STUDY OF THE EFFECT OF H$_2$O$_2$ USED IN WHITENING TREATMENTS ON HUMAN ENAMEL WEAR RESISTANCE

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Abstract: Tooth whitening treatments are based on the application of bleaching agents on the teeth surface, which can impair teeth health, due to its effect on enamel properties. The present work has the purpose of studying the effect of pH of H$_2$O$_2$ solutions on wear resistance of teeth. Friction coefficient, enamel microhardness, morphology and topography were also evaluated to better understand the induced changes. Three pH values (2, 4 and 6) of H$_2$O$_2$ solution were tested. First, human molars and premolars were disinfected, polished, cut in four equal pieces and darkened in a black tea solution. After, the teeth samples were bleached in 30% H$_2$O$_2$ solution with the different pH values. Vickers microhardness, surface roughness and wear measurements were performed before and after bleaching. The results showed that the enamel wear resistance depends on the solution pH: the lowest wear resistance was obtained for the sample bleached with H$_2$O$_2$ at pH 2.

Key words: Hydrogen peroxide, enamel, microhardness, surface topography/morphology, wear resistance

1. INTRODUCTION

Teeth whitening is a highly desirable aesthetic dental procedure that is frequently requested by people to remove teeth stains to have a whiter and perfect smile. These treatments are usually performed by the application of peroxides on the tooth surface. Peroxides are chemically active components potentially able to induce substantial alterations in dental enamel properties which can arise from the oxidative effect, pH and concentration. They may induce changes in the enamel composition and surface morphology, leading to erosion, demineralization and increased porosity [1, 2]. This may result in a decrease in the enamel microhardness and wear resistance [3, 4].

The peroxide pH values play an important role in enamel surface integrity due to the fact that low pH peroxides may increase the adverse impacts on the tooth structure. [2] Indeed, according to some authors, enamel erosive pattern is more likely to happen when bleaching agents with low pH are used. The acidity of these products is believed to be the main cause for the demineralization effect. A pH value below 5.5 has been reported to be the responsible for enamel dissolution. Values of pH below this critical point are also likely to cause increased wear and surface roughness of the enamel [3–5].

The main goal of this work is the study of the pH effect of 30% hydrogen peroxide in enamel wear resistance, relating it with the changes in microhardness, morphology and topography of the surface. Three pH values (2, 4 and 6) were chosen in order to evaluate eventual differences. Since demineralization only occurs for solutions with pH below 5.5, pH=6 was chosen to investigate how enamel properties would be influenced in the absence of demineralization. There are few systematic studies about the effect of pH on the enamel properties, which makes the obtained results relevant for the optimization of bleaching treatments, concerning the pH of the products used. The study of the

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mechanical and tribological properties of enamel gives an overview of the harmful effects of hydrogen peroxide on enamel.

2. EXPERIMENTAL

Five human molar teeth were disinfected with 1.0% chloramine-T trihydrate solution, washed with a toothpaste and stored in distilled and deionized (DD) water at 4°C, so that enamel does not suffer changes in its physical and chemical properties. Then, teeth were sequentially polished with two silicon carbide sandpapers of 1000 and 4000 mesh. To get a smooth surface free from scratches, a polishing with diamond spray (6 µm, 3 µm), lubricant and two polishing cloths was performed. The teeth were cut in four similar pieces in a microtome (Struers Accutom–50), each one containing a cusp. Before bleaching, the colour of each part of the five teeth was determined using a spectrophotometer (EasyShade 2.0). To have all teeth with the same initial coloration, they were darkened in a black tea solution for 48 hours (2 g of black tea for 50 mL of water). Finally, the measurement of the colour of all parts was performed again.

Teeth samples were divided in four groups (five parts each, all of different teeth) and bleached in 30% H$_2$O$_2$ solutions with different pHs. To prepare the 30% hydrogen peroxide solution at pH=2, a solution of 35% hydrogen peroxide was diluted with DD water. Solutions with pH 4 and 6 were obtained, adjusting the pH by adding small amounts of NaOH 1 M and/or 0,2 M solutions. The control group (containing one of the four parts of each tooth) did not suffer bleaching. The remaining groups suffered bleaching with the H$_2$O$_2$ solutions at different pHs. The bleaching treatments with each pH solution included 11 bleaching sessions until the same degree of whitening was reached. Each bleaching session consisted of 10 minutes of tooth immersion in solution with exposure to blue light (Optilux 501) each 2 minutes, for 30 seconds, to activate the peroxide (Figure 1). After each session, the colour was measured again. The control group (no bleaching) was kept for comparison.

![Figure 1. Blue light exposure scheme.](image)

Microhardness tests were conducted in a microdurometer (HSV–30 Shimadzu model) with an applied load of 1.96 N for 15 seconds.

Ball-on-plate reciprocating wear tests were performed for 20 min (2600 cycles) in a nanotribometer (CSM Nano Tribometer) using a zirconia ball of 3 mm diameter as counter-body, sliding distance of 1 mm/cycle, vertical applied load of 25 mN and frequency of 2.12 Hz.

The enamel topography/morphology was analysed by atomic force microscopy (AFM Nanosurf Easyscan 2) and scanning electron microscopy (Hitachi S2400 microscope). The average surface roughness (Ra) was determined from 10×10 µm$^2$ AFM images using the software WSxM 4.0.

3. RESULTS AND DISCUSSION

Microhardness

A decrease in the enamel microhardness values was observed after the several bleaching sessions with H$_2$O$_2$ solution at pH=2. The H$_2$O$_2$ solution at pH=4 also led to a decrease in the microhardness mean value. However, this decrease was lower than the one observed with the H$_2$O$_2$ solution at pH=2. There were almost no differences before and after bleaching with H$_2$O$_2$ solution at pH=6.

The mean overall microhardness values of the samples for all experimental conditions are shown in figure 2.
According to the results, the pH of the bleaching solution influences the final enamel microhardness, especially for low pH values. This is in agreement with the work of Xu et al. [4], that observed significant alterations in enamel microhardness after bleaching with hydrogen peroxide at low pHs: pH values of 3 and 5 induced an increase in the enamel erosion rate, reducing the enamel microhardness, but the neutral or more alkaline values (pH 7 and 8) did not affect it.

It can be concluded that the acidity of hydrogen peroxide is the main cause for the demineralization of enamel.

**Surface morphology**

After bleaching, it was observed that surface roughness decreased for all the groups (Figure 3). The control group showed the highest surface roughness, followed by the treatment with H$_2$O$_2$ at pH=2. The treatment with H$_2$O$_2$ at pH=4 presented the lowest mean surface roughness value.

The low pH of the bleaching solutions affects significantly the enamel structure, promoting a softening of the dental hard tissue, decreasing enamel surface roughness values [6].

Figure 4 shows the enamel surface morphology before (a) and after bleaching with pH 2, 4 and 6 (b, c and d, respectively).

The enamel surfaces were prepared by a mechanical polishing process described in section 2. After polishing, a smear layer made of agglomerated particles with delaminated areas was observed on the samples surface (Figure 4 a), thus leading to the highest roughness values (Figure 3).

The surface of the samples treated with H$_2$O$_2$ (Figure 4 b, c, d) presents different roughness due to the action of the pH (pH <5.5 induces demineralization). In the case of the pH=6 group, the difference lies
only in the peroxide oxidizing effect, which maybe compromises the organic matrix of enamel, influencing also the final surface roughness.

![Figure 4. SEM images of the enamel surface before and after bleaching with H₂O₂ at different pH values. The red arrows depict the formation of localized erosion zones after de attack with H₂O₂ at pH=2. a) control (no bleaching); b) bleaching at pH=2; c) bleaching at pH=4; d) bleaching at pH=6.](image)

After bleaching treatment, it was observed a decrease in the surface roughness values for all the groups.

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SEM images of the control group (Figure 4 a) shows some delamination and particles on the surface, thus leading to the highest roughness values.

For pH=2 group (Figure 4 b)), the surface roughness decreased due to the action of the aggressive pH, which smoothed surface. However, is possible to observe that the surfaces present pits and randomly distributed defects throughout the entire surface, with a significant formation of localized erosion zones (red arrows in Figure 4 b).

Regarding bleaching at pH=4, the surface roughness decreased even more, giving rise to the smoothest surface due to a significant flattening of the surface. (Figure 4 c)). For these samples, some pitting was also observed but not sufficient to increase roughness compared to the other pH bleaching treatments in this study.

Finally, bleaching with H₂O₂ at pH=6 also decreased enamel surface roughness compared to the control group. However, in this group, the average surface roughness was higher than at pH=4, presumably because the effect of the pH was not strong enough to smooth the surface as pH=4 did.

**Tribology**

The mean friction coefficient values for all experimental conditions are shown in figure 5.

The mean sliding friction coefficient assumed higher values after bleaching with solution at pH=2. The absence of bleaching treatment (control group) led to the lowest value of the friction coefficient.
Since tooth enamel is a brittle structure, when wear tests are performed and as reciprocating sliding goes on and wear increases, micro cracks and delamination processes are gradually initiated on the enamel surface due to fatigue processes. These cracks propagate rapidly with the increase of test cycles and the decrease of pH. Subsequently, hard and brittle debris of enamel which are attached to the contact interface, act as rigid abrasives, increasing friction coefficient (Figure 5) [7]. This phenomenon is more evident at pH=2, where the friction coefficient assumes higher values.

Figure 6 depicts the wear rate before and after bleaching treatments. The wear rate was higher at pH=2, which allows stating that bleaching with acid solutions leads to the decrease of wear resistance of enamel. The lowest values were obtained for pH=4 and 6.

During the wear tests, the contact between the counter-body asperities and the enamel surface, promotes the releasing of hydroxyapatite crystals wrapped with glycoproteins [8]. These glycoproteins (essentially collagen) act as binders, promoting the formation of agglomerated hydroxyapatite particles at the surface of the tooth, forming a compact layer, also known by tribo-layer. Thus, enamel wear is determined by the way this tribo-layer is formed, which depends on the number of particles that are released from the surface and how the various particles adhere to each other during wear processes. This layer is the main responsible for the enamel protective effect against the mechanical action of the wear sphere. This compact layer is destroyed over time as the wear tests progress. The denser the layer, the lower the tendency for it to fracture, and the lower the wear [9].
4. CONCLUSION

The study of pH on enamel properties is very important and contributes to the knowledge and development of a different set of conditions that should be considered when performing bleaching procedures. According to the results, it can be stated that the pH of H$_2$O$_2$ affects the enamel composition and structure:

1. The lower the pH values of the solution, the greater the changes in the enamel microhardness after the bleaching treatment. A significant microhardness decrease was observed for pH=2.
2. Surface roughness is affected by the pH of the H$_2$O$_2$ solution used. After bleaching, decreased roughness occurred for all the groups.
3. The friction coefficient increases after bleaching treatments. The highest increase was observed for pH=2.
4. Enamel wear rate is higher for low pH values, meaning a lower wear resistance. Enamel wear resistance was lower for pH=2.

In conclusion, pH=2 leads to the most adverse effects on enamel. Low values of pH shall be avoided in bleaching treatments in order to maintain the good health and performance of the teeth.

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