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Hardness, friction and wear studies on hydrogen peroxide treated bovine teeth

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ABSTRACT

A possible problem with peroxide based tooth whitening is the loss of tooth hardness and higher susceptibility to enamel surface wear. This study focussed on the effects of acidic and neutral hydrogen peroxide solutions (6 and 30% w/v) on hardness, friction and wear of bovine enamel. The experiments showed that treatments with neutral peroxide reduced wear and the loss of enamel hardness up to 2–3 times. In addition, further investigation on remineralisation with amorphous calcium phosphate showed an increase in hardness after treatment. Friction coefficients of teeth against steel varied between 0.25 and 0.7, and wear coefficients ranged between $\approx 10^{-6}$ and 10^{-7} $mm^3/N\,m$. From this study, it is possible to explain the wear behaviour of HP treated enamel with changes in hardness.

1. Introduction

The tooth is the strongest, hardest and toughest organ of the human body. It consists of four major tissues: enamel, dentine, cementum, and pulp [1]. The enamel is the outermost tooth structure and forms a protective coating on the tooth surface. Enamel can be considered a functionally graded natural composite with anisotropic mechanical behaviour, made up of 96% inorganic hydroxyapatite ($Ca_{10}(PO_4)_6(OH)_2$) crystals and 4% organic compounds and water [1–3].

Tooth whitening, or bleaching is a growing industry where consumers demand a whiter smile. Aesthetic dental treatments have seen an increasing demand as consumers have become more conscious about their image in the society [4]. In tooth whitening, peroxide based agents are normally applied to the teeth in order to remove the discolouration of the teeth that builds up over time. One of the most commonly used bleaching agents is hydrogen peroxide (HP) [5].

The exact mechanisms of tooth whitening, using oxidising materials such as HP, have not been fully understood and are still under scientific debate [5–8]. Most publications in the scientific literature suggest that peroxide initially diffuses through the enamel, reaches the dentino-enamel junction (DEJ) and finally enters the dentine [6,9,10]. During this process of diffusion, chromophores such as polyphenol compounds, within the tooth structure react with free oxygen radicals from the peroxide and oxidise, leading to a reduction in the colour and an increase in the whiteness [11]. More recently, significant effects of bleach on the opalescence and translucency of enamel have been found, which prompted speculations that optical scattering of enamel may partially contribute to the observed whitening responses [8].

A possible problem of peroxide based tooth whitening treatments is the loss of tooth hardness and higher susceptibility to enamel surface wear, both of which can jeopardise tooth health [6,12–15]. Lawson et al. [14] and Lucas et al. [16] highlighted the physiological parameters of intraoral wear associated with mastication. The fundamental mechanisms underlying tooth wear have been reviewed by d'Incau et al. [17] as well as Arsecularatne and Hoffman [18]. Although, there is a plethora of information available on the friction and wear behaviour of enamel and dental materials [19–26], there is limited quantitative experimental data on the wear behaviour of peroxide treated teeth. Moreover, there is inconsistent data available on the effect of hydrogen peroxide on enamel hardness. Especially, the influence of the pH of peroxide solutions on tooth hardness changes and wear has largely been unexplored [27]. According to reviews conducted by Attin et al. [13] and Joiner [6] on the effect of peroxide on enamel and dentine properties, there are conflicting results between the findings of different researchers on the impact of bleaching agents on the microhardness of the enamel. Studies which simulated the intraoral conditions, by using either human or surrogate saliva, showed reduced risk of enamel microhardness decrease due to bleaching [6,13].

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This study was focussed on gaining better understanding of hardness, friction and wear behaviour of tooth enamel undergoing repeated whitening treatments. This research also included studying the effects of bleaching with peroxide solutions of neutral pH, and using a remineralising solution of amorphous calcium phosphate (ACP), on enamel surface hardness post whitening treatment. The objectives of this study were (a) to investigate the effect of repeated peroxide (6% w/v and 30% w/v HP, pH 3.9 and pH 2.7) treatments on enamel hardness and wear, and (b) to study enamel hardness and wear after peroxide treatments with mouth-neutral, pH adjusted peroxide (pH 7.1), and remineralisation treatment with amorphous calcium phosphate.

2. Materials and experimental methods

2.1. Bovine teeth

The study protocol was reviewed and approved by the Internal Committee Biomedical Experiments of Philips Research (ICBE2013-41-1795). Nerve-free and BSE-free bovine incisors were procured from Intertek, UK. Prior to sample preparation, the teeth were stored in a refrigerator set at 4 °C and allowed to dry under a fume hood for 24 h. Before commencement of experiments, the teeth were mounted in cylindrical blocks of resin (height: 15 mm, diameter: 20 mm) to carry out hardness and wear tests. After resin embedment, the labial surface of teeth underwent a series of grinding steps, and a final polishing step to render them smooth and plane-parallel for the hardness and wear tests. The resin-free area was about 5–6 mm in diameter, and approximately 0.5–0.7 mm of each specimen was ground and polished off to obtain a flat enamel surface. After polishing, the teeth had an average surface roughness R_a between 15 and 25 nm, and each specimen was viewed under a light microscope (Leica) at 20 × magnification to check for the presence of an intact enamel surface.

2.2. Chemicals

Hydrogen peroxide was used as a bleaching agent. Hydrogen peroxide (60% w/v) stock solution was obtained from Fisher Scientific UK (Code: H/1862/15). The stock solution was diluted with distilled water to prepare 6% w/v and 30% w/v HP solutions, typical concentrations for over-the-counter (at-home) and in-office whitening treatments [7,28,29] that were used to treat bovine teeth. The pH of both solutions was measured using a pH probe and found to be 3.9 and 2.7 for 6% w/v and 30% w/v HP solutions, respectively. Potassium hydroxide (KOH) pellets (BioXtra, ≥85% KOH basis) were obtained from Sigma-Aldrich (P5958-500G). A 1 M KOH solution (pH 13.3) was used to adjust the pH of 6% w/v and 30% w/v HP solutions to neutral pH (pH 7.1). For remineralisation treatments, ACP was prepared by mixing 1 part of 15% calcium nitrate solution to 1 part of a 6% sodium phosphate solution directly onto the enamel surface. To create a realistic intraoral environment while testing the wear behaviour of teeth, a moisturising mouthwash solution containing various enzymes present in human saliva (Biotene triple enzyme, GlaxoSmithKline) was used. The Biotene solution has a viscosity of 15.2 mPa•s at 21 °C and shows Newtonian fluid behaviour over a shear rate 1–1000/s.

2.3. Enamel hardness measurements

Enamel surface microhardness was determined using a Leica VMHT MOT Vickers indenter. Hardness tests were carried out with a normal load of 1.96 N, dwell time of 30 s and an indentation speed of 45 μm/s. Pre-tests on peroxide treated teeth showed that the maximum indentation depth of the Vickers indenter was <20 μm. According to Chuang et al. [30], the enamel thickness of bovine incisors varies between 1 and 2 mm; even after removing 0.5–0.7 mm of the enamel layer from sample preparation, the hardness measurements can be assumed to be reliable because indentation depths were less than 10 % of the enamel thickness.

For each tooth sample tested at baseline and each treatment time point, three to five hardness tests were performed and the pooled average value calculated. Vickers hardness (HV) data is presented as pooled means ± 1 standard deviation (SD).

2.4. Study protocols

2.4.1. Enamel hardness after treatment without pH adjusted peroxide solutions

In this study, the effect of multiple whitening treatments with 6% w/v and 30% w/v HP bleaching solutions without adjustment of their pH (3.9 and 2.7, respectively) was investigated. The Vickers hardness of four bovine samples (two samples treated with 6% w/v HP and two samples treated with 30% w/v HP) was measured before HP treatment (baseline), and after up to seven consecutive treatments (lasting 3.5 h in total). Each treatment involved the immersion of the bovine samples into 20–25 ml of fresh HP solutions for 30 min at 37 °C. Such duration is typical for an in-office whitening treatment. Depending on the whitening procedure and tooth type, the treatment regimens can last for 2 to 6 weeks. After each treatment, the samples were rinsed with distilled water for 5 s and dried for 5 min at 37 °C. To establish stable hardness test conditions, the teeth samples were kept in a fume hood for around 15 min before they underwent hardness testing.

Following the last HP treatment and related hardness measurements, the tooth samples were treated with ACP to assess possible restoration of enamel hardness due to the remineralising effect of ACP. An aliquot of 80–100 μl of both ACP pre-cursors, i.e., calcium nitrate and sodium phosphate solutions, were spread evenly across the tooth surface using a micropipette.

During this process, both the solutions were mixed and a calcium phosphate precipitate was formed. Finally, this precipitate was spread evenly across the tooth surface. The precipitate was removed after one hour of application by rinsing teeth with distilled water, following which a final hardness measurement was conducted according to the parameters mentioned above.

2.4.2. Enamel hardness after treatment with pH adjusted peroxide solutions

This study was focussed on studying the change in hardness of the bovine samples over multiple whitening treatments when the pH of 6% w/v (pH 3.9) and 30% w/v (pH 2.7) HP solutions was adjusted to realistic oral physiological and neutral pH of 7.1. The treatment protocol as mentioned above, was used for this study, the only difference being the use of pH adjusted (pH 7.1) HP solutions instead of the non-adjusted bleaching solutions. The Vickers hardness measurements of six bovine samples (three samples treated with 6% w/v HP and three samples treated with 30% w/v HP) were taken before treatment (baseline) and after each treatment (up to 20 treatments) with each treatment being 30 min long (lasting in total for 10 h). After the 20th peroxide treatment, the teeth were exposed to ACP in the same way as previously described. Vickers hardness was measured with the same operating parameters as described in Section 2.3.

2.5. Wear and friction tests

To investigate the wear behaviour of HP treated teeth, friction tests were carried out using a CETR UMT2 tribometer. In the