# Some factors affecting the concentration of available chlorine in commercial sources of sodium hypochlorite

**S. Frais, Y-L. Ng & K. Gulabivala**
Department of Conservative Dentistry, Eastman Dental Institute for Oral Health Care Sciences, University College London, London, England, UK

## Abstract

**Frais S, Ng Y-L, Gulabivala K**. Some factors affecting the concentration of available chlorine in commercial sources of sodium hypochlorite. *International Endodontic Journal*, **34**, 206–215, 2001.

**Aims** To evaluate the availability, effect of storage, dilution and heating on some commercial sources of sodium hypochlorite (NaOCl).

**Methodology** All pharmacies in a Regional Health Authority were telephone surveyed to establish the proprietary and nonproprietary NaOCl products available, their cost and concentration. Ninety-six freshly produced bottles of a commercially available thin household bleach (J. Sainsbury's) were randomly stored in four different modes (full 1 L bottles in dark at room temperature, half-empty 1 L bottles under the same conditions, full 200 mL bottles under the same conditions, full 200 mL bottles stored at 37 °C) up to 6 months. The available chlorine in the bleach solutions was evaluated at specific time intervals (0, 1, 2 weeks, 1, 2, 3, 4, 6 months) using iodometric titration. Four brands of commercially available bleaches (J. Sainsbury's Thin Household Bleach, Tesco's Value Bleach, Tesco's Red Label Bleach, Safeway's Savers Bleach) were diluted (by 1 : 1, 1 : 2 or 1 : 3 dilution factors) and samples taken from each dilution for titration to test predictability. NaOCl solutions (5% w/w) were heated in covered or uncovered beakers up to 60–85 °C. Samples were taken from each beaker at time 0 and each h up to 4 h, for titration.

**Results** Of the 116 pharmacies, 33% could supply a proprietary product and 53% a nonproprietary product. In all, 78% were able to supply a NaOCl product. There was no significant difference ($P < 0.05$) in available chlorine at baseline between the different modes of storage. After storage at room temperature for 6 months, there were no significant differences (3% of original; $P > 0.05$) between the different modes of storage. When the storage temperature was 37 °C, however, there was a significant difference (38% of original; $P < 0.01$) at 6 months. Comparison of baseline with the concentrations of available chlorine at 6 months showed significant differences ($P < 0.01$) for all conditions. The available chlorine concentration in four brands of 'thin' supermarket bleaches could be predictably reduced according to the dilution factor. When 5% NaOCl was heated to 60–85 °C for up to 4 h, the available chlorine was increased to 6% in covered solutions and 9% in uncovered solutions.

**Conclusions** Both proprietary and nonproprietary solutions of NaOCl should be relatively easily obtainable in the UK. Stored under appropriate conditions, commercially available thin bleaches and nonproprietary solutions of NaOCl may be diluted to obtain predictable concentrations. Heating solutions of NaOCl may cause unpredictable changes to the concentration, depending upon conditions.

**Keywords:** concentration, sodium hypochlorite, storage, temperature.

*Received 20 January 2000; accepted 8 May 2000*

Correspondence: K. Gulabivala, Department of Conservative Dentistry, Eastman Dental Institute for Oral Health Care Sciences, University College London, 256 Grays Inn Road, London WC1X 8LD, England, UK (fax: +020 79151028; e-mail: K.Gulabivala@eastman.ucl.ac.uk).

## Introduction

Sodium hypochlorite (NaOCl) is a well known bleaching and disinfecting agent. Its multiple, desirable properties have led to its adoption as the most popular root canal irrigant (Harrison 1984). Amongst its desirable properties are its universal antimicrobial characteristics (Shih *et al.* 1970, Harrison & Hand 1981, Byström & Sundqvist 1983, Byström & Sundqvist 1985), its superior ability to disintegrate organic tissue (Grossman & Meiman 1941, Senia *et al.* 1971, Hand *et al.* 1978, Koskinen *et al.* 1980, Cunningham & Balekjian 1980, Abou-Rass & Oglesby 1981, Gordon *et al.* 1981) and to denature toxins (Buttler & Crawford 1982).

The choice of concentration of NaOCl has been a matter of debate, the range extending traditionally from 0.5% to 5.25% but in recent years even further to 10% (Nakamura *et al.* 1985, Matsumoto *et al.* 1987). The precise concentration selected is based on a balance (Harrison 1984) of the perceived efficacy in bacterial killing (Bloomfield & Miles 1979, Harrison & Hand 1981, Raphael *et al.* 1981, Byström & Sundqvist 1983, Byström & Sundqvist 1985), tissue dissolution (Hand *et al.* 1978, Thé 1979, Koskinen *et al.* 1980, Gordon *et al.* 1981, Baumgartner & Cuenin 1992), toxicity to host tissue (Spångberg *et al.* 1973, Harrison *et al.* 1978, Lamers *et al.* 1980, Thé *et al.* 1980, Koskinen *et al.* 1981) and desirability to prevent damage to dentine (Grigoratos *et al.* 2000, Sim *et al.* 2000). In the absence of true evidence-based guidance from outcome studies, this array of studies leaves the option open for the clinician to select the concentration of their preference, based on their interpretation and judgement.

Whichever concentration is selected by the clinician as optimal, ultimately the effectiveness of the strategy rests on the ability to acquire a solution of the appropriate concentration.

The two main sources of sodium hypochlorite are pharmaceutical supplies or commercially available household bleaches. Some may deem the quality control of pharmaceutical sources as more appropriate for intradental use, but commercial sources have been advocated and used without significant problems for many years. Commercially available household bleach (Chlorox – 5.25% available chlorine) was first recommended by Lewis (1954), and has since gained wide acceptance (Trepagnier *et al.* 1977). Other suggested brands include, Hypo (5% available chlorine), Domex (5% available chlorine) (Piskin & Turkun 1995) and Sainsbury's household bleach (3% available chlorine) (Stock 1990). The latter source has been used in the department of Conservative Dentistry, Eastman Dental Hospital since at least 1983 without adverse incidents. Another possible confounding problem of commercially available sources is the presence of other additives such as perfumes, thickening agents and surfactants. It is possible these may interfere with the dilution process, resulting in unpredictable concentrations of available chlorine. Another potential problem with the strategy lies in the inherent instability of NaOCl solutions that can be affected by light, air, pH, organic and inorganic contaminants (Hoffmann *et al.* 1981). The shelf-life or stability of sodium hypochlorite solutions has been investigated previously (Fabian & Walker 1982, Pappalardo *et al.* 1986, Gerhardt & Williams 1991, Johnson & Remeikis 1993, Piskin & Turkun 1995, Gambarini *et al.* 1998).

Martin (1975) stated, though without any support, that the shelf-life of NaOCl was 3 months. Fabian & Walker (1982) found that low concentrations (< 1%) of NaOCl remained stable (90% of initial concentration) for up to 23 months when stored in two-thirds full, amber glass bottles potentially exposed to sunlight. Pappalardo *et al.* (1986), comparing two chlorine-containing antiseptics, found that a NaOCl solution obtained by an electrolytic process was a chloroxydiser and more stable than Dakin's solution. They found an association between the stability of pH and available chlorine concentration. Johnson & Remeikis (1993) tested the shelf-life of different concentrations of NaOCl by determining the 'mean dissolving time' for standard human umbilical cord samples. They found the dissolving time for 5.25% NaOCl remained relatively consistent over 10 weeks. However, diluted solutions did not retain stability for longer than 1 week. Piskin & Turkun (1995) measured the stability of three commercially available sources of NaOCl at different concentrations and temperatures over 200 days. The only significant reduction in available chlorine was caused by storage of 5% NaOCl at 24 °C. Cunningham & Balekjian (1980) showed warming of NaOCl to 37 °C caused a 9.5% and 4% reduction in the available chlorine for the 5% and 2.5% solutions, respectively, after 24 h.

Recently the trend for heating NaOCl solutions up to 50 °C or more has become popular (Ruddle 1995, personal communication; Berutti & Marini 1996). Gambarini *et al.* (1998) tested the stability of heated NaOCl and found the available chlorine in a commercial source declined by less than 1% when heated to 50 °C and tested up to 30 days later. This trend is

in contrast to that suggested by others, albeit at lower temperatures (Cunningham & Balekjian 1980, Piskin & Turkun 1995).

Previous studies provide some insight into the behaviour of NaOCl solutions under various storage conditions, but the findings appear to be somewhat contradictory.

The present study was initiated to:

**1** establish the availability of proprietary and non-proprietary solutions

**2** evaluate the effect of storage conditions on the concentration of available chlorine in a household bleach

**3** establish whether dilution of commercially available NaOCl solutions resulted in reduction of available chlorine predicted by the dilution factor

**4** ascertain the effect of heating NaOCl on its available chlorine concentration.

## Materials and methods

### Survey of pharmacies in a Regional Health Authority

A telephone survey was carried out in all pharmacies in the Camden and Islington Health Authority, London, UK. The purpose of the survey was explained and the information requested included:

**1** whether they would be able to provide NaOCl for irrigating root canals

**2** the names of products, minimum quantities and their cost

**3** whether the NaOCl could be provided at requested concentrations

**4** the concentrations available if those requested could not be provided

Contact numbers and addresses of the manufacturers were sought in order to ascertain the source and grades of manufacture.

### Effect of ageing and storage conditions on available chlorine concentration and pH of commercially available bleach

Ninety-six bottles (1 L) of thin household bleach (Sainsbury's 3% ± 0.2% w/w available chlorine) were supplied by the manufacturer (Jeyes Group PLC, Thetford, Norfolk, UK) and manufactured 8 days prior to the commencement of the study. The bottles were randomly divided into four groups and stored in four different storage conditions (Table 1). Each bottle was fitted with an air-tight screw cap and contained a small volume of head space to accommodate evolved chlorine gas.

The available chlorine was determined at baseline, 1 and 2 weeks, and then at 1, 2, 3, 4 and 6 months. Three bottles were used for each storage condition and time interval. The percentage of available chlorine was estimated by iodometric titration using potassium iodide, soluble starch, acetic acid and sodium thiosulphate (British Pharmacopoeia 1973). At least two samples were titrated for each bottle of bleach and the mean obtained. The pH of samples was measured using a glass pH electrode/pH meter (Jenway 3020 pH meter, Dunmow, UK).

### Effect of dilution on available chlorine concentration and pH

Two bottles of each commercially available bleach selected were obtained from suppliers (Table 2). Samples were taken from each of the two bottles of each brand of bleach and were diluted using tap water to various dilution factors (whole, 1 : 1, 1 : 2, 1 : 3). The percentage of available chlorine and pH of the solutions were estimated as previously described.

### Effect of temperature on available chlorine concentration and pH

Two bottles of undiluted NaOCl solution (Unbranded, 15% w/w of available chlorine) were supplied by Jeyes Group PLC (Thetford, Norfolk, UK). The first bottle had been stored for 49 days and the second one for 81 days from manufacture prior to experimental use. The concentrated solution was diluted to give a solution of approximately 5% by weight of available chlorine. The 5% solution was aliquoted into two 250 mL beakers. One beaker was covered with a watch glass and the other was left open. The beakers were heated on a hot-plate (Bibby HB502 Hot-plate, Bibby Sterilin Ltd, Stone, Staffordshire, UK) to

**Table 1** Modes of storage tested for their effect on available chlorine and pH

| Modes of storage |
|---|
| 1) 1 L, nonreactive, air-tight screw capped, opaque plastic bottles with a litre of NaOCl were stored in the dark, at room temperature (RT) |
| 2) Identical 1 L bottles with 500 mL of the NaOCl removed (to create head of space), were stored in the dark, at RT |
| 3) 200 mL bottles fabricated from identical material to the 1 L bottles were filled with 200 mL of bleach and stored in the dark, at RT |
| 4) 200 mL bottles were prepared as in number 3 but stored in an oven maintained at 37 °C in the dark |

**Table 2** The brands and prices of supermarket bleaches tested for the effect of dilution on available chlorine concentration

| Brand of supermarket bleach | Available chlorine concentration | Quantity | Manufacturer | Cost/L (£) | Cost per 1% of available chlorine per litre (£) |
|---|---|---|---|---|---|
| Sainsbury's Thin Household Bleach | 3% ± 0.2% | 1 L | Jeyes Group PLC, Thetford, Norfolk, UK | 0.65 | 0.22 |
| Tesco Value Bleach | 1.8% ± 0.2% | 1 L | Tesco Stores Ltd, Cheshunt, UK | 0.11 | 0.6 |
| Tesco Thin Household Bleach | 4.5% ± 0.4% | 1 L | Tesco Stores Ltd, Cheshunt, UK | 0.65 | 0.14 |
| Safeway Savers Bleach | 1.8% ± 0.2% | 1 L | Safeway Ltd, Hayes, Middlesex, UK | 0.21 | 0.12 |

between 60 and 85 °C. When this temperature range was attained, samples were taken from each beaker at time 0 and then at hourly intervals up to 4 h, for titration as previously described. Since the bleach was hot, samples for testing were weighed to avoid inaccuracies due to density changes. The pH values of the undiluted NaOCl, and that of the diluted solution after 4 h of heating were also measured.

## Results

### Survey of pharmacies in a Regional Health Authority

The results of the survey are presented in Tables 2 and 3. There was a 100% response to the survey. In all cases, the respondent was the chief pharmacist. All information was provided either immediately or within 3 days of the request. In general, more extensive product information was available when the response was delayed. Of the 116 pharmacies listed 33% could supply a proprietary product: Milton Sterilizing Fluid (Proctor & Gamble, Egham, UK) – 1% or 2% available chlorine; Chlorasol (Seton Healthcare, Middleton, UK) – 0.3% or 0.4% available chlorine; 53% could supply a non-proprietary product: NaOCl, 1% available chlorine (De Puy Healthcare, Leeds, UK) NaOCl – 12% available chlorine (JM Loveridge PLC, Southampton, UK); 78% were able to supply one of the sources of NaOCl. The cost per unit volume and per unit of available chlorine are shown in Tables 2 and 3.

Table 2 gives a comparison of the cost of commercially available bleaches. It shows that, with the exception of one of the pharmaceutical products, the cost of commercially available household bleaches is smaller.

Fifty-eight (50%) of the pharmacies were prepared to dilute solutions to required concentrations. Only seven pharmacies were able to supply the 12% solution from which higher concentrations (5%) could be diluted.

### Effect of ageing and storage conditions on available chlorine concentration and pH

The results of titration of available chlorine in the aged, stored solutions are shown in Fig. 1. The solution only became unstable over the tested period when subjected to a raised temperature. The range of pH values are given in Table 4. The changes in available chlorine were analysed using Mann–Whitney nonparametric statistics. The base-line measurements showed no significant differences ($P > 0.05$) between the solutions in the various storage conditions. There were no significant differences ($P > 0.05$) in the available chlorine between the 1 L, 200 mL and half-full 1 L bottles after 6 months stored at room temperature. However, there was a significant ($P < 0.01$) difference in available chlorine between these and the solutions stored at 37 °C in 200 mL bottles. There were significant ($P < 0.01$) differences between the baseline measurements and those taken after 6 months of storage for all conditions. There was also a significant ($P < 0.01$) difference in the available chlorine between any two sequential storage periods for the bleach stored in 200 mL bottles at 37 °C.

### Effect of dilution on available chlorine concentration and pH

The results of titration of available chlorine are shown in Fig. 2 and demonstrate that the reduction in available chlorine is consistent with the dilution factor. The range of pH values are shown in Table 5 and remain relatively consistent with dilution.

### Effect of temperature on available chlorine concentration

The effect of temperature on the available chlorine is shown in Fig. 3. The uncovered sample evaporated leaving crystals and at 4 h virtually no solution was left

**Table 3** Survey of pharmacies ($n = 116$) in a Regional Health Authority to determine availability and cost of sodium hypochlorite solutions

| Products available (% weight of available chlorine) | Number of pharmacies able to supply a given product[a] | Quantity supplied | Approximate cost per litre[c] (£) | Approx. cost per 1% of available chlorine per litre[c] (£) | Source |
|---|---|---|---|---|---|
| Milton Sterilizing Fluid (1%)[b] | 1 (0.9%) | 5 L | 0.90 | 0.90 | |
| Milton Sterilizing Fluid (2%) | 21 (18.1%) | 600 mL | 2.98 | 1.49 | |
| | | 1200 mL | 2.70 | 1.36 | Procter & Gamble, Egham, Surrey, UK |
| Chlorasol Sterilizing Fluid (0.3–0.4%) | 16 (13.8%) | 25 mL sachets (× 25) | 14.24 | 40.69 | Seton Healthcare, Stakehill Industrial Estate, Middleton, UK |
| Sodium hypochlorite solution (1%) | 55 (47.4%) | 500 mL | 2.20 | 2.20 | De Puy Health Care, Leeds, UK |
| | | 5 litre | 0.82 | 0.82 | |
| Sodium hypochlorite solution (12%) | 7 (6%) | 2 L | 2.14 | 0.18 | J. M. Loveridge PLC, Southampton, UK |

[a] Number of pharmacies able to supply NaOCl = 91 (78.5%).
[b] Number of pharmacies prepared to dilute the Milton Sterilizing Fluid (1%) solutions = 58 (50%).
[c] Price correct at the time of survey.

for sampling. In all samples, the concentration of the available chlorine increased with heating time. Because of the limited number of determinations, statistical analysis was not possible. However, for a given bottle at all time periods and for all determinations, the uncovered solution always had a higher percentage by weight of available chlorine than that for the covered solution.

## Discussion

Historically, NaOCl solutions were produced on a commercial scale by passing chlorine gas through a solution of sodium hydroxide (Brooks 1986). In more recent times, electro-synthesis of NaOCl has become standard practice. Currently, sodium hypochlorite, regardless of ultimate use, is generally manufactured by electrolysis of a sodium chloride solution. A 15% solution of NaOCl of pH 12–14 is produced by this method and then diluted by deionized water to produce solutions meeting the retailers specifications. Other agents, such as perfumes, surfactants and thickening agents, may be added at this stage. The same basic solution is therefore modified and packaged according to commercial requirements. So called thin household bleaches are usually pure NaOCl solutions with no other additives. Sodium chloride is always present as a residue of the initial solution, from which it is manufactured or as a by-product of decomposition. Some solutions have sodium chloride added to make them isotonic. The choice between the use of a pharmaceutical grade NaOCl and a household bleach is dependent on whether the handling and packaging of the latter product is considered appropriate for a solution destined to be used in contact with vital tissues. It is well known that some of these products have been used widely without reported ill effects. A cost differential is apparent between pharmaceutical and household bleach products, but is minimal. One of the products (Chlorasol Sterilizing Fluid) was supplied in 25 mL sachets, at a concentration of 0.3–0.4% and an approximate cost of £14 per litre. Details of manufacture of this product were refused by the source. Availability of a NaOCl product, whether proprietary or nonproprietary, appears to be only a limited problem in the Health Authority surveyed, although obtaining a high concentration (5%) nonproprietary product seems more of a problem. Only seven pharmacies supplied the high concentration (12%) product and one was unprepared to dilute it on grounds of safety.

NaOCl solutions are inherently unstable and on standing, the hypochlorite anions decompose to chlorate

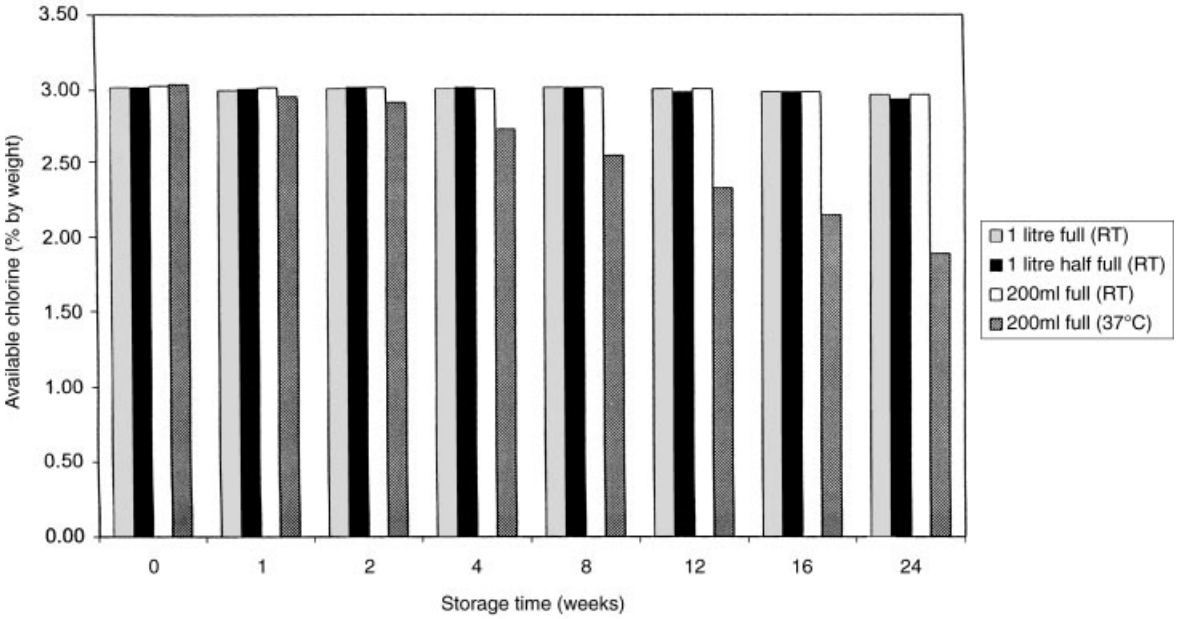


**Figure 1** Effect of storage of conditions during ageing on available chlorine (RT, room temperature).

**Table 4** Range of pH values measured during storage of sodium hypochlorite solutions

| Time | pH measured range |
|---|---|
| 0 | 12.20–12.40 |
| 1 week | Not obtained |
| 2 weeks | 12.23–12.37 |
| 1 month | 12.17–12.78 |
| 2 months | 12.21–12.76 |
| 3 months | 12.23–12.58 |
| 4 months | 12.27–12.60 |
| 6 months | 11.93–12.39 |

($ClO3^-$) and chlorine ($Cl^-$) ions. The decomposition rate is dependent on the pH and the concentration of hypochlorite. In addition, temperature, exposure to UV light and presence of trace ions are important for the kinetics of the decomposition. It has been reported that solutions are most stable above pH 11 where concentrated solutions decompose much faster than dilute solutions. Below pH 11, the decomposition rate is pH dependent and increases rapidly from pH 11–7 with a peak rate at pH 7. Based on this, it was concluded that solutions of less than 6% available chlorine with a pH 11 or higher should have an acceptable shelf-life if stored at temperatures below 30 °C (Farr *et al.* 1992).

The study on storage conditions aimed to test the influence of container volume, the presence of head space, ambient temperature and time, on available chlorine concentration. In this study, nonreactive, opaque, plastic containers with air-tight screw caps were used, as they were in the study by Johnson & Remeikis (1993). Others have used amber glass bottles (Fabian & Walker 1982, Piskin & Turkun 1995), whilst Johnson & Remeikis (1993) also tested the effect of translucent versus opaque bottles, but were unable to draw firm conclusions on their effect. The Sainsbury's Household Bleach (3% available chlorine) evaluated in this study had been manufactured 8 days prior to the experimentation and it is improbable that any major breakdown of the solution would have occurred in this time (Farr *et al.* 1992). For the 1 L bottles stored at room temperature, the percentage of original available chlorine lost over the 6 months storage amounted to 1.7%; in the case of the 200 mL bottles stored at room temperature, this was 2.0%; and for the half-full 1 L bottles stored at room temperature, this value was 2.6%. In all cases, the pH was over 11. Although for each of these storage conditions, there was a statistically significant ($P < 0.01$) difference in available chlorine between baseline and 6 months, this was not clinically significant. Under these conditions, a 3% solution of bleach has been shown to undergo minor breakdown when stored at room temperature (approximately 20 °C) even when exposed to an increased volume of air. This is in agreement with Farr *et al.* (1992) who state that a solution of less than 6% available

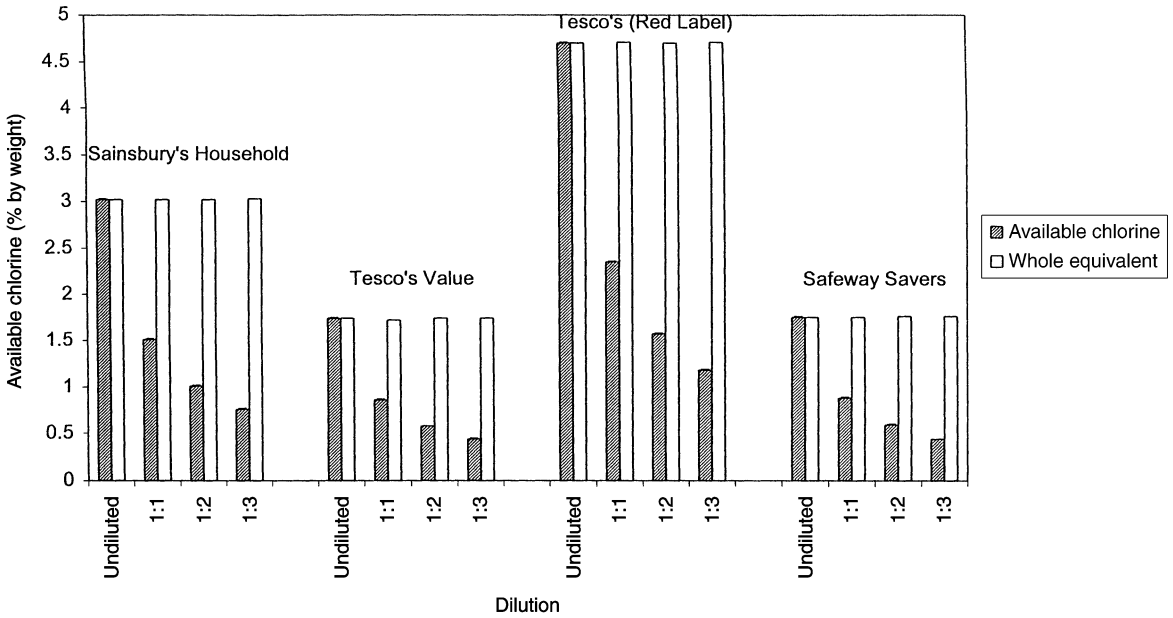


**Figure 2** Effect of dilution on available chlorine.

**Table 5** Effect of dilution on pH of sodium hypochlorite solutions

| Brand | Dilution | pH mean ($n$ = 2) |
|---|---|---|
| Sainsbury's Thin | Undiluted | 12.19 |
| Household Bleach | 1 : 1 | 12.28 |
| | 1 : 2 | 12.14 |
| | 1 : 3 | 12.15 |
| Tesco Value Bleach | Undiluted | 11.84 |
| | 1 : 1 | 11.45 |
| | 1 : 2 | 11.02 |
| | 1 : 3 | 10.76 |
| Tesco Bleach | Undiluted | 12.18 |
| (Red Label) | 1 : 1 | 12.19 |
| | 1 : 2 | 12.17 |
| | 1 : 3 | 12.05 |
| Safeway Savers | Undiluted | 11.98 |
| Bleach | 1 : 1 | 11.68 |
| | 1 : 2 | 11.20 |
| | 1 : 3 | 10.86 |

chlorine with pH 11 stored below temperatures of 30 °C has an acceptable shelf-life. However, during clinical use, there will be frequent removal of NaOCl from the bottle, and the effect of this factor is unknown.

In contrast, the 200 mL samples stored at 37 °C showed a 37.5% loss of the original available chlorine after 6 months. Again, this is in agreement with Farr *et al.* (1992). Statistical analysis showed that there was a significant ($P < 0.01$) difference in available chlorine between baseline and 6 months. In addition, although there was no significant ($P > 0.05$) difference in available chlorine between the 1 L, 200 mL and half-full 1 L bottles after 6 months stored at room temperature, there was a significant ($P < 0.01$) difference between these bottles and those stored at 37 °C. These differences were clinically significant in that the tissue dissolution properties of the bleach stored at 37 °C could be impaired (Koskinen *et al.* 1980). There was a statistically significant ($P < 0.01$) difference in the available chlorine between any two sequential storage periods at 37 °C. Cunningham & Balekjian (1980) found a 4% decrease in available chlorine of a 2.5% solution stored at 37 °C after 24 h. This is a higher rate of breakdown than would have been anticipated from the present study. After 1 week of storage at 37 °C, the 3% solution lost only 2.6% of the original available chlorine. Many other factors could account for this difference, chief amongst them may be the method of manufacture of the NaOCl solution used (Pappalardo *et al.* 1986). The solution in Cunningham & Balekjian's study was prepared by mixing sodium carbonate and chlorinated lime (Grossman 1943). After 3 months of storage, all bottles stored at elevated temperature showed the presence of a crystalline precipitate. This was due to the decomposition of the hypochlorite ions to form chloride ions. As the concentration of the chloride ions increases, the solution becomes saturated and sodium chloride crystals are formed.

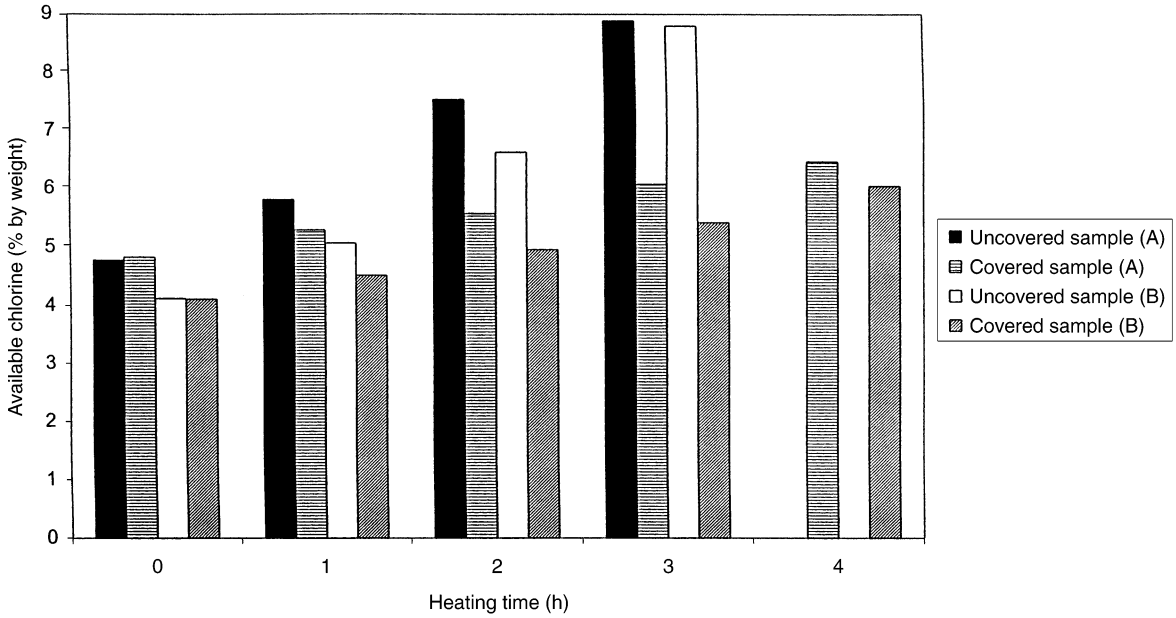


**Figure 3** Effect of heating on available chlorine.

Bleach products are stored at temperatures in the range of 15 °C to 20 °C at factories, storage depots and supermarkets. If commercial household bleaches (3% available chlorine) are purchased within 6 months of manufacture, only minor breakdown of the solution should be expected. Factors such as nature of container (nonreactive), amount of head space and total volume of solution, appear to play a comparatively small part in any degradation. The cap should be capable of forming an air-tight seal.

Dilution of all the bleaches caused a reduction in the percentage by weight of available chlorine. Consideration of the approximate whole equivalent values indicated that the percentage by weight of available chlorine of the diluted solutions could be precisely predicted using the dilution factor. Unknown ionic species which could buffer or accelerate the dilution process would appear not to be present in any of the bleaches. In view of this, where clinically desirable, the dentist could dilute a 'thin' bleach solution of known concentration accurately to the appropriate level. The maintenance of pH values above 11, except in the case of the 1.8% solutions diluted by 1 : 3, would imply stability during storage at room temperature (Farr *et al.* 1992). The impact of using tap water or sterile water was tested by Johnson & Remeikis (1993) but they reported no obvious differences. They were unable to support the observation statistically because of lack of power.

The instability of a highly concentrated solution was well illustrated in this study. One of the bottles of the '15%' solution used experimentally 81 days after manufacture had lost 14.1% of the original quoted available chlorine. The breakdown of the solution resulted in evolution of chlorine gas which caused a pressure build-up in the bottles. The bases of the opaque, plastic bottles ballooned outwards even though stored at room temperature. Following dilution to approximately 5%, the solutions were used in the study on heated NaOCl.

The merits of using heated NaOCl for root canal irrigation have been described (Cunningham & Balekjian 1980, Abou-Rass & Oglesby 1981, Berutti & Marini 1996) but its effect on available chlorine concentration has not been extensively investigated. Gambarini *et al.* (1998) tested the decomposition rates of NaOCl, heated to 50 °C (two bottles) and that unheated (two bottles). They found no differences between the two sets of conditions. They heated the bottles and allowed them to cool prior to taking the samples. The mode of heating employed clinically, is liable to be different. In the present study, the solutions were heated in a beaker (as recommended by Ruddle, 1995, personal communication), both covered and uncovered, and samples obtained of the hot solution. A temperature range (60–85 °C) was used rather than a specific temperature because in a clinical situation the former is more relevant. Accurate temperature control requires devices more elaborate than are likely

to be available clinically. After 3 h of heating, crystals began to form in the uncovered solutions, and at 4 h, so much evaporation had taken place that only a crystalline mass was left.

Figure 3 shows that the available chlorine concentration of all solutions increased with time, the amount and rate of increase was always greater for the uncovered solutions. The difference was obviously due to a greater loss of water by evaporation from the uncovered samples. Although some breakdown of NaOCl could have been expected as a result of heating, this was more than offset by the effective increase in concentration due to evaporation of water. After 3 h of heating the uncovered specimens, the concentrations of available chlorine rose to high levels (8.89% and 8.80%). Ruddle (1995, personal communication) has recommended heating a 5.25% solution in an uncovered beaker at temperatures of 70–75 °C for periods of up to 4 h. Inevitably, there will be evaporation and increases in available chlorine concentration with time. This may have important implications regarding the effects of such a solution on the physical properties of tooth tissue (Sim *et al.* 2000, Grigoratos *et al.* 2000). The mode of heating employed may vary, though some employ devices such as baby-bottle warmers to heat syringes containing NaOCl.

In conclusion, numerous factors may influence the concentration employed, from that available from retail sources to their conditions of storage, dilution, and heating. Assuming careful control over, storage (in cool, dark place/or opaque nonreactive bottles with an air-tight cap) and dilution, correct concentrations should be achievable.

## Acknowledgements

The authors gratefully acknowledge the guidance and assistance given by Dr M. Anstice (formerly of the Department of Biomaterials Science, Eastman Dental Institute) in the chemical evaluation of sodium hypochlorite. The kind cooperation of the manufacturers is also acknowledged, both in providing freshly prepared solutions and information on the processes of manufacture and storage.

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