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Difference between the Actual and Labeled Concentrations of Several Domestic Brands of Sodium Hypochlorite

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ABSTRACT

Introduction: Sodium hypochlorite (NaOCl) is extensively used in root canal treatment and its efficacy depends on the concentration of free available chlorine (FAC). This study aimed to assess the chlorine content of 10 domestically manufactured household bleach products available in the Iranian market and evaluate the effect of temperature, time and daily bottle uncapping on FAC concentration and pH of these products. **Methods and Materials:** One-liter bottles of 10 available brands of household bleach (n=4 of each brand) were collected and randomly divided into four groups (n=10). Two groups were refrigerated at 4°C while the remaining two were stored at room temperature. One group of refrigerated and one group of room temperature samples were subjected to daily bottle uncapping followed by agitation and recapping for 3 months (six times a week to simulate weekly office work). The remaining bottles remained untouched and served as controls. The concentration of FAC in each sample was measured using the iodometric titration assay, and the pH was measured using a calibrated pH-meter at baseline and 1, 2 and 3 months. The results were analyzed using the one-way ANOVA and t-test. **Results:** The mean concentration of FAC in the solutions was 4.87±0.19% at baseline. The measured concentration of sodium hypochlorite was different from the labeled value. The concentration of FAC decreased over time in all samples; the greatest reduction occurred in room temperature samples subjected to daily uncapping while the smallest reduction occurred in refrigerated, capped bottles (19% and 1.9%, respectively). The pH of all products decreased over time. The mean reduction in pH was 1.1 for the samples stored at room temperature for 3 months and 0.8 for the refrigerated samples. **Conclusion:** This *in vitro* study showed that the expected concentration of sodium hypochlorite solution made of household bleach for endodontic purposes is different from its actual concentration.

Keywords: Chlorine Compounds; Hydrogen-ion Concentration; Root Canal; Sodium Hypochlorite

Introduction

According to the surveys conducted in different countries worldwide, sodium hypochlorite (NaOCl) is the most commonly used irrigating solution in root canal treatment [1]. The concentration of sodium hypochlorite used for endodontic purposes varies from 0.5 to 5.25% [2]. The concentration of NaOCl for use in the clinical setting is a matter of individual choice for dental clinicians, which should be made by creating a balance between the expected activities (*i.e.* killing of bacteria and serving as tissue solvent) and preventing damage to dentin and the surrounding tissues [3].

The concentration of NaOCl solution indicates the concentration of free available chlorine (FAC). Depending on the acidic, neutral or alkaline pH, the available chlorine can be seen with chemical formulations of Cl_2, HOCl and ClO^-, respectively [1]. HOCl has 80 to 100 times stronger antibacterial activity than hypochlorite ion [4]. On the other hand, the tissue solving ability of NaOCl depends on its ClO^- concentration [1]. NaOCl solutions are inherently volatile and undergo self-decomposition. Thus, any change in the pH of solution affects the form of chlorine in the solution and consequently its efficacy [5]. Although hypochlorite solutions for medical purposes are available in the market, many dental clinicians worldwide use household bleach (diluted or undiluted) as endodontic irrigant [2], which has an unpredictable chlorine concentration [6]. Aside from the inherently volatile nature of this solution, factors such as environmental conditions, time, exposure to light, heat and air, contact with metals or metallic ions or organic compounds as well as the quality of packaging can negatively affect the chlorine concentration of these products [2]. A recent study by van der Waal *et al.* [7], on NaOCl solutions collected from dental offices revealed that the measured concentration of chlorine in 27% of the solutions was different from the concentration expected by dentist. On the other hand, in presence of high amounts of organic materials in the root canal system and exposure of dentin to hypochlorite, some of the available chlorine is consumed. Thus, the possibility of insufficient amount of chlorine, which would compromise its efficacy, exists especially in low-concentration solutions [4]. Many studies have assessed the effect of packaging of solution (color, make, bottle volume, head space, *etc.*) and storage conditions on decomposition of NaOCl [8]. Most of these studies, however, have been carried out on products directly obtained from the manufacturing company [6]. But, dentists often purchase these solutions from supermarkets or drugstores, which have stored the products under different conditions. Moreover, the commercially available products have a wide variety in terms of type and packaging. Therefore, this *in vitro* study was designed to *a)* determine the chlorine content of the available products and *b)* assess the effect of temperature, time and daily bottle uncapping on the concentration of chlorine and pH of 10 domestically produced household bleaches available in the market. Assessments were made at baseline and 1, 2 and 3 months.

Material and Methods

This *in vitro* study was performed on 10 domestic household bleach products commercially available in the Iranian market (Table 1). These products were the commonly used household bleaches by dental clinicians practicing in Iran. Forty 1 L bottles (*n*=4 of each brand) with similar production dates (produced 30 days earlier) were purchased from the same supermarket. The purchased bottles all had production information and the standard quality stamp and were perfectly sealed (had no leakage). The labels were removed and all bottles were coded and randomly divided into four groups (*n*=10). Two groups were refrigerated at 4°C while the remaining two were stored at room temperature away from light. One group of refrigerated and one group of room temperature samples were subjected to daily bottle uncapping (6 times a week to simulate routine weekly office work) followed by agitation and recapping for 3 months. The remaining bottles remained untouched (capped) and served as controls. The concentration of FAC and pH of the samples were measured at baseline (immediately after purchase) and after 1, 2 and 3 months by collecting 25 cc of each solution and transferring it into sterile test tubes. At least two samples of each bottle were titrated at each time point.

Table 1. Included Brands of Sodium hypochlorite

A1	Sehat bleaching liquid, 5% , Sehat Co., Iran
A2	Tage bleaching liquid, 5.2%, Tage Co., Iran
A3	Active bleaching liquid, 5%, Active Co., Iran
A4	Golrang bleaching liquid, 5%, Golrang Co., Iran
A5	Barf bleaching liquid, 5% , Paxan Co., Iran
A6	Latifeh bleaching liquid, 5%, Pak rokh Co., Iran
A7	ABC bleaching liquid, 5% , Condor Co., Iran
A8	Paknaz bleaching liquid, 5% , Tirak Co., Iran
A9	Kija bleaching liquid, 5%, Goliran Co,Iran
A10	Domestos bleaching liquid, 5%, Unilever Co., Iran

The FAC concentration was measured by measuring the active form of molecular chlorine (Cl_2) in the solution including hypochlorous acid (HOCl) and hypochlorite ion (OCl^-) with the iodometric titration assay using potassium iodide, acetic acid and sodium thiosulfate (3). The pH of the solutions was measured using a calibrated pH-meter.

Statistical analysis

Data were analyzed using SPSS version 16 (SPSS Inc., Chicago, IL, USA). One-way ANOVA was applied to assess the change in chlorine concentration and pH over time, and *t*-test was used for pairwise comparisons of the groups.

Results

The measured concentration of sodium hypochlorite was different from the labeled concentration. Household bleaches showed a mean concentration of 4.87± 0.19% (range 3.63±0.3 to 5.31±0.24%) at baseline. Among the tested brands, in only three brands the tested concentration was the same as the labeled concentration. In the remaining seven, the measured concentration was lower than the labeled concentration. Over time, the FAC concentration significantly decreased in all samples subjected to daily uncapping compared to control (capped) samples (Figure 1A and 1B). The mean percentage of reduction in FAC concentration during 3 months was as follows: Room temperature bottles subjected to daily uncapping (19%), refrigerated bottles subjected to daily uncapping (17%), room temperature capped bottles (7%) and refrigerated capped bottles (1.9%).

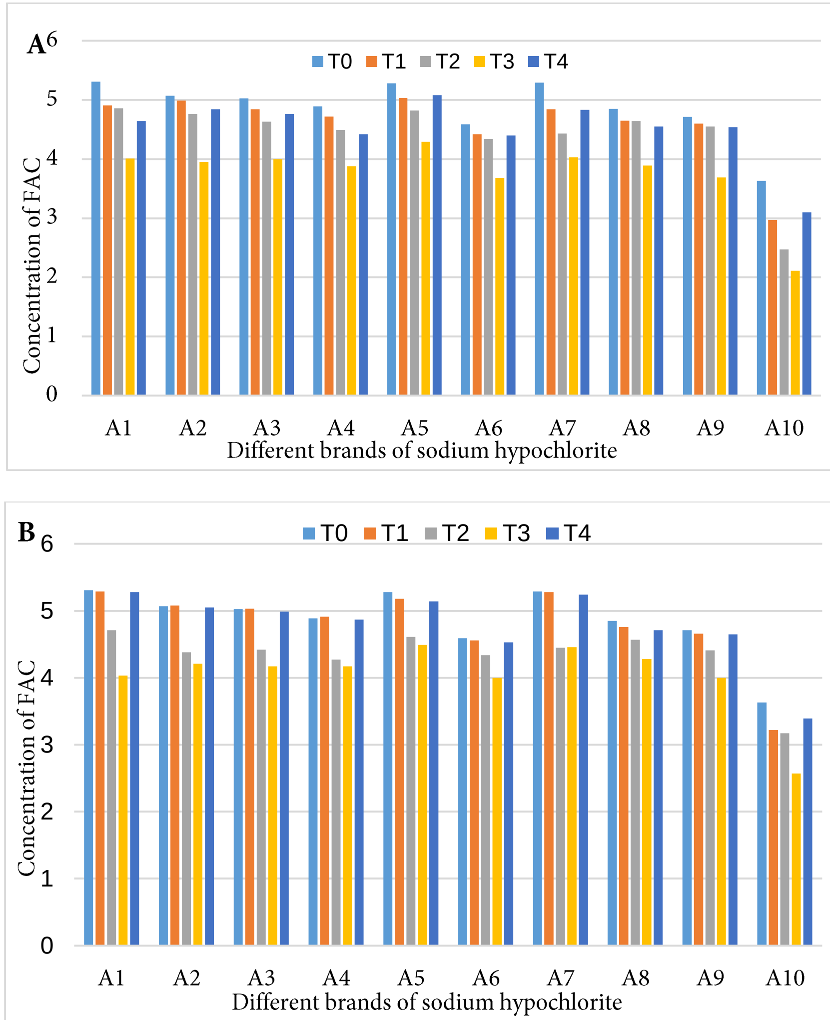

Figure 1. Measured concentration of FAC (%) by weight in sodium hypochlorite solutions stored over 3 months in room temperature (A) and refrigerated samples (B). (T0=Baseline time, T1=1 month daily uncapped bottles, T2=2 month daily uncapped bottles, T3=3 month daily uncapped bottles, T4=3 month Capped bottles)

The mean pH of different brands was 12.5±0.17 (range 12.3±0.03 to 12.9±0.03) at base line. After 3 months, the mean pH was 11.13±0.21 in room temperature and 11.4±0.19 in refrigerated samples. The pH of all samples significantly decreased over time. The mean reduction in pH was 1.1 for room temperature and 0.8 for refrigerated samples at 3 months; this difference was not statistically significant. The reduction in pH in room temperature and refrigerated samples in the first month was greater than that in the second and third months.

Discussion

In this study, 10 different brands of household bleach were analyzed in terms of FAC concentration and pH and their alterations over time. Only three brands had a FAC concentration similar to the labeled value and the remaining seven had FAC concentration lower than the labeled value at baseline. In this study, the time interval between the production date and purchase date of products was approximately one month; this time interval may explain the difference between the labeled and measured concentrations. Thus, dental clinicians that purchase a household bleach with 5.25% concentration and dilute it for clinical use, would have a solution with a much lower concentration than expected.

After 3 months, the greatest reduction of the FAC concentration occurred in room temperature bottles subjected to daily uncapping while the least reduction occurred in refrigerated capped bottles. No statistically significant difference was noted in FAC concentration of refrigerated and room temperature samples subjected to daily uncapping;

whereas, the difference between refrigerated and room temperature capped bottles was significant in this respect. It seems that daily uncapping has a greater effect than the storage temperature on the chlorine content. Also, higher storage temperature increases the degradation of solution. Braitt *et al.* [9] showed that within 7 days, 6% NaOCl solution stored at room temperature lost its active chlorine by 58.3%. However, their methodology was different from ours in that they repeatedly opened the container with 2-h intervals (8 times a day). Several other studies reported that frequent uncapping of bottles would result in further loss of chlorine [9]. Thus, considering the role of oxygen in degradation of sodium hypochlorite solution [10] and the need for frequent uncapping of bottles in endodontic clinics, sodium hypochlorite solutions are recommended to be kept in 500 cc containers [2, 9]. Guastalli *et al.* [5], in their study on hypochlorite solutions stored in screw-top containers at 20°C for 213 days reported a significant reduction in FAC concentration in the primary phase of assessment followed by a gradual reduction until the end of experiment. Another study evaluated samples stored at ambient temperature and transparent glass bottles and reported rapid FAC loss initially, which was followed by a slower trend of loss [11]. Some other researchers reported more favorable shelf-life for refrigerated samples and reported that refrigeration was the best available option [9]. In our study, the chlorine concentration of all refrigerated and room temperature samples decreased after 3 months. Time passed since the production date, packaging conditions, storage conditions in the market, environmental conditions and duration of storage in the office and dilution of solution for use in clinical setting all affect the FAC concentration. On the other hand, additives to control odor, thickening agents and surfactant may interfere with the process of dilution and result in unexpected FAC concentrations [3]. Thus, the expected concentration of sedum hypochlorite is different from the labeled concentration, which should be kept in mind when used for endodontic purposes.

The activity of sodium hypochlorite solutions is determined by their FAC concentration. Methods available for measurement of FAC include N-diethyl-p-phenylenediamine (DPD), titration against sodium arsenite, tissue solubility, ferrous ammonium sulfate analysis and iodometric titration with different end-point detection procedures including titration against sodium thiosulfate and amperometric titration against phenylarsine. The accuracy of iodometric titration against sodium thiosulfate is questionable due to the presence of breakdown products such as chlorates and chlorites, which have the potential to cause false high reading. However, it is the simplest technique widely used for this purpose. Moreover, it does not have any disposal problem or toxicity of arsenic compounds. Thus, we adopted this method in our study [2]. Alternative methods such as flame infrared emission and thermometric ammonium ion titration are suggested to decrease the interference of products that cause false high reading; although these methods are not extensively accessible [5].

Sodium hypochlorite solutions are inherently volatile in nature, and hypochlorite anions breakdown into chlorite (ClO_3^-) and chlorine (Cl^-). The speed of decomposition depends on the pH and concentration of hypochlorite. Moreover, temperature, UV exposure, oxygen exposure and presence of trace ions play a role in the kinetics of decompositions. The solutions are stable at a pH more than 11. In lower pH, their speed of decomposition depends on pH such that their peak rate of decomposition is 7. In this study, the mean reduction in pH was 1.1 in room temperature samples and 0.8 for refrigerated samples after 3 months. Although the pH of all samples significantly decreased during the 3-month period, the pH was still more than 11; this reduction in pH is not clinically important and has no significant effect on properties of the product because the dominant chlorine form (hypochlorite ion) remains unchanged at this pH. Gradual reduction in pH over time noticed in our study was in agreement with previous reports [12, 13].

Conclusion

This study evaluated 10 household bleach brands available in the Iranian market and found that the measured concentration of sodium hypochlorite was different from the labeled concentration at the time of purchase. Daily uncapping of bottles had a greater effect than storage temperature on the concentration of FAC. Thus, small-volume sodium hypochlorite solutions with disposable packaging are recommended for medical purposes. If remained capped, storage temperature significantly affects the FAC concentration, and higher storage temperatures would result in further reduction in FAC concentration. Increased storage time decreases the pH of sodium hypochlorite, although alterations in pH that occurred within 3 months in our study were not clinically significant. In conclusion, the expected concentration of sodium hypochlorite prepared for endodontic purposes is different from its actual concentration but it seems that because the concentrations of these brands are in range of therapeutic suggestion so they can be used routinely.

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Conflict of Interest: 'None declared'.

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