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The durability of a hydroxyapatite paste used in decreasing the permeability of hypersensitive dentin

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ABSTRACT

Objectives: Various agents are currently available for treatment of hypersensitive dentine, however, their resistance to erosion intraorally by various erosive drinks is still questionable. The aim of this study is to test the efficacy of a hydroxyapatite paste to decrease dentine permeability and resist an erosion challenge.

Methods: Hydroxyapatite powder was mixed with 25% phosphoric acid to form a paste which was applied on dentine having patent dentinal tubules orifices (treated with EDTA, 2 min) and the resulting layer formed on top of dentine was irradiated by Nd:YAG laser. The treated dentin surfaces were exposed to erosion challenge (6% citric acid, 1 min). Dentine permeability was measured before/after the application of the hydroxyapatite paste before/after the erosion challenge before/after the application of Nd:YAG laser using a split chamber device. The top and the fractured dentine surfaces were examined with scanning electron microscope (SEM). Moreover, the chemical nature of the compounds formed on top of dentine surface was examined using the SEM equipped with energy- dispersive X-ray spectroscope (EDS) and FTIR/ATR (Attenuated Total Reflectance Fourier Transform Infrared) techniques. The Mann-Whitney test ($p < 0.05$) was used to compare the effects of using the hydroxyapatite paste on dentine permeability and calcium/phosphate ratio of the treated dentine surfaces.

Results: The application of the hydroxyapatite paste to dentine significantly decreased dentine permeability ($p < 0.05$). Hydroxyapatite paste was able to occlude patent dentinal tubule orifices with a layer of calcium-phosphate compounds and the application of Nd:YAG laser on the aforementioned layer improved its erosion resistance.

Conclusion: Hydroxyapatite paste applied with the technique adopted in the current study has a high potential to be a useful aid in the treatment of dentine hypersensitivity. However, cost and knowledge for using Nd:YAG laser are important factors should be taken into consideration before using the aforementioned technique.

1. Introduction

Dental problems and its related pain symptoms are mostly related to dental caries, however other dental lesions which are non-cariogenic in origin may lead to similar pain symptoms. These non-caries lesions may lead to loss of the enamel surfaces and exposure of the dentinal tubules to the oral cavity [1] causing pain sensation upon drinking hot or cold drinks. This phenomenon is referred to as "Dentin Hypersensitivity" [1–3]. Many theories were introduced to explain the phenomenon of the dentin hypersensitivity and its concomitant pain symptoms; one of the most accepted theories explaining the dentin hypersensitivity phenomenon is the "Hydrodynamic theory" [4,5]. This theory was strongly supported by many researches which demonstrated that [6,7] any partial reduction of the functional radii of the dentinal tubules will lead to significant reduction in the fluid flow with consequent reduction in dentin hypersensitivity pain symptoms [6–11].

Various aids and materials were employed for treating dentin hypersensitivity, however, the clinical success of these materials is still questionable [1,3]. Other attempts were used to melt some types of low fusing bioglasses on the dentinal surface using considerably low

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energy produced by CO_2 laser; these results showed that CO_2 laser if used with low output energy will not cause a significant increase of temperature on the pulpal side [12], however, no clinical trial was done to support the use of this technique inside patients' mouths for the treatment of dentin hypersensitivity. Moreover the dentin permeability was not measured after the application of the aforementioned technique.

A technique was recently introduced [11,13–15] during the past few years that included the temporary coverage of a bioactive glass paste by a layer of bonding agent for 24 h and then removal of this temporary bonding agent layer after 24 h. This technique showed plugging of the dentinal tubules by a calcium-phosphate rich layer [11] that was resistant to brushing abrasion challenge [8]. In the current study we used the same previously introduced technique but using the hydroxyapatite paste as a possible dentin desensitizing agent.

The aim of this experiment was to examine the capability of a hydroxyapatite paste to occlude dentinal tubules orifices by a layer of calcium phosphate compounds, moreover, the acid resistance of the formed layer was tested after the application of a low energy Nd:YAG laser.

The hypotheses adopted in this study was that the formed calcium phosphate layer will decrease dentin permeability and that application of Nd:YAG laser will render the formed layer more resistant to acidic-erosion.

2. Materials and methods

2.1. Dentin specimen preparation

40 extracted non-carious third molars were used following the guidelines approved by University Ethical Committee. The enamel on the buccal and the lingual surfaces of theses teeth were cut using a slow speed saw (Isomet, Buehler, Lake Bluff, IL, USA). The superficial dentin surfaces of teeth utilized in the current experiment were ground to obtain 80 dentin discs. All the dentin surfaces were ultrasonicated for 30 s, etched with 0.5% M EDTA (pH 4.7) for 2 min, and then rinsed with air/water for 30 s [8,16]. The specimens were randomly distributed 4 groups. Specimens of group I served as controls. The Nd:YAG laser was applied on the dentin surfaces of group II. The hydroxyapatite paste was applied on the dentin surfaces in group III, while Nd:YAG laser and hydroxyapatite paste were applied in group IV Table 1, Fig. 1(A–D).

2.2. Hydroxyapatite paste application

One-tenth of a gram of hydroxyapatite powder (011-14882 Apatite HAP, Wako Chemical, Osaka, Japan.) was mixed on a glass slab for 1 min by spatula with 0.2 ml of 25 wt% phosphoric acid that was prepared by the dilution of 85 wt% phosphoric acid (Wako Chemicals, Osaka, Japan) in distilled water to form a paste (pH 3.3) (Table 2). The acidic paste was immediately applied to the dentin surfaces of groups III and IV specimens by microbrush (Microbrush International, Grafton, WI, USA). A layer of bonding agent (Clearfil SE Bond, Kuraray Medical, Tokyo, Japan) was immediately applied over the hydroxyapatite-phosphoric-acid paste and then light-cured (Table 2). After storage in de-ionized water for 24 h in an incubator at 37 °C, the thin layer of the bonding agent was gently removed by means of an excavator, and then rinsed with water spray for 30 s.

Table 1
Summary for the Experimental groups.

Group IV	Group III	Group II	Group I	
+	+	–	–	Hydroxyapatite Application
+	–	+	–	Nd:YAG Laser Application

2.3. Nd:YAG Laser Application

The Laser system used in this study was Nd:YAG laser system (ULTRA Big Sky Quantel, USA), operating at wavelength in the NIR 1064 nm and delivers Q-switched pulses at of approximately 8 nanosecond durations with repetition rates of 1–20 Hz. and a maximum average power output of 600 mW. The laser was focused onto the dentin surfaces using a 7.5 cm focal length plano convex lens. The distance between the target area and laser beam focusing lens was 50 mm, and the diameter of the Nd:YAG laser beam at this distance was 1 mm. The laser pulse energy used was 30 mJ using a sweeping motion to ensure irradiation of all treated surfaces. The energy density/pulse was 5.1 J/cm^2.

2.4. Dentin permeability analysis

The dentin permeability of all groups ($n = 10$) was measured using an in vitro fluid-transport system before and after the acidic challenge. The system (Fig. 1A) was composed of a reservoir having phosphate-buffered saline (PBS, pH 7.0, Wako Pure Chemical, Osaka, Japan) solution which was placed between a tank of compressed nitrogen gas and a split-chamber device [17]. A pressure of 0.070 MPa [8,18] was used to force the PBS through polyethylene tubing to the split-chamber holding the dentin disk. A 25 μl micropipette connected to the polyethylene tubing was used to insert an air bubble into the tubing. The rate of air bubble movement was monitored using a millimeter ruler and the rate of the air bubble progression through the tubing was recorded every 2 min over a 6 min interval. The rate of dentin permeability was measured for each specimen at baseline and after application of the tested materials. Therefore each disk served as its own control [19]. The obtained values represented 100% permeability, which represented the baseline permeability for the discs. After application of the tested materials in groups III and IV, the dentin permeability was re-assessed and the percent of dentin permeability reduction was calculated.

2.5. Erosion challenge

The specimens in all groups which had their permeability measured were exposed to 6% citric acid pH 2.1 for 1 min with continuous stirring by a magnetic stirrer at room temperature [20]. The permeability of all specimens was re-measured after the erosion challenge and the percentage of permeability reduction was calculated.

2.6. SEM-EDS surface examination for dentinal orifices closure

Five dentin specimens were selected before/after the acidic challenge in the four groups. All specimens were gradually dehydrated in an ascending ethanol series (50–100%), gold coated and the specimens' surface chemical characterization and morphological features were examined by SEM-EDS (JCM-6000 NeoScope, JEOL, Tokyo, Japan.).

2.7. Cryofracture examination

Five dentin specimens from each group were selected before/after the acidic challenge. A slit was made along the pulpal side of each dentin disk using an Isomet saw under water cooling to facilitate cryofracture of the disk into two halves. The specimens