Fluoride Levels in Different Dental Pastes Marketed in Morocco

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Abstract

Toothpaste plays an essential role in maintaining oral health by helping users remove plaque and debris through its detergent action. In our modern lives, the daily use of toothpaste is common, and it can be a source of various therapeutic agents, including fluoride. Fluorine is incorporated into toothpastes primarily in the form of sodium fluoride (NaF), sodium monofluorophosphate (MFP), amine fluoride, and stannous fluoride. These fluoride compounds are added to strengthen tooth enamel, prevent tooth decay, and promote oral health.

The use of fluoride toothpastes is an effective means of preventing tooth decay. However, excessive fluoride intake over several months or years during the tooth formation period can lead to fluorosis. The aim of our study is to determine the fluoride content and chemical composition of toothpastes marketed in Morocco and the toothpaste formulations indicated in the pakets. The study included 22 toothpastes from different suppliers. 68.2% of the toothpastes met the WHO guidelines for expiry date. 77.2% of the packages indicated the nature of fluoride and 72.7% of the samples evaluated indicated the concentration of fluoride. Formulations Taking into account the requirements set by the WHO and the European Union, the results revealed that 68.2% of the toothpastes tested may have a cario-protective impact and 22.7% may present a risk of fluorosis. The results of the fluoride analyses range from 0 to 3560 ppm, with a relative deviation ranging from 2.4% to 100% compared to the fluoride values indicated on the toothpaste packaging and to the analyzed values.

The results of this study show the need for quality control of fluoride toothpastes sold in Morocco, as well as raising awareness of both the preventive role of fluoride and the danger of its excess, in order to ensure optimal use of fluoride toothpastes.

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Introduction

Recent reports in the dental literature show a decrease in tooth decay associated with the use of fluoride in dental paste. Fluoride affects the demineralization and remineralization of the hard tissues of the teeth, promoting remineralization by replacing the hydroxyl group in hydroxyapatite and forming the more stable fluorapatite $(Ca_{10}(PO_4)_6 F_2)$ which is more

*Corresponding author: Chadia Ouazzani Laboratory of Biochemistry and Molecular Biology, Faculty of Medicine and Pharmacy, Mohammed V University of Rabat, Morocco E-mail: ouazcom@yahoo.fr resistant to acid dissolution, and preventing "cariogenic" bacteria from producing acid by decreasing the ability of bacteria to tolerate acid. Its action is revealed at an acidic pH. Low fluoride concentrations of 0.1 mM can arrest the glycolysis of intact Streptococcus mutans cells in the acidic environment of cariogenic plaque ^{1–7}.

The antibacterial action of fluoride is attributed to the acidification of the bacterial cytoplasm through the formation of H+ and Fions from hydrogen fluoride, as well as to the disruption of bacterial metabolism through the inhibition of vital enzymes such as protonreleasing adenosine triphosphatase and enoase. These mechanisms disrupt the processes essential to the functioning of bacteria, thus contributing to their inhibition and the reduction of the bacterial load^{8.9}.

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However, the use of fluoridated dental pastes can be influenced by a variety of factors that can affect both benefit and therapeutic efficacy. These factors include the concentration of fluoride, the amount of toothpaste used, and individual differences, including the duration and frequency of brushing and rinsing^{10,11}.

The caries preventive effect varies with different concentrations of fluoride in the toothpaste, with higher concentrations being associated with greater caries control^{1,12}.

Toothpastes containing 1450 ppm fluoride recover enamel surface hardness ¹³. At relatively excessive concentrations, its incorporation during tooth formation into the mineral phase of the skeleton leads to pathological alterations in skeletal and dental tissues^{14,15}. The amount of fluoride can be exacerbated by other sources of fluoride, such as drinking water^{16,17}, beverages^{18,} and food¹⁹.

The use of a fluoride-containing mouthwash is recommended for children over 6 years of age who have active dental caries, as well as for patients undergoing fixed orthodontic treatment to reduce the risk of demineralization around orthodontic brackets. In addition, the use of a fluoride mouthwash is recommended for patients with decreased salivary flow and those with reduced manual dexterity^{8,20}.

It is essential to formulate a children's toothpaste to maximize the availability of fluoride, while ensuring that it is appropriately abrasive for effective cleaning. In addition, it is important to select the levels and types of flavors and surfactants to provide a pleasant brushing experience¹⁴.

Flavors that are perceived as pleasant during brushing have been shown to enhance the duration of brushing, leading to improved delivery and effectiveness of fluoride from toothpastes. By incorporating enjoyable flavors, individuals are more likely to brush for a longer period of time, allowing for greater exposure to the fluoride in the toothpaste and ultimately enhancing its therapeutic benefits. While adult toothpastes may offer benefits in the prevention of caries in at-risk children, it is important to note that these formulations may also contain higher levels of abrasive agents to meet the staining needs of adults, due to factors such as smoking and consumption of food coloring agents such as coffee and tea, which are normally not a large part of a child's diet ²¹.

Toothpastes are mostly considered cosmetic products (fluoride content below 1500 ppm) and can escape quality control. The study of the conformity of the information, the fluoride content on the packaging of toothpastes relative to international standards allows us to classify dental pastes according to their conformity and their use according to the age of the consumers. We seek to determine the fluoride content of different dental pastes marketed in Morocco from different suppliers, as recorded on the packaging and determined by potentiometric analysis in toothpaste extracts. The analysis of the dental pastes will make it possible to determine the cario-protective and excessive effects of fluorine in the population.

Materials and methods

Toothpastes were collected from pharmacies, dental practices, supermarkets, perfumeries, grocery shops and street vendors. All the toothpastes collected were in paste form and packaged in tubes of different volumes. They are obtained from different shopping locations. 95.5% of the toothpastes were of foreign origin and 4.5% of local origin. (Table 1)

Preparation of soluble crude dental paste extracts

Each toothpaste sample weighing 0.5g was carefully added to a 100ml beaker containing 100ml of distilled water. The mixture was vigorously stirred until complete dissolution occurred. Subsequently, the toothpaste extracts were meticulously prepared and transferred into polyethylene bottles for storage, ensuring their integrity and quality.

Determination of fluoride levels

The fluoride determination was carried out at room temperature using the potentiometric technique and the fluoride-specific ion electrode (HI-4110). The HI-4110 accurately measures the total fluoride content. The HI-4110 fluoride ISE combination is suitable for the detection of free fluoride in drinking water, soft drinks, wine, plants, emulsified foods, and electroplating acids.

Statistical study

In our study, we employed several statistical methods to analyze the collected data. Among these methods, Tukey's test and relative deviation were used to obtain significant information. The Tukey test allowed us to

compare the means of more than two groups for a quantitative variable and thus determine if significant differences were present between these groups. This statistical analysis was performed using GraphPad Prism 9.

In our study, we undertook a thorough comparison of the fluoride content values on toothpaste packaging with the values actually measured during sample analysis. This was done to assess the agreement between the two by calculating the relative difference for each sample. By calculating this relative deviation, we were able to determine the extent to which the actual values diverged from the values reported on the packaging. This comparative analysis allowed us to provide an accurate assessment of the compliance of the toothpastes studied and to better understand the potential discrepancies between the information provided and the observed results.

To reduce potential bias, we made the decision to exclude some samples from our analysis, namely samples 2, 3, 6, 7, 8, 10, 11, 12, and 19, because they did not include information on the fluoride level indicated on the package. By removing these samples, we were able to focus only on those with complete and comparable data, which allowed us to calculate the relative difference more accurately. This approach allowed us to obtain more reliable and meaningful results in our study of fluoride levels in toothpastes. By excluding samples without appropriate data, we were able to reduce the potential influence of missing information and ensure the integrity of our analysis.

Results

Data on the packaging of dental pastes

The results of the labeling information specified by ISO 11609:2017 (Table 1) on toothpaste packages from street vendors and grocery shops reveal a lack of display of the word "toothpaste" or equivalent, the expiry date, and the safety warning related to the use of toothpaste with a fluoride concentration of 1000 ppm/g or more by children under six years of age. Toothpaste from supermarkets and perfumeries was affected by expiration date violations. (68.2%) of the toothpastes had an expiration date, while (31.2%) had no date, the latter being from street vendors. (Table 2)

However, it is important to note that a significant portion of the toothpastes available in grocery stores, approximately 33.3%, do not have a specific safety warning for use by children under six years of age. In addition, it should be noted that this omission of the warning is also observed in toothpastes sold by street vendors.

Variation in fluoride composition quoted in dental pastes

It is interesting to note that the vast majority of toothpastes available on the market, about 86.4%, explicitly mention fluoride concentration in their composition. However, it should be noted that one toothpaste from a specific dental practice (16) is distinguished by its total absence of fluoride and the absence of information about its fluoride content.

The nature of the fluoride molecule was specified in 77.2% of the packages. The dental pastes (1, 3, 7, 11, 18, 19, and 22) and (2, 4, 5, 6,13,14 and 20) contained sodium fluoride (NaF) and sodium monofluorophosphate (NaHPO₄), respectively. The dental pastes (15, 17) from pharmacies contained both components (NaF and fluorophosphate). The dental pastes (9) supplied by street vendors and (21) contained NaF, synthetic fluorophosphate, and fluorinol (fluoramine), respectively.

Samples (1, 3, 7, 11, 18, 19, and 22) formulated with sodium fluoride (NaF) contained silica (SiO2) as an abrasive. Toothpastes (2, 4, 5, 6, 13, 14, and 20) formulated with Sodium Monofluorophosphate (NaHPO4) contained either silica (SiO2) or a combination of calcium carbonate $(CaCO_3)$ and silica (SiO₂) as abrasives. Toothpastes (15 and 17) formulated both sodium fluoride (NaF) with and (NaHPO₄), monofluorophosphate with а combination of sodium fluoride (NaF) and synthetic fluorophosphate (sample 9), and with fluorinol of grocery origin (21), contained silica (SiO_2) as an abrasive.

Variation in the fluoride content of dental pastes

72.7% of the samples have the fluoride concentration indicated on the packaging. Fluoride levels determined in toothpastes for users over 6 years of age and those for users under 6 years of age range from 12 ppm to 3560 ppm and 34 ppm to 1848 ppm, respectively. 40% of toothpastes have fluoride levels below 1000 ppm. One toothpaste (from grocery stores) for children aged 2–5 years has a fluoride content of

over 1500 ppm. The samples from pharmacies and supermarkets were intended for customers under six years of age and did not have high levels (34–408 ppm) of fluoride that could pose a risk of fluorosis. However, they did not have the concentrations mentioned (Table 3).

In terms of relative deviation, it is interesting to note that only one sample (sample 21) has a relative deviation of less than 10%, which is considered low. The specific relative deviation for this sample is 2.4%. This indicates that the measured value is about 2.4% below the expected value.

Discussion

The nature and concentrations of fluorides determined in the different dental pastes are close to those determined by Touré et al. 22, who showed a compliance of 75% in their study in Senegal. In our study, the nature of the fluoride molecule was indicated in 77.2% of the tubes, and the fluoride concentration was mentioned in 72.7% of the toothpastes. A previous study in Morocco showed the accuracy of the type of fluoride on 67.8% of the packages and the concentration used in 62.5% of the samples tested ²³. Van Loveren et al. found 77% in a larger study of seven developing countries²⁴.

According to WHO and EU standards ²⁵, 68.2% of the toothpaste samples studied could have a cario-protective effect. These results are close to those characterized by Touré et al. ²². Similar studies carried out in Japan revealed that dental pastes in Brazil met current standards (content above 1000 ppm) ^{26,27}. The most frequently used active fluorinated compound in the samples (Table 3) was sodium fluoride (n=10), followed sodium by monofluorophosphates (n=9), fluorinol (fluoramine) (n=1), and synthetic fluorphosphate (n=1). The abrasive agents used were silica (n=15), calcium bicarbonate alone (n=1), both (n=5).

The total fluoride content of the toothpaste is not fully available because some forms of fluoride may bind to the abrasive in the formulation ²⁸. The fluoride in $PO_3F_2^{-1}$ is firmly bound to the phosphate and cannot bind with soluble calcium to form insoluble calcium fluoride²⁹. Three toothpastes, 8, 10, and 12 (Table 3), did not reveal the nature of this combination, two of which did not show the

nature of the fluoride molecule and one did not reveal the nature of the abrasive.

According to the results of the study, it was found that 25% of the toothpastes sold in the streets and grocery stores were labeled with the term "toothpaste" or equivalent. In addition, analysis of labeling information for pediatric toothpastes in Pakistan and Saudi Arabia revealed significant differences. In Saudi Arabia, a large majority of pediatric toothpastes (78.9%) had warning labels, whereas in Pakistan, this figure was only 47.6%⁸.

Labeling rules, especially at street vendors and grocery shops, are not respected. Our results show that 68.2% of toothpastes meet the WHO and EU expiration date standards ^{24,30}. A study in Senegal found 75% compliance with the toothpastes analyzed ²². The studies of Van Loveren et al. ²⁴ found 77% in a larger study of seven developing countries. According to Conde et al. ³¹, these dates are very important for the storage of toothpastes, one year of storage at a temperature of 28.90 ± 1.16°C results in a significant loss of 40% of free fluorine. This instability may hamper the therapeutic effect of fluoride. This may explain the situation observed in samples 1, 5, 9, 13, 114, 15, 17, 18 and 20, where the fluoride values indicated on the package are higher than the values actually analyzed.

After analysis of the results, it is evident that only one toothpaste sample complies with the dose indicated on the package, with a measured value below the expected value of 2.4%. This sample was obtained from a pharmacy. On the other hand, nine samples (1, 5, 9, 13, 14, 15, 17, 18, and 20) have doses lower than those indicated on the package. We observed that the values measured in our study were considerably smaller than the expected values, with percentages of difference ranging from 57.68% to 100%. On the other hand, two samples (4 and 22) showed higher doses. The results of our study reveal that the measured values were higher than the expected values, with a range from 45.8% to 59.2%. These findings highlight the importance of monitoring product quality and taking corrective action to ensure consumer safety.

It should be noted that the concentration of fluoride in toothpastes can vary according to country-specific government regulations, which can make comparative studies more complex.

For example, the Food and Drug Administration (FDA) in the United States allows toothpastes containing 850 to 1150 ppm (parts per million) total fluoride for children aged two years and older and 1500 ppm fluoride for children aged six years and older ³².

Based on the recommendations, it is interesting to note that only samples 2 and 10 meet the recommended doses for children over 6 years of age but exceed the recommended doses for children 2 years of age and older. However, it is important to note that these samples are explicitly intended for children 6 years of age and older, as indicated on their Three samples labeling. exceeded the recommendations for children 6 years and older, while five samples exceeded the recommended doses for children 2 years and older. It is crucial to understand that toothpastes containing high concentrations of fluoride increase the risk of dental fluorosis in children whose teeth are still developing ¹⁴. On the other hand, it can be seen that 18 samples have values below the recommendations for children 6 years and older, while 17 samples have values below those recommended for children 2 years and older. Previous research regarding brushing habits in children under two years of age is limited. A longitudinal study in a Norwegian city examined the early oral behavior of 231 preschoolers. The results of this study revealed that 83% of mothers of children aged 18–36 months practiced regularly³³. brushing their children's teeth Statistical data also show that in the Netherlands, about 70% of children aged 1 to 2 years use fluoridated toothpaste regularly. Furthermore, in England, it was found that 78% of children start using toothpaste before they reach the age of 18 months ³⁴.

A study was conducted to assess brushing habits in children aged 12–24 months and to estimate the amount of fluoride ingested during brushing. Of the 36 parents who took responsibility for brushing their children's teeth, 69% used toothpaste. In addition, 20% of the children were found to have ingested more than 0.25 mg of fluoride per day just by brushing their teeth. These results highlight the widespread fact that children aged 12–24 months brush their teeth and use fluoride toothpaste. In addition, the amount of fluoride ingested from toothpaste may represent a significant portion of their daily fluoride intake ^{16,18,19,34,35}.

It is clear that the main problem is the ingestion of fluoride from the toothpaste. Consider sample 4, which has a fluoride concentration of 3560 ppm, equivalent to 3.56 mg/g. It is clear that this concentration can lead to fluorosis, even without considering other sources of fluoride. This applies to all samples above 1000 ppm, as the child age group is particularly vulnerable to fluoride overdoses that can lead to one or more types of fluorosis. The recommended value for fluoride intake is 0.05 mg of fluoride per kilogram of body weight, and it is that this recommendation essential be considered for children ³⁶.

It is important to note that children who are not able to spit out fluoride toothpaste should not use it. Starting at age 3, when children are able to spit out toothpaste, they can use a peasized amount of toothpaste for brushing. At this age, it is recommended that parents supervise the brushing of young children's teeth to ensure that they do not swallow the fluoride toothpaste. From the age of 6, children who are particularly prone to tooth decay can benefit from the use of fluoride-containing mouthwashes. This approach helps to strengthen cavity prevention and protect children's oral health. Commercially available toothpastes contain adequate levels of fluoride for consumers aged 6 years and older, exceeding 1000 ppm in 80% of cases. However, it is of concern that 33.3% of these toothpastes have no specific warning for children under 6 years of age, putting them at potential risk of fluorosis. It is essential that appropriate safety measures are taken to protect the dental health of the youngest children and that parents are clearly informed about the appropriate use of fluoridated toothpastes according to the child's age. Toothpastes intended for adults may have significant benefits for caries prevention in at-risk children. However, it is important to note that these formulations may also contain higher levels of abrasives to address the specific staining needs associated with tobacco use and food chromogens such as coffee and tea, which are not typically part of the diet of children²¹.

Conclusions

The results showed that over-the-counter toothpastes were the most affected by noncompliance with the relevant standards. Some pharmaceutical toothpastes for children are also

affected. The results of this study highlight the need for permanent control of toothpastes on the Moroccan market in order to ensure protection against carious infections while reducing the dangers of fluorosis. Salivary fluoride determinations will allow the diagnosis of the presence of excess fluoride and the prevention of this condition. The consumption of fluoride toothpastes should be monitored, especially in children under 6 years of age, in order to limit the consumption of fluoride-rich toothpastes and to avoid the onset of dental fluorosis.

Declaration of Interest

The authors report no conflict of interest.

| Dental pastes | Origin | Place of purchase | Target consumers (years) |
|---------------|-----------------|-------------------|-----------------------------|
| 1 | Egypt | Grocery shop | > 6 |
| 2 | PRC | Grocery shop | > 6 |
| 3 | Inde | Street vendors | > 6 |
| 4 | Egypt | Grocery shop | > 6 |
| 5 | China | Grocery shop | >6 |
| 6 | India | Street vendors | > 6 |
| 7 | India | Street vendors | > 6 |
| 8 | Egypt | Grocery shop | > 6 |
| 9 | Morocco | Street vendors | > 6 |
| 10 | Germany | Grocery shop | > 6 |
| 11 | Italy | pharmacy | > 6 |
| 12 | India | Street vendors | > 6 |
| 13 | United Emirates | Street vendors | > 6 |
| 14 | Swiss | dental practice | > 6 |
| 15 | Italy | Pharmacy | > 6 |
| 16 | Italy | Pharmacy | 0-3 |
| 17 | Italy | Pharmacy | 3-6 |
| 18 | China | BIM | 2-5 |
| 19 | Japan | Perfumery | 2-6 |
| 20 | England | Perfumery | 0-3 |
| 21 | French | Perfumery | 2-6 |
| 22 | * | Grocery shop | 2-5 |

 Table 1. Origin and characteristics of dental pastes. *not stated.

| Packaging data | Dental pastes (%) | | |
|--------------------------------------|-------------------|--|--|
| toothpaste or equivalent | 75 | | |
| trade name | 100 | | |
| manufacturer's contact details | 100 | | |
| tracing code date of manufacture | 100 | | |
| complete list of constituents | 100 | | |
| net volume (ml), or mass (gr) | 100 | | |
| expiry date | 70 | | |
| Child safety standards (≤ six years) | 33 | | |

Table 2. Variation in packaging data for toothpastes.

| Dental pastes | Fluorinating agent | Abrasive agent | F.R.P (ppm) | F.R.A (ppm) | relative deviation |
|------------------|-----------------------------------|---|----------------|--------------------------|--------------------|
| 1 | NaF | SiO ₂ | 1450 | 280 ± 2 ª | 80,69 % |
| 2 | MFP | CaCO ₃ / SiO ₂ | * | 1460 ± 8 ^b | - |
| 3 | NaF | SiO ₂ | * | 348 ± 2,6 ^d | - |
| 4 | MFP | CaCO ₃ / SiO ₂ | 1450 | 3560 ± 25 ° | 59,2% |
| 5 | MFP | CaCO ₃ / SiO ₂ | 1450 | 22 ± 0,12 ^f | 98.48% |
| 6 | MFP | CaCO ₃ / SiO ₂ | * | 224 ± 0,9 ^g | - |
| 7 | NaF | SiO ₂ | * | 12 ± 0,09 ^{f,h} | - |
| 8 | * | - | * | $260 \pm 1.3^{\circ}$ | - |
| 9 | NaF/ SYNTHETIC FLUORPHLOGOPITE | SiO ₂ | 1450 | 56 ± 0,43 ^j | 96.14% |
| 10 | * | CaCO ₃ | * | 1554 ± 16 ^k | - |
| 11 | NaF | SiO ₂ | * | 252 ± 1,2 ⁱ | - |
| 12 | * | CaCO ₃ / SiO ₂ | * | 1778 ± 19 | - |
| 13 | MFP | SiO ₂ | 950 | 0 ± 0 ^h | 100% |
| 14 | MFP | SiO ₂ | 430 | 74 ± 0,8 ^m | 83% |
| 15 | NaF+MFP | SiO ₂ | 950 | 34 ± 0.4 f | 96,42% |
| 16 | No fluorine | SiO ₂ | No fluorine | 252 ± 2,6 ⁱ | |
| 17 | NaF+MFP | SiO ₂ | 950 | 408 ± 3,5 ⁿ | 57.68% |
| 18 | NaF | SiO ₂ | 500 | 211 ± 1 ,2 ^g | |
| 19 | NaF | SiO ₂ | * | 361 ± 2,1 d | - |
| 20 | MFP | SiO ₂ | 1000 | 246 ± 2,2 ⁱ | |
| 21 | Fluorinol | SiO ₂ | 500 | 488 ± 2,9 ° | 2,4% |
| 22 | NaF | SiO ₂ | 1000 | 1848 ± 11 P | 45,88% |

Table 3. Relative deviation and Fluoride contents (ppm) of different dental pastes, fluoride salts, abrasives mentioned in dental paste formulations.

*No information; F.R.P: Fluorine rate on the packaging; F.R.A: Fluorine rate analyzed; Data presented in this study are expressed as means \pm standard error (n = 3). If there are significant differences (p < 0.05) between values, they will be indicated by different letters, as determined by Tukey's test.

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