DENTAL MATERIALS 32 (2016) 807–816

Available online at www.sciencedirect.com

ScienceDirect

journal homepage: www.intl.elsevierhealth.com/journals/dema

Effects of coating materials on nanoindentation hardness of enamel and adjacent areas

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ARTICLE INFO

Article history:
Received 19 June 2015
Received in revised form
31 December 2015
Accepted 22 March 2016

Keywords:
Demineralization
Nanoindentation
Nanohardness
Fluoride
Bioactive

ABSTRACT

Objectives. Materials that can be applied as thin coatings and actively release fluoride or other bioavailable ions for reinforcing dental hard tissue deserve further investigation. In this study we assessed the potential of resin coating materials in protection of underlying and adjacent enamel against demineralization challenge using nanoindentation.

Methods. Enamel was coated using Giomer (PRG Barrier Coat, PBC), resin-modified glass-ionomer (Clinpro XT Varnish, CXT), two-step self-etch adhesive (Clearfil SE Protect, SEP) or no coating (control). After 5000 thermal cycles and one-week demineralization challenge, Martens hardness of enamel beneath the coating, uncoated area and intermediate areas was measured using a Berkovich tip under 2 mN load up to 200 μm depth. Integrated hardness and 10-μm surface zone hardness were compared among groups.

Results. Nanoindentation and scanning electron microscopy suggested that all materials effectively prevented demineralization in coated area. Uncoated areas presented different hardness trends; PBC showed a remarkable peak at the surface zone before reaching as low as the control, while CXT showed relatively high hardness values at all depths.

Significance. Ion-release from coating materials affects different layers of enamel. Coatings with fluoride-releasing glass fillers contributed to reinforcement of adjacent enamel. Surface prereacted glass filler-containing PBC superficially protected neighboring enamel against demineralization, while resin-modified glass-ionomer with calcium (CXT) improved in-depth protection. Cross-sectional hardness mapping of enamel on a wide range of locations revealed minute differences in its structure.

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http://dx.doi.org/10.1016/j.dental.2016.03.023

1. Introduction

Dental enamel is the hardest tissue of the human body but susceptible to dissolution of the mineral phase by acid. In the caries process, acid produced from bacterial metabolism diffuses into enamel and then dentin, and dissolves the mineral. White spot lesions are the early stage of caries development characterized by an enamel surface zone with subsurface demineralization [1,2]. Once this surface is broken due to continued effect of caries development, the lesions are cavitated and generally need to be restored. On the other hand, dental erosion is different from caries; it is defined as the chemical dissolution of dental enamel without bacterial involvement due to effects of acidic foods and drinks and considered as an increasingly common problem among different ages [3,4]. Subjects with gastric disorder may also suffer from sever loss of tooth enamel caused by acid reflux [4].

Emerging concepts in management of oral health involve preventive strategies as well as new approaches to protect the teeth against demineralization, and to provide non-surgical management options for early lesions [1,2,5]. Tooth surface coverage appears to be an immediate, simple and effective way to protect at-risk enamel against acid. Historically, pit and fissure sealants in newly erupted teeth have been well known to decrease caries prevalence [6–9]. Areas with frequently extensive plaque accumulations adjacent to bonded orthodontic brackets have also been suggested to benefit from extending proper coating materials [10].

In addition to physical protection effects, newly developed dental resins may act as a reservoir of bioactive ingredients such as fluoride (F) and calcium (Ca) ions or other elements [5,11]. F-releasing sealants have been suggested to provide additional caries inhibition effect, since fluoride inhibits demineralization and favors the remineralization processes [8,9,12,13]. However, the potential advantages of newly developed thin coating materials that release F or other ions are unknown and deserve further investigation [12].

Enamel consists of 95 wt.% minerals, mainly an impure calcium hydroxyapatite (Ap) crystals (100–1000 nm in length, 25–90 nm in thickness) make up larger formations as prisms (3–5 μm in diameter) [14]. The microstructure of enamel has been adapted to withstand mechanical and abrasive stresses. Loss of mineral content makes enamel vulnerable to deformation under mechanical load [1,2,15]. Moreover, enamel may be physically reinforced through remineralization and improvements in crystalline structure depending on the composition of the surrounding environment, such as degree of saturation with regard to the minerals [1,16,17]. In the dental literature, transverse microradiography (TMR) method has been widely employed to estimate the mineral content of dental hard tissue based on their radio opacity [16,18,19]. The cross-sectional microhardness measurement has been traditionally accepted as an alternative method to evaluate enamel in de/remineralization studies [19,20]. Nanoindentation (NI) technique has enabled investigations of local mechanical properties of materials under various loading regimes based on load displacement data of indentations on submicron scale [15,17,21–23]. Measurement of hardness by this technique has been suggested as advantageous over the conventional microhardness test methods for its high resolution of force and accurate indent positioning. This technique has been employed in assessing enamel erosion and demineralization/remineralization [17,23], and has shown a good sensitivity to hardness changes at different depths of enamel [1].

Hardness mapping of enamel beneath and adjacent to the bioactive materials can reveal information on potential benefits of the ions and compounds released from these materials. Thus, the aim of current laboratory study was to evaluate the effect of resin coating materials on nanoindentation hardness of coated enamel and adjacent area after demineralization challenge. The null hypotheses proposed were that covering enamel by resin material does not influence hardness of the enamel and adjacent area, and that there were no differences among the materials investigated.

2. Materials and methods

2.1. *Specimens preparation*

Twenty extracted, sound bovine incisors obtained from a local slaughter house (Yokohama, Japan) and checked to be free from any evidence of enamel cracks were collected and used according to a protocol approved by the Institutional Review Board of Tokyo Medical and Dental University for animal studies. The teeth were cleaned with deionized water to remove any surface debris and stored at −25 °C until needed. Enamel blocks 6 mm × 3 mm × 3 mm (length × width × depth) were cut from the bovine incisors using a low speed diamond saw (Isomet; Buehler, Lake Bluff, IL, USA) under running water, and embedded in epoxy resin (Epoxycure resin; Buehler). The outer surface was slightly polished with 800-grit silicon carbide (SiC) paper (Sankyo, Saitama, Japan) to remove the superficial layer and expose enamel.

2.2. *Coating materials*

According to the study design, the enamel blocks were divided into 4 groups ($n = 5$/group) corresponding to the materials used and control. In the control group, specimens received no treatment, three different resin-based materials were used to coat enamel in other groups; giomer coating PRG Barrier Coat (PBC; Shofu, Kyoto, Japan), resin-modified glass-ionomer Clinpro XT Varnish (CXT; 3M ESPE, St. Paul, MN, USA); and two-step self-etch adhesive resin Clearfil SE Protect (SEP; Kuraray Noritake Dental, Tokyo, Japan). Two areas, namely coated (C), and uncoated (UC), were assigned on the polished enamel surface of each block. Half surface of each block was carefully treated with the materials in accordance with the instructions supplied by the manufacturers as listed in Table 1 for all groups that served as the C area while the other half stayed intact as UC area. The specimens were then stored in water for 24 h at 37 °C.

2.3. *Thermo cycling procedure*

All specimens were placed in wire-mesh basket and subjected to 5000 thermal cycles between 5 °C and 55 °C (Yamato