Effect of four over-the-counter tooth-whitening products on enamel microhardness

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ABSTRACT

Objectives: This *in vitro* study evaluated the effect of four over-the-counter tooth-whitening products on enamel microhardness.

Methods: Fifty enamel blocks were prepared from extracted human molar teeth. The enamel surfaces were polished up to 1200 grit fineness and the specimens randomly divided into five groups. Enamel blocks were exposed to: Rapid White (n=10); Absolute White (n=10); Speed White (n=10) and White Glo (n=10) whitening products, according to the manufacturers' instructions. As control, ten enamel blocks were kept in artificial saliva at 37˚C without any treatment. Microhardness values were obtained before exposure (baseline) and after 1, 7 and 14-day treatment periods using a digital hardness tester with a Vickers diamond indenter. Data were analysed using Wilcoxon Signed Rank Sum Test, one-way ANOVA and Tukey-Kramer Multiple Comparison Test ($p<0.05$).

Results: Both Rapid White and Absolute White reduced enamel microhardness. Speed White increased the microhardness of enamel, while White Glo and artificial saliva had no effect on hardness.

Conclusions: Over-the-counter tooth-whitening products might decrease enamel microhardness depending on the type of product.

Keywords: Tooth-whitening, enamel, microhardness, over-the-counter, peroxide, sodium chlorite

Clinical significance: Numerous over-the-counter products are available on the market. Dentists should caution patients about the possible side effects these products might have on enamel, especially a product containing sodium chlorite due to its combination with citric acid.

INTRODUCTION

Vital tooth bleaching has become increasingly popular in recent years. Two commonly used methods for vital tooth bleaching include night-guard vital bleaching introduced by Haywood and Heymann[1] in 1989 and in-office bleaching.[2]

In the late 1990s, manufacturers introduced in the USA, and later in Europe, a new range of bleaching products available to the public for self-application. These products are commonly known as over-the-counter bleaching products, and include whitening strips or "trayless" whitening systems, paint-on-gels, gels with prefabricated trays and whitening toothpastes. Although most over-the-counter whitening products contain hydrogen peroxide or carbamide peroxide in some form as an active ingredient, sodium chlorite ($NaClO_2$) with an acid activator has also been used in some products. The latter liberates chlorine dioxide (ClO_2) in the presence of acid which results in bleaching.[3]

During the whitening process, the bleaching agent is in direct contact with the enamel surface which could result in harmful effects. Although the effects of various professional tooth-whitening products on enamel microhardness have been studied previously, the reported findings are conflicting. Some studies[4-6] reported that tooth bleaching decreased enamel microhardness, while others reported no detrimental effects of bleaching on enamel.[7,8]

However, the negative effects of over-the-counter products on tooth structure were not studied thoroughly before their introduction into the market.[3] In an *in vitro* study Zantner *et al*[3] investigated the effects of two home bleaching products and three over-the-counter products on enamel microhardness. They reported a significant decrease in enamel microhardness associated with the use of all three over-the-counter whitening products after a 14-day treatment period.

It has been reported that both the composition of the bleaching product and its pH value can affect the microhardness of enamel.[9] The use of acidic solutions also increases the risk of alterations in enamel surface.[10] Attin *et al*[11] demonstrated a significant decrease in subsurface enamel and dentine microhardness following bleaching with an over-the-counter product containing sodium chlorite and citric acid. Professional and over-the-counter whitening products containing carbamide peroxide or hydrogen peroxide effected a reduction which was limited to enamel only.

Currently, over-the-counter whitening products are widely available to the public at pharmacies, supermarkets and can be or-

Table 1: Composition of artificial saliva*	
Composition	**g/l**
Sodium carboxymethyl cellulose	10.0
Sorbitol	30.0
Potassium chloride	1.2
Sodium chloride	0.844
Magnesium chloride	0.052
Calcium chloride	0.146
Potassium dihydrogen phosphate	0.342
pH	7.0
*prepared in the laboratory (CiplaMedpro, Bellville, RSA)	

dered over the internet.[12] The risk of harmful effects on soft and hard tissues is high, because these products can be bought and used indiscriminately by patients. Therefore, the purpose of this *in vitro* study was to evaluate the effect of four over-the-counter tooth-whitening products containing hydrogen peroxide, carbamide peroxide or sodium chlorite on enamel microhardness. It was hypothesised that over-the-counter tooth-whitening products do not reduce enamel microhardness when applied according to the manufacturers' instructions.

MATERIALS AND METHODS

Specimen preparation

Freshly extracted, non-carious human molar teeth were collected and stored in distilled water with a few crystals of thymol. The roots were sectioned approximately 2-3 millimetres apical to the cemento-enamel junction using a double-sided diamond saw in a low-speed motor. The crowns were sectioned longitudinally to obtain enamel blocks of approximately 5x5mm.[2] The enamel blocks were then examined under a stereomicroscope at 25x magnification, and those with stains or cracks were discarded. Fifty of the selected blocks were individually embedded, with the enamel surface exposed, in autopolymerising acrylic studs enclosed by PVC rings. The exposed enamel surfaces of the specimens were polished with water cooled carbide paper up to 1200 grit fineness (3M, St. Paul, MN, USA), using a universal polisher (Metaserv, Betchworth, Surrey, UK). The specimens were then randomly divided into five treatment groups (1-5), with 10 specimens in each.

Treatments were performed as follows:

Group 1 (Control) (n=10):

The enamel blocks were stored in the prepared artificial saliva (Table 1) at 37ºC without any whitening treatment. The artificial saliva was replaced on a daily basis.

Group 2: Rapid White (n=10):

Firstly, Rapid White accelerator was applied to the polished enamel surface and then the Rapid White whitening gel was applied in a layer of approximately 1mm thickness for a period of 10 minutes per day for 14 days, as suggested by the manufacturer. During the treatment period (bleaching) the specimens were kept in 100% relative humidity at 37 ˚C. After each bleaching procedure, the bleaching gel was removed gently from the enamel surfaces using a paper towel. The specimens were then thoroughly rinsed and stored in the artificial saliva as per the control specimens until the next treatment. The artificial saliva was replaced on a daily basis.

Group 3: Absolute White (n=10):

Absolute White paint-on-gel was applied to the enamel blocks, using a brush and left in place for 30 minutes per day for 14 days, as suggested by the manufacturer. The rest of the procedure was exactly as for Group 2.

Group 4: Speed White (n=10):

In this group, the Speed White gel was applied to the enamel blocks and left in place for 5 minutes per day for 14 days, as suggested by the manufacturer. The rest of the procedure was exactly as for Group 2.

Group 5: White Glo (n=10):

In this group, the White Glo gel was applied to the enamel blocks and left in place for 20 minutes per day for 14 days, as suggested by the manufacturer. The rest of the procedure was exactly as for Group 2.

Microhardness measurements

Surface microhardness of the enamel was measured using a digital hardness tester with a Vickers diamond indenter. Before any treatment, four indentations were made on the polished enamel surface of each enamel block with a 300g load applied for 15 seconds to establish baseline hardness values. The artificial saliva-soaked specimens were wiped gently with a tissue paper, rinsed with distilled water and blot dried before each subsequent microhardness measurement. The indents were repeated after 1, 7 and 14 days of active bleaching treatment close to the above mentioned baseline indents (approximately 10µm).[13] All data were saved as Vickers Hardness Values (HV). Median microhardness values were calculated for the four baselines and for the post-bleaching indents of each sample and used for further statistical analysis.

Table 2: General information about the bleaching products according to the manufacturers						
Products	**Manufacturer**	**Composition**	**Treatment time**	**Total treatment time**	**Active ingredient**	**pH***
Rapid White	Rapid White products, Tonawanda, NY, USA	Accelerator: aqua, sodium chlorite Whitening gel: aqua, glycerine, carbomer 974P, polysorbate 20, citric acid, sodium hydroxide, aroma, methylparaben	10 min/day	140	Sodium chlorite	3.76
Absolute White	Dr. Fresh, Inc. La Mirada, CA, USA	Hydrogen peroxide, glycerin, SD alcohol 40-B, water, carbomer, PEG-8, triethanolamine, PEG-2M phosphoric acid, sodium phosphate, BHT	30 min/day	420	Hydrogen peroxide	3.94
Speed White	CCA Industries, Inc. E Rutherford, NJ, USA	Aqua, poloxamer 407, glycerine, hydrogen peroxide, methyl salicylate, sodium saccharin, phosphoric acid	5 min/day	70	Hydrogen peroxide	4.65
White Glo	Barros Laboratories Pty Ltd. NSW, Australia	Propylene glycol, glycerine, carbamide peroxide, carbomer 940, triethanolamine, peppermint oil,	20 min/day	280	Carbamide peroxide	6.30

Table 3: Means ± standard deviations of enamel surface microhardness values at different time intervals.

Groups	Baseline	1 Day	7 Days	14 Days
Saliva Control	341.08 ± 15.19	344.13 ± 17.64	356.25 ± 13.94	341.53 ± 18.04
Rapid White	319.80 ± 41.91	304.68 ± 40.17	296.95 ± 36.04	275.98 ± 29.53
Absolute White	313.03 ± 16.76	310.18 ± 21.98	300.45 ± 16.94	305.15 ± 18.84
Speed White	319.33 ± 27.41	325.78 ± 18.99	332.03 ± 16.88	338.48 ± 20.65
White Glo	325.50 ± 28.52	316.48 ± 25.96	334.80 ±26.96	325.18 ± 30.01

For each group baseline and post-bleaching microhardness values at days 1, 7 and 14 were compared using the Wilcoxon Signed Rank Sum Test, significant at $p<0.05$.

For multiple comparisons, differences in Vickers microhardness values were calculated between measurements at baseline and at 1, 7 and 14-day post-bleaching. The microhardness data were then analysed using the Kruskal-Wallis one-way ANOVA, followed by the Tukey-Kramer multiple comparison test for differences amongst the different groups (significance level was 5%).

The pH measurement

The pH of three samples of each over-the-counter bleaching product was also measured using an Orion Expandable Ion Analyser EA940, with a Sure-Flow®, Epoxy-body combination pH electrode (Thermo Fisher Scientific Inc., Beverly, MA, USA).

RESULTS

Table 2 gives the composition, treatment time, active ingredient and mean pH values of the different bleaching products.

Table 3 shows the mean and standard deviation of the microhardness values at different time-intervals.

Figure 1 depicts the box-and-whisker plots of the median Vickers microhardness differences between the baseline, and the 14 day post-treatment hardness values for the different groups. In each diagram, the top line shows the maximum and the bottom line the minimum hardness values. The box shows the location of 50% of the values and the line in the box the median hardness value for a specific group.

Paired comparisons between the hardness values at baseline and after 1, 7 and 14 days of bleaching treatment were carried out using the Wilcoxon Signed Rank Sum test to evaluate the effect of treatment over time within each group. Whitening treatment with Rapid White showed a significant reduction in enamel microhardness from baseline to 1, 7 and 14-day time periods ($p<0.05$). A statistically significant difference in a reduced microhardness was observed for Absolute White group at 7 day treatment only but not at 14 days. White Glo group showed a statistically significant initial reduction in enamel microhardness from baseline to the 1 day treatment period, but no significant differences were found, however, from baseline to the 7 and 14-day treatment periods.

Multiple comparisons amongst different treatment groups demonstrated the following results. Rapid White group showed higher reduction in enamel microhardness after 1 day of bleaching treatment than other groups, and differed significantly from the saliva control, Absolute White and Speed White groups (Tukey-Kramer Multiple Comparison Test, $p<0.05$). After 7 days of treatment, Rapid White and Absolute White differed significantly from all other groups ($p<0.05$). However, after 14 days of bleaching treatment, only Rapid White group differed significantly from all other groups showing higher reduction in enamel microhardness ($p<0.05$). Speed White group showed an increase in enamel microhardness after 14 days of treatment, and differed significantly from the Rapid White and Absolute White groups ($p<0.05$).

DISCUSSION

Microhardness determinations give a reliable indication of changes in the mineral content (de- and re-mineralisation) of enamel or dentine.[11,14] Microhardness experiments have been used to evaluate the effect of whitening products on tooth structure and restorative materials.[13,15,16] The American Dental Association also recommends that enamel hardness should be evaluated to ensure that exposure to tooth-whitening products does not produce substantial changes in the structure and/or properties of enamel when applied.[17]

Human enamel exhibits large regional variations in its structure and consequently, hardness of enamel may vary from area to area.[18] Therefore, it has been suggested previously that when investigating the microhardness of enamel pre- and post-treatment indents should be done close to each other (within 10µm).[19] This is even more important when the changes in hardness are small and could be easily masked when different areas on enamel are used.

In the control group, enamel blocks were stored in artificial saliva for the whole period of the experiment (14 days) without any bleaching treatment. The control group showed almost no change in enamel microhardness over 14 days. This finding demonstrated that the artificial saliva solution did not affect the hardness of sound enamel either positively or negatively, and could therefore be rightfully used as a soaking medium in the experiment.

In this study, the general trend was a reduction in enamel microhardness for Rapid White and Absolute White after 1, 7 and 14 days, but an increase for Speed White, while White Glo and the saliva control did not affect the enamel hardness (Table 3). However, only Rapid White showed statistically significant reduction in enamel microhardness after 14 days of active bleaching treatment. Rapid White is a non-peroxide whitening product containing sodium chlorite and citric acid, among other ingredients.

The findings are in agreement with the results of previous studies.[3,9] Attin *et al*[9] reported a significant decrease in subsurface microhardness of enamel and dentine with Rapid White as compared with the other products tested containing carbamide peroxide or hydrogen peroxide. Zantner *et al*[3] also reported a significant decrease in enamel microhardness, and increased surface cracks along enamel prisms following treatment with a product (Odel-med3 Beauty-Kur) containing components similar to those of Rapid White. Absolute White was the second product which resulted in relatively more reduction in enamel microhardness as compared with White Glo, Speed White and saliva control groups. Significant enamel demineralisation has been reported for pH levels of 5.2-5.8 and lower.[20,21] Of all the products tested, those with the lowest pH levels (Rapid White and Absolute White) showed

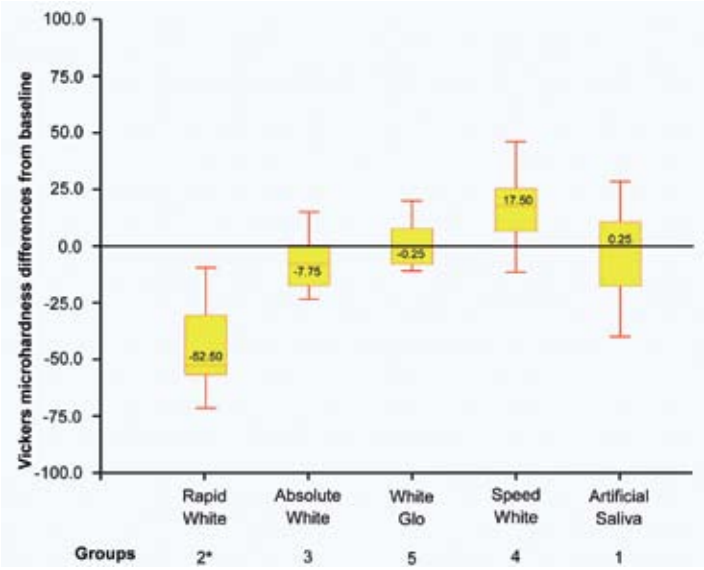

Figure 1: Box-and-whisker plots of the Vickers microhardness differences from baseline for the control and treatment groups after 14 days of treatment. Group 2 (Rapid white) that differed significantly from the control (artificial saliva) group and other treatment groups is marked with an asterisk (*).

the most damage to enamel, independent of the treatment period and chemical composition.

The exposure time could be another factor which could have influenced damage to the enamel. However, in this study it does not seem to have a major influence. Absolute White with a longer treatment time (420 minutes over 14 days) showed less damage to enamel than Rapid White with a shorter treatment time (140 minutes over 14 days). Grobler *et al*[13] reported that lower peroxide concentrations applied for longer treatment periods resulted in more damage to enamel than higher peroxide concentrations applied for shorter time periods. Treatment with Speed White showed hardening of enamel, which could be a result of the combined effect of the short treatment period (70 minutes), the composition, and the storage in artificial saliva containing calcium and phosphate ions. It is known that saliva containing these ions plays an important role in the remineralisation of enamel[22] and re-hardening of softened enamel.[3,23-25] However, remineralisation studies of initial enamel lesions in artificial saliva demonstrated that the regaining of microhardness was dependent on the storage period.[24] Furthermore, softening of enamel as a result of acid attack as seen with Rapid White could be so serious that it cannot be repaired within a short time in clinical situations. This makes enamel more susceptible to surface loss due to abrasive influences such as tooth-brushing.[26,27]

Microhardness experiments require a flat polished surface in order to make indentations and to measure them. However, there is no standardised method of grinding and polishing the specimens. Polishing of the enamel surface removes the resilient hyper-mineralised layer, thus making the enamel more prone to the softening or demineralisation effect of bleaching products.[3] Therefore, in the present study, a very small area was prepared for microhardness measurements to prevent the excessive loss of surface and subsurface enamel.

The number of whitening products on the shelves is increasing day by day and the majority of over-the-counter products differ in their chemical composition, preparation and application methods. The overall effect of the different whiteners on the enamel hardness requires at least laboratory investigations before being introduced into the market.

CONCLUSIONS

1. Over-the-counter tooth-whitening products might adversely affect enamel microhardness, depending on the type of product.
2. Rapid White containing sodium chlorite in combination with citric acid reduced enamel microhardness significantly, possibly due to its low pH.
3. Speed White showed a significant increase in enamel microhardness after 14 days of treatment.
4. Acidity and lack of control over the use of over-the-counter whitening products can predispose enamel to excessive damage.

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