An Evaluation of the Accuracy of Labeling of Percent Sodium Hypochlorite on Various Commercial and Professional Sources: Is Sodium Hypochlorite from These Sources Equally Suitable for Endodontic Irrigation?

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Abstract

Introduction: The shelf life of sodium hypochlorite (NaOCl) is limited, and a previous article showed that there can be a discrepancy between the expected concentration of free available chlorine (FAC) and the actual FAC concentration in NaOCl solutions intended for endodontic irrigation. The current study investigates the FAC content of domestic and professional NaOCls and evaluates the influences of dilution and storage on FAC concentration. **Methods:** First, domestic and professional NaOCls not obtained from manufacturers were iodometrically titrated. Then, NaOCls were diluted with demineralized water or tap water and stored at 4°C or 18°C and analyzed at baseline and 2 and 22 weeks. Statistical analyses included paired samples, independent samples *t* tests and repeated multivariate analysis of variance. Correlations were calculated with the Pearson or Spearman rank correlation test. A $P < .05$ was considered significant. **Results:** Label specifications of domestic NaOCl were very imprecise (ie, <5% NaOCl). Domestic NaOCl contained 1.8%–3.5% NaOCl (w/v). Professional NaOCl varied from 14.3% relative less FAC than specified on the label to 23.5% relative more FAC than specified. After 22 weeks, the relative average loss of FAC in all conditions was 5.4% FAC ($P = .002$). Dilution, diluents, or storage temperature had no effect on the decline of FAC caused by aging. **Conclusions:** There is a great variation in NaOCl concentrations, with domestic NaOCl being the least accurate. NaOCl can be stored up to 5 months. The FAC concentration of domestic NaOCl is unpredictable, and, therefore, it appears less suitable for clinical application as root canal irrigant. *(J Endod 2014;40:2049–2052)*

Key Words

Concentration, endodontic, free available chlorine, irrigation, shelf life, sodium hypochlorite

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Sodium hypochlorite (NaOCl) is an important tool for endodontic treatment. During and after root canal preparation, the canals are irrigated with NaOCl to clean and disinfect the root canal system. For tissue dissolution, concentrations of 1% NaOCl and more are required (1, 2). The higher the concentration of NaOCl is the better the tissue-dissolving properties are (3). For disinfection purposes, the concentration of NaOCl must be at least 0.5% (4).

The percentage concentration of NaOCl in aqueous solutions refers to the weight per volume of free available chlorine (FAC), which is the hypochlorous acid (HOCl) and OCl^- form of chlorine. NaOCl is the sodium salt of HOCl. In alkaline solutions, when NaOCl is dissolved in water, the following reactions occur: $NaOCl \rightarrow Na^+ + OCl^-$ and $OCl^- + H_2O \rightleftharpoons HOCl + OH^-$.

HOCl is a weak acid, meaning that in an aqueous environment it tends to undergo partial dissociation to form a hydrogen ion (H^+) and a hypochlorite ion (OCl^-) as follows: $HOCl \rightleftharpoons H^+ + OCl^-$.

The shelf life of NaOCl is limited (5–7). Storage and exposure to light, air, metals, and organic matter can lower the FAC concentration with concurrent loss of antimicrobial and tissue-dissolving properties (1, 8–10). Previously, the stability of NaOCl has been investigated often, but some factors are still uncertain. For instance, to prevent the loss of FAC, which tends to occur in highly concentrated NaOCl solutions (11), Clarkson and Moule (12) recommended dilution of NaOCl as soon as possible after its purchase and then storing the diluted solutions. However, dilution of NaOCl can lower the pH, and at a near neutral pH the shelf life of NaOCl is very limited (6). Also, studies that analyzed the shelf life of domestic bleaches obtained their samples from the factory directly after manufacturing (8, 11). However, the dentist buys the domestic bleach in a supermarket or drugstore, and then it is unknown whether distribution and storage have had an effect on the FAC concentration.

A recent study showed that in NaOCl from dental offices, the expected or assumed FAC concentration of a NaOCl solution and the measured FAC concentration often did not match (13). In this particular study, NaOCl solutions intended for endodontic disinfection were obtained from 84 dental offices and analyzed. The participating dentists were requested to complete a questionnaire from which data were obtained about how much FAC the sample was supposed to contain, where and when it was purchased, whether the solution was ready to use or diluted by the dentist, which diluents were used, and how the solution was stored. After analyses, 27% of measured samples had less FAC than was expected by the participating dentists, and NaOCl obtained from professional sources appeared more reliable in FAC concentration than domestic NaOCl. Also, the diluted samples met the expectations of the dentist less often than undiluted samples. In this study, no conclusions could be drawn about the causes of the sometimes much lower FAC content because the baseline concentrations of the samples were unknown.

Seemingly somewhat contradictory recommendations plus the disturbing results from the previous study demanded further examination. Therefore, the purpose of the current study was to investigate the influence of source, dilution, diluents, storage, and storage temperature on the FAC concentration of NaOCl solutions used for endodontic irrigation.

Materials and Methods

Materials

For the first part of the study, 10 brands of NaOCl were included: 5 different brands of domestic NaOCl solutions were purchased from supermarkets and drugstores, and 5 professional NaOCl brands were purchased from dental suppliers and pharmacists (Table 1). Per brand, 3 containers from different batches were bought. Domestic NaOCl contained NaOCl in water without additives (thin bleaches). For the second part, of the 30 analyzed solutions, 8 were stored under 6 different conditions; each parent solution was diluted 1:1 with demineralized water (demi water) or tap water (Waternet, Amsterdam, The Netherlands; Table 2). Then, 150 mL of each solution was stored at 4°C or 18°C in the dark in a 180-mL sterile polypropylene screw cap container (Sterilin Beldico BV, Duiven, The Netherlands). The solutions were protected from light by wrapping each container in aluminum foil. The pH and FAC were determined at baseline, which was immediately after the purchase, at 2 weeks, and at 22 weeks. Dilution with demi water provided the negative control for the tap water–diluted group (14).

Chemical Analyses

After acidification of the sample with glacial acetic acid ($C_2H_4O_2$; Fluka Riedel-de Haën, Buchs, Switzerland), the FAC concentration of each sample was determined by iodometric titration with sodium thiosulfate ($Na_2S_2O_3$, Titripure; Merck, Darmstadt, Germany) (15). Each sample was analyzed 3 times, and the measurements in milliliters of $Na_2S_2O_3$ were averaged. Because 0.1 N $Na_2S_2O_3$ was titrated per microliter, the maximum errors were between $\pm$ 0.00355 mg FAC, which equals $\pm$ 0.071 g/L. Grams per liter NaOCl equals g/L FAC $\times$ 1.05.

The pH was measured with a calibrated pH meter (Radio PHM220 Lab Meter; Radiometer Analytical SAS, Villeurbanne Cedex, France).

Statistical Analyses

The data were entered into IBM SPSS Statistics for Windows (Version 21.0; IBM Corp, Armonk, NY).

In the professional NaOCl group, the difference between the specified FAC concentration and the measured FAC concentration was expressed as the relative percentage, which was calculated as follows:

$$relative\% = \frac{(measured\ FAC - specified\ FAC)}{specified\ FAC} \bullet 100$$

To correct for the 1:1 dilutions, the outcomes of the measurements were multiplied by 2. Paired samples and independent samples *t* tests were used to compare the means between the 2 groups. The repeated measures analysis of variance with a paired samples *t* test post hoc was used to evaluate the effects of source and the various handling and storage conditions on the decline in FAC caused by aging. Correlations were calculated with the Pearson or Spearman rank correlation. A $P < .05$ was considered significant.

Results

Free Available Chlorine Concentrations

According to the specification on the container, the included NaOCl solutions should contain 2%–5% NaOCl (professional) or <5% NaOCl (domestic). Figure 1 shows that domestic NaOCl brands had 1.68% $\pm$ 0.15% to 3.34% $\pm$ 0.57% NaOCl with an average of 2.46% $\pm$ 0.80% NaOCl. The average concentration of the domestic NaOCl was 2.35% $\pm$ 0.77% (w/v). Figure 2 shows the variations in FAC in the professional NaOCl. In this group, NaOCl of different concentrations was included. The relative % (difference) varied from $-14.27\% \pm 7.46\%$ FAC to $23.50\% \pm 23.97\%$ FAC. All included NaOCl solutions contained the minimal required concentration for tissue dissolution of 1% NaOCl (w/v).

All solutions (parent, diluted, stored at 4°C or at 18°C) showed a loss of FAC in time. At 22 weeks, the average reduction of all the specimens was 5.4% $\pm$ 4.8% ($P = .002$). The loss of FAC because of aging was not influenced by dilution, diluents, or storage temperature (all $P > .05$). Also, domestic NaOCl aged similar to professional NaOCl.

At 2 weeks, after correction for dilution, tap water–diluted NaOCl contained relative less 0.15% FAC than the parent solutions ($P = .021$). Also, tap water–diluted NaOCl stored at room temperature showed a relative loss of 0.06% less FAC than tap water–diluted NaOCl stored at 4°C ($P = .009$). As expected for the negative control group, these differences were not seen in the demi water–diluted groups. At 22 weeks, the differences, tap water versus demi water or parent solution, had disappeared.

The pH

The pH of the various brands ranged from 11.10 $\pm$ 0.14 to 12.18 $\pm$ 0.06 with an average of 11.84 $\pm$ 0.38. Domestic or professional NaOCl had similar pH levels ($P > .05$). The pH decreased in time to an average of 11.3 $\pm$ 0.4 at 22 weeks ($P = .018$). This decline was not significant when the parent solutions were stored at 4°C ($P = .18$). Immediately after dilution, tap water lowered the pH to an average of 11.2 $\pm$ 0.6, which was significantly lower than the parent solution ($P = .018$); dilution with demi water did not lower the pH ($P = .052$).

Correlations

There was no relationship between the baseline FAC concentration and the loss of FAC at 22 weeks. In other words, the higher concentrations did not deteriorate more than the lower concentrations. In the measurements immediately after purchase, no correlations were found between the pH and the FAC concentration; a lower pH was not correlated with a lower FAC concentration. Also, no relationship was found

TABLE 1. Included Brands of Sodium Hypochlorite

Domestic	Professional
C1000 Basis, C1000, Amersfoort, The Netherlands	Parcan; Septodont, Saint-Maur-des-Fossés, France
AH Basic, Albert Heijn BV, Zaandam, The Netherlands	Solutio natrii hypochloritis chirurgicalis 3%; Denteck, Zoetermeer, The Netherlands
Piek, Van Dam Bodegraven BV, Bodegraven, The Netherlands	Natriumhypochloriet oplossing 2%; Reymerink, Graveland, The Netherlands
Bleek, Burg Groep BV, Heerhugowaard, The Netherlands	Natriumhypochloriet 2%; Orphi Farma, Lage Zwaluwe, The Netherlands
Superschoon, CIV Superunie BA, Beesd, The Netherlands	Solutio natrii hypochloritis chirurgicalis 5% buffered; Denteck, Zoetermeer, The Netherlands