applications [12].

Several sensitivity methods are available for determining the concentration of fluoride in samples, such as ion chromatography, [13], high-performance liquid chromatography [14], colorimetry [15] and atomic absorption [16], and ion-selective electrodes have been applied to measure the concentration of fluoride in drinking water [17], tea [18], serum and urine [19]. Of these, ion-selective electrodes are most often used because the fluoride quantity in water has been found at low levels and its calculation required very sensitive methods. Moreover, the ion-selective electrode method is easy to use so it is a suitable and sensitive method for continuous monitoring [20], [21].

## 2. Instruments and Chemicals

In this present study, the fluoride concentrations in the water and toothpaste samples were determined using fluoride selective electrodes (WTW, model F800) coupled with an ion analyser (WTW, model inoLABpH/ ION 735, GmbH Company), which was prior standardised with F standards have within 0.05–8.0 ppm. All chemicals were grade and purchased from (Merck, Darmstadt, Germany) and they were used without further purification. All the experiments were done by using distilled water. Prior to being used, all the glassware was kept overnight in 5% nitric acid solution. Sodium fluoride was used to prepare the fluoride stock solution ( $1000 \mu\text{g}\cdot\text{mL}^{-1}$ ), and then kept in polyethylene terephthalate (PET) bottles.

### 2.1. Preparation of the Solutions

The buffer solution, total ionic strength adjustment (TISAB), contained 4.0 g of 1, 2-cyclohexanediamine-N,N,N-tetraacetic acid (CDTA), 58.0 g of sodium chloride and 57.0 mL of glacial acetic acid; the pH was adjusted to 5.0–5.5 by

adding a basic solution from NaOH 6 M. Then, the solution was diluted with distilled water to 1 litre and kept in a plastic bottle.[22]

## 2.2. The Fluoride Standard Solution

Sodium fluoride (NaF) was put in a drying oven at 110 °C for 2 h. After being cooled in desiccators, it was added to a 1.0 liter volumetric flask containing an accurately weighed of the sample 0.22 g. It was then dissolved and diluted with distilled water and kept in a plastic bottle.

## 2.3. Determination of the Fluoride Concentration in Water

Thirty-five samples of drinking tap water were collected from six regions in the city of Kirkuk in Iraq, 28 packaged drinking water samples were collected from eight different brands and 25 groundwater samples were collected from six villages around Kirkuk (Figure 1). The tap water and groundwater samples were collected in 1litre polyethylene terephthalate (PET) bottles. Before filling, the bottles were rinsed with distilled water, and then they were carefully sealed and labeled. Next, 50.0 mL of the water samples were transferred to a volumetric flask (100 mL) and diluted with TISAB solution to the mark. A fluoride solution 5 ppm was prepared from the standard solution 100 ppm by diluting 25 mL to 500 mL in a volumetric flask. Then, 5.0, 10.0, 25.0 and 50.0 mL aliquots of the 5.0 ppm solution were transferred to a 100 mL volumetric flask, and 50 mL of TISAB solution was added and diluted to the mark. These solutions correspond to 0.5, 1.0, 2.5 and 5.0 ppm fluoride, respectively, in the sample. Later, through increasing and drying with a paper tissue, the electrode was immersed in the 0.5 ppm standard fluoride solution. The sample was stirred mechanically for 3 min, and then the potential was recorded. The procedure was repeated for the remaining samples and standards. The calculated potentials were plotted against the log of the concentration of the standards. This plot was used to calculate the quantity of the unknown samples in parts per millions of fluoride. [23]

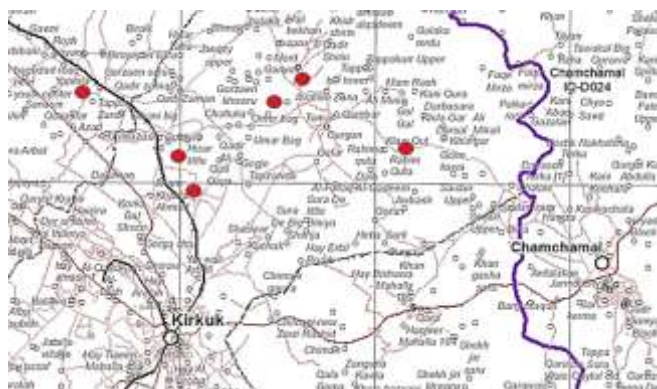


Figure 1: The common operational dataset (COD), Iraq - Kirkuk governorate, 2014.

## 2.4. Measurement of the Fluoride quantity in Toothpaste

The concentration of Fluoride in the toothpaste was calculated as reported previously [3]. The estimation of the

TF and TSF concentration was carried out using an analytical study in the seven widely used toothpaste in Iraq. Briefly, an amount of 100-110 mg of each type of toothpaste was weighed ( $\pm 0.01$ mg), mixed with deionized water 10.0 mL and duplicates of 0.25 mL. The suspension was subjected to the TF analysis. The residue of the suspension was centrifuged (3,000 g, 10 rpm.) to remove the bound IF to the abrasive. Duplicates of 0.25 mL of the supernatant were subjected to TSF analysis to determine concentrations. HCl 0.25 mL, 2.0 M was added to both TF and TSF tubes, after 1 h at 45 °C, the samples were neutralized with NaOH 0.5 mL, 1.0 M and buffered with 1.0 mL of TISAB II (1.0 M acetate buffer, pH 5.0 containing 1.0 M NaCl and 0.4 % CDTA).

Microsoft Excel was used to calculate a linear regression between Fluor quantity in standards and mV using the Fluor concentration in each sample, in ppm. The duplicate was lower than 2%. The standard deviation and the mean for each kind were measured using Statistical Package for Social Science (SPSS version 21.0)[4].

## 3. Results and Discussion

### 3.1. The result of Toothpaste

The seven toothpaste samples with the different brand name were identified in the markets of Kirkuk city- Iraq which was shown in Table 1. All the toothpaste were analyzed. As labeled in their contents, the fluoride concentration of three of them was 1500 ppm, while the concentration of the remaining four types was 1450ppm. The present study analyzed the concentration of total fluoride (TF) and total soluble fluoride (TSF) of seven kinds of toothpaste. As shown in Table 2, all the TF concentrations were lower than the fluoride contents that are declared by the manufacturer. The total soluble fluoride (TSF) concentration was generally less than total fluoride (TF) concentration in the analyzed toothpaste. However, the concentration of (TSF) soluble fluoride can be lower than that of the (TF) total fluoride which is labeled on the Packaging. The mean (average) of TF was high in toothpaste 1, 4, and 5 which were 1025.650, 917.29, and 981.07 ppm consecutively. Whereas the mean TF of toothpaste (2, 3, 6, and 7) was low which were (711.51, 731.54, 833.44, and 787.36 ppm) consecutively. The mean of TSF of the toothpaste was high Fluoride for toothpaste (1, 4 and 5) with values (996.60, 893.79, 835.97 ppm). Whereas, the low mean Fluoride toothpaste had attributed to (toothpaste 2, 3, 6 and 7) with (655.18, 682.19, 819.71 and 732.13 ppm F).

The current research analyzed the concentration of fluoride ion in the toothpaste (1, 4, 5 and 6) with the form of MFP and CaCO<sub>3</sub> as a corrosive agent as labeled. Whereas, the toothpaste (2, 3, and 7) containing NaF and SiO<sub>2</sub> as the abrasive agent. This toothpastess were formulated with fluoride salts that produce fluoride ions [24].

**Table 1:** Information about the toothpaste analyzed in the study

| Commercial Brand (manufacturer) | fluoridate agent | Labeled Fluoride content(ppm) | Abrasive agent             |
|---------------------------------|------------------|-------------------------------|----------------------------|
| 1. Sensodyne                    | NaF              | 1500                          | Silica                     |
| 2. Signal                       | MFP              | 1450                          | CaCO <sub>3</sub> , Silica |
| 3. Colgate                      | MFP              | 1500                          | CaCO <sub>3</sub>          |
| 4. Close -up                    | NaF              | 1450                          | Silica                     |
| 5. Crest                        | NaF              | 1450                          | Silica                     |
| 6. Astera                       | NaF              | 1500                          | CaCO <sub>3</sub>          |
| 7. Crest\BeAD()VENTUROUS        | MFP              | 1450                          | CaCO <sub>3</sub>          |

**Table 2:** Concentrations of expected fluoride measured total fluoride

| Toothpaste               | Total Fluoride | Soluble Fluoride |
|--------------------------|----------------|------------------|
| 1. Sensodyne             | 1025.65±43.73  | 996.60±35.44     |
| 2. Signal                | 711.51±93.88   | 655.18±78.79     |
| 3. Colgate               | 731.54±107.24  | 682.19±71.98     |
| 4. Close -up             | 917.29±76.65   | 893.79±62.81     |
| 5. Crest                 | 981.07±23.81   | 835.97±59.03     |
| 6. Astera                | 833.44±12.76   | 819.71±54.09     |
| 7. Crest\BeAD()VENTUROUS | 787.36±87.37   | 732.13±34.19     |

All countries around the world have advocated for the use of fluoride-based oral care products because of their ability to limit tooth decay [4], [18]. Results of the present study revealed that (seven) kinds of toothpaste had met total fluoride TF concentration, but not in accordance with the declared concentration (Table 1). It is worth mentioning that both total soluble fluoride (TSF) and total fluoride (TF) concentration will decrease in the toothpaste over a period of time as compared to analysis of fresh samples [11]. While in the present study, fresh samples of toothpaste were used for analysis to avoid this effect by ensuring that the manufacturing dates of all toothpaste selected were within two months of the period of analysis. The majority of toothpaste contain the fluoride in their composition as sodium mono fluorophosphate (MFP) or as sodium fluoride (NaF) which is considered active forms for controlling caries [25]. But, the TF contained in the toothpaste is not completely available as some forms of fluoride; this is may be due to link to the abrasive contained in the toothpaste formulation [26]. The optimum TSF showed concentrations below (1000ppm) which are close to the standard limit; this is a required proof as effective anti-caries toothpaste [27]. Although the soluble fluoride (TF) concentration of Sensodyne toothpaste was (1025.65±43.73ppm) in which is not less than 1000ppm and this is higher than all the other types. While the TSF concentration of Close up and Crest toothpaste was (893.79±62.81ppm) and (835.97±59.03ppm), respectively. These TSF concentrations are not within the allowed limit, but the best of other types. On the other hand, the value of TSF concentration in (Colgate, signal and Crest ADVENTUROUS) toothpaste were not within the standard limit (1000-1500 ppm) and calcium carbonate (CaCO<sub>3</sub>) as based abrasive, except (Sensodyne, Crest and close up) which is used SiO<sub>2</sub> as abrasive. This could be attributed to the fact that TSF concentration in Ca-containing toothpaste was less than the amount declared by their manufacturers

which indicate that Ca-based abrasives decrease F stability [28]. Numerous studies agreed with our result showed that incompatibility between the abrasive agent (CaCO<sub>3</sub>) and (MFP=Na<sub>2</sub>FPO<sub>3</sub>) leads to lower concentration of TSF [28], [29], [30]. Cury JA et al (2014) [24] described (CaCO<sub>3</sub>) used as abrasive, Ca<sup>+2</sup> ions react with fluoride ion (F<sup>-</sup>) creating into the toothpaste tube an insoluble salt of calcium fluoride (CaF<sub>2</sub>), which does not have anti-caries characterization, but silica(SiO<sub>2</sub>) has been used as abrasive agent which allows all fluoride added remain soluble throughout shelf life of the toothpaste. The absence of quality control has led to the existence of industrial fraud in this country. The toothpaste which is available in markets with an international brand, in fact, does not comply with the same international standards because they are counterfeit products. Recently, the growing problems at the global level in both developed and developing nations" are counterfeit and false products. These problems are more common in the "countries experiencing economic problems" though the true extent of the problem is not known. Thus, the estimated fluoride in the toothpaste is not the same concentration found in the label. Hence, there is no regulation of the actual total free available fluoride in the toothpaste in several countries [31], [32].

### 3.2. The result Tap water

Tap water's samples (supply by a directory of Kirkuk water) were taken from eight different regions and districts inside Kirkuk city, one liter is taken in a sterile glass bottle for each zone, shown sampling area inside Kirkuk city. The pH value of tap water in the study area varies from (7.5- 8.1). The result (Table 3) shows that pH values for all districts are within the limits permitted by the specification of the Iraqi and the World Health Organization. The result of E.C was ranged between (268 -422 μ s/cm) also are within the limits permitted.

Fluoride has a beneficial effect on the human health; drinking water is a single contributor daily fluoride [33]. The fluoride levels determined in drinking water samples collected from eight residential neighborhoods inside Kirkuk city, five samples from each region, the fluoride ion quantity in drinking water is shown in the Table (3). Maximum mean fluoride concentration in drinking water was found in the area of Shaterlo ranged between (0.144-0.061ppm). While minimum mean fluoride concentration in drinking water was found in the area of Almas ranged between (0.023-0.021 ppm). According to the (Bureau of Iraqi Standards) for drinking water, the maximum permissible and the recommended levels of fluoride is (1 mg. L<sup>-1</sup>) by (WHO 2002) [34], however, drinking water in some countries can contain great quantity of naturally occurring fluoride, which is higher than the recommended value reported in WHO' s Guideline (1. 5 mg. L<sup>-1</sup>). [35]

There is a report that has been published by (Abbas et al.) about the fluoride content of tap and river water, they cited that the fluoride concentration of drinking water was ranged between (0.102-0.289) ppm. While the level of fluoride in Lesser Zap river was (0.0265-0.0863) ppm [36]. Another study determined fluoride concentration of 50 samples of tap water in Babil city, the mean fluoride content was (0.184 ±

0.041). [37] The quantity of fluoride in drinking water in different parts of Baghdad city was (0.351-0.026) ppm, and in the surface waters of Tigris River from the first point of entry into the Baghdad city until arrival at the end of the track, the results showed that the fluoride ion concentration in the river ranged from (0.041- 0.025) ppm [38].

**Table 3:** Concentrations of expected fluoride measured total fluoride

| A sample of Water Collected from Municipal Water Supply. | Fluoride Concentration In (ppm) |       | pH  | E.C (μ s/cm) |
|--|---------------------------------|-------|-----|--------------|
|  | Max.                            | Min.  |     |              |
| 1. Shaterlo  | 0.144                           | 0.061 | 7.6 | 289          |
| 2. Almas   | 0.084                           | 0.072 | 7.5 | 268          |
| 3. Adhar   | 0.078                           | 0.063 | 7.7 | 285          |
| 4. Shorija   | 0.062                           | 0.048 | 7.9 | 412          |
| 5. Emam Kassim   | 0.071                           | 0.056 | 8.1 | 343          |
| 6. Almas (tapa)  | 0.085                           | 0.069 | 7.6 | 336          |
| 7. Arafa   | 0.122                           | 0.083 | 7.8 | 287          |
| 8. Shoraw  | 0.023                           | 0.011 | 7.6 | 385          |

### 3.3. The result and discussion of bottle water:

Water quality parameters of (8) types of packaged water samples were analyzed after collecting them from the markets in Kirkuk city. The Table (4) illustrates Physical properties (pH and conductivity) and fluoride concentration of packaged drinking water. The pH values for all samples ranged between (7.64- 6.63). The limit of pH values for drinking water permitted by the specification of the Iraqi and the World Health Organization. The electrical conductivity (EC) results of the samples ranged (461- 248 μ s/cm). The (EC) of water samples is an indicator of their salinity, these values shown that water salinity. The fluoride concentration of the studied samples is shown in Table (3), which showed a varied difference, ranged between (0.131 ppm) in Aqua to (0.010 ppm) in Kani. This shows that the packaged drinking waters in Kirkuk city are with fluoride deficiency when they compared with the recommended level by WHO [35]. The similar result reported by another researcher who identified low fluoride levels in five types of packaged drinking water in Kirkuk city which were ranged (0.043- 0.127 ppm) [37]. Whereas Sulaiman et al. [39] estimated the concentration of fluoride in two types of bottle water in Kirkuk city in the area of this study, the first was Hayat (0.139 ppm) and Chiaa (0.010 ppm). The fluoride levels obtained in this study are very close to those recorded in cities of Iraq, Reza et al. [40] measured F<sup>-</sup> concentrations in the packaged waters of Karbala and Najaf, they reported the concentration ranging from (0.13 to 0.5ppm), but Matloob [37] carried out a study during 2010–2011 in Babil-Iraq, finding that the average packaged water fluoride concentration was (0.073±0.066 ppm). Also, the fluoride concentration of eleven branded different types of packaged drinking water in Basra city was measured and the result showed that the concentration was less than the normal value [41]. The concentrations of Fluoride may be modestly great in some cases such as a fluoride binds to glass, decreasing the concentration of fluoride in water plastic containers and leading to a difference between the reported and the actual values. [42], [43]

**Table 4:** Physical properties (pH and conductivity) and fluoride concentration of packaged drinking water

| Brand      | Labelled Fluoride quantity (mg/L) | Measured Fluoride quantity (mg/L) | pH   | E.C (μ s/cm) |
|------------|-----------------------------------|-----------------------------------|------|--------------|
| 1. Ma'ali  | 0.02                              | 0.041                             | 7.91 | 382          |
| 2. Life    | 0.03                              | 0.073                             | 7.51 | 224          |
| 3. Masafi  | N.L                               | 0.032                             | 7.41 | 341          |
| 4. inci    | 0.04                              | 0.081                             | 7.31 | 128          |
| 5. Kani    | 0.031                             | 0.063                             | 7.2  | 236          |
| 6. Mina    | 0.07                              | 0.29                              | 7.65 | 231          |
| 7. Aqua    | N.L                               | 0.131                             | 7.42 | 202          |
| 8. jawhara | 0.08                              | 0.110                             | 6.63 | 232          |

### 3.4. The result and discussion of ground water:

Acidity Index (pH) is the negative logarithm of hydrogen ion in natural water. It has been measured directly in situ with a range of (7.61-8.10), all the water samples were within the safe limits. The results of the electric conductivity (EC) ranged between (393.60-728.87 μs.cm-1).

Table (5) shown the Fluoride quantity in samples from the groundwater in numerous places of the study area. As of the results, the fluoride concentration in the seven wells at villages outside of Kirkuk city varied between (0.292 - 0.059ppm). The highest concentration of fluoride in the studied region found at the Klawkut village with (0.292ppm), while the minimum was detected in Hassar villages (0.059 ppm). The results of groundwater show the concentrations of fluoride were very low in this area, but it seems to be better than the concentration of tap and packaged drink water in addition of toothpaste which was used in Kirkuk city. A similar low concentration has been determined in a study in 22 samples tested in wells water in different areas of Sulaimani province; average concentration was ranged between (0 - 0.157) ppm [44]. Calcium is an important constituent of bone and teeth and it is a positive ion which is attracted by fluoride in water, as fluoride is a highly electronegative element; hence it leads to developmental changes [45]. Many factors may contribute to fluoride deficiency in ground water such as the non-attendance of fluoride-containing minerals or fluoride bearing magmatic solutions in the layers through which ground water is circulating.[46]

**Table 5:** Fluoride concentration of ground waters

| Villages       | Fluoride Concentration in (ppm) | pH     | E.C (μ s/cm) |
|----------------|---------------------------------|--------|--------------|
| 1. Sakizly     | 0.074                           | 387.00 | 8,44         |
| 2. Hassar      | 0.059                           | 428.87 | 7.84         |
| 3. Goldara     | 0.292                           | 493.00 | 8.19         |
| 4. Papilan     | 0.226                           | 588.25 | 8.62         |
| 5. Jolhan      | 0.109                           | 392.60 | 7.92         |
| 6. Haji-bekhan | 0.178                           | 561.29 | 7.81         |
| 7. klawkut     | 0.312                           | 475.20 | 8.21         |
| 8. Shwan       | 0.224                           | 538.77 | 8.54         |

## 4. Conclusion

In conclusion, the toothpaste, tap water, groundwater, and packaged drinking water in Kirkuk are a shortage in fluoride

content this is when they are compared with recommended level by WHO and Bureau of Iraqi standards. Therefore, the fluoridation of tap water and packaged drinking water is necessary. As for toothpaste, this is impossible because it falls within the imported materials and is not manufactured in Iraq. Hence, quality control comes into play an important role in estimating fluoride concentration in toothpaste before it is a distribution in the market.

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