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Experimental antimicrobial orthodontic adhesives using nanofillers and silver nanoparticles

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ABSTRACT

Objectives. Experimental composite adhesives (ECAs) containing silica nanofillers and silver nanoparticles were compared with two conventional adhesives (composite and resin-modified glass ionomer [RMGI]) to analyze surface characteristics, physical properties and antibacterial activities against cariogenic streptococci.

Methods. Surface roughness and surface free energy (SFE) characteristics were measured using confocal laser scanning microscopy and the sessile drop method. Shear bond strength and bond failure interface were analyzed to compare the physical properties. Antimicrobial activities were analyzed by a bacterial adhesion assay, a disk diffusion test, and an optical density measurement of bacterial suspension containing each adhesive.

Results. ECAs had rougher surfaces than conventional adhesives due to the addition of silver nanoparticles. ECAs had more similar SFE characteristics to composite than to RMGI. Bacterial adhesion to ECAs was less than to conventional adhesives, which was not influenced by saliva coating. Bacterial suspension containing ECAs showed slower bacterial growth than those containing conventional adhesives. There was no significant difference in shear bond strength and bond failure interface between ECAs and conventional adhesives.

Significance. This study suggests that ECAs can help prevent enamel demineralization around their surfaces without compromising physical properties.

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1. Introduction

Enamel demineralization is a commonly recognized complication of orthodontic treatment with a fixed appliance. Preventing these lesions is an important concern for orthodontists because the lesions are unaesthetic, unhealthy and potentially irreversible [1]. The placement of fixed orthodontic appliances creates a favorable environment for

the accumulation of microorganisms, which causes enamel demineralization or exacerbates the effects of any pre-existing caries [2].

A previous study reported that the remaining orthodontic adhesives on the enamel surface provide sites for the rapid attachment and growth of oral microorganisms due to their rough surfaces [3]. In addition, a recent study reported the presence of 10-μm wide gaps at the adhesive–enamel junction

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around the bracket base, within which bacterial accumulation was consistently detected [2]. These findings show that orthodontic bonding adhesives are one of the most potent risk factors for enamel demineralization during orthodontic treatment and clinical observations have indicated that the most common sites for demineralization are at the junctions of the bonding adhesives and the enamel [2,4].

One method to prevent enamel demineralization is to use orthodontic adhesives resistant to bacterial accumulation. For this purpose, various antimicrobial agents have been incorporated into orthodontic products and approved for intraoral use. Fluoride and chlorhexidine are the most common preventive additives for orthodontic use. Although initially strong, the released amounts of fluoride and chlorhexidine do not last for long periods [5–7]. In addition, when fluoride and chlorhexidine are used, antibacterial adhesives have a higher bond failure rate because incorporation of antibacterial agents into bonding adhesives affects their mechanical properties [5,8–10]. To overcome these disadvantages, experimental composite adhesives (ECA) in this study were made by incorporating silver nanoparticles and silica nanofillers.

Silver has a long history of use in medicine as an antimicrobial agent [11]. Resin composites containing silver ion-implanted fillers that release silver ions have been found to have antibacterial effects on oral streptococci [12]. Several studies have demonstrated that silver ions are selectively toxic for prokaryotic microorganisms with little effect on eukaryotic cells [13–15].

Recently, dental nanofilled resin composites were introduced in the market. The advantages of nanocomposite materials include excellent optical properties, easy handling characteristics, and superior polishability [16]. In addition, nanofillers can decrease surface roughness (SR) of orthodontic adhesives, which is one of the most significant factors for bacterial adhesion [17,18].

To be accepted clinically, the new adhesives must provide superior antimicrobial activity and display comparable physical properties when compared with conventional adhesives. The purpose of this study was to compare the surface characteristics, physical properties and antimicrobial activity of ECAs with those of the orthodontic adhesives currently available.

2. Materials and methods

2.1. Materials

The light-cure ECAs were formulated with 29.65 wt% of 1,6-bis(methacryloyloxy-2-ethoxycarbonylamino)-2,4,4-trimethylhexane (Esstech, Essington, PA, USA), 29.65 wt% of 2,2-bis[4-(2-hydroxy-3-methacryloyloxypropoxy)phenyl]-propane (Esstech), 19.75 wt% of triethylene glycol dimethacrylate (Esstech), 9.90 wt% of mono-2(methacryloyloxy)-ethyl phthalate (Sigma, St. Louis, MO, USA), 0.7 wt% of di-camphorquinone (Sigma), 0.35 wt% of 2-(dimethylamino)ethyl methacrylate (Sigma), and uniformly dispersed silica nanosized filler particles (10 wt%, particle diameter < 7 nm). To increase antimicrobial activities, various concentrations of silver nanoparticles (diameter < 5 nm) (Miji Tech Co, Ansan, Korea) were added to the composite adhesive: 0 ppm, 250 ppm, and 500 ppm, which

were named ECA1, ECA2 and ECA3, respectively. Then, ECAs were stirred for 12 h at 37 °C to get uniform mixtures.

Two commercial light-cure orthodontic adhesives were compared with ECAs, specifically, a non-fluoride-releasing composite (Transbond XT, 3 M/Unitek, Monrovia, CA, USA) (TB) and a RMGI (Fuji Ortho LC, GC Corporation, Tokyo, Japan). The average size of incorporating micro-sized fillers and silicate glasses is about 10.0 µm (approximately 1–20 µm) and 7.0 µm (approximately 2–25 µm), respectively (information provided by the manufacturers).

Each specimen was prepared using Teflon templates. Template plates were positioned on top of the glass slides. Each of the five bonding materials was placed into the holes until the material was flush with the top of the template. A second slide was placed on top, pushed down to ensure a flat dorsal surface, and then gently removed. All materials were handled according to the manufacturers' instructions and light cured for 40 s (20 s from the top and 20 s from the bottom).

2.2. Surface characteristics test

Disk-type (10.0 mm × 2.0 mm) specimens were used for surface characterization. Surface roughness was analyzed using confocal laser scanning microscopy (Axiovert 200M; Carl Zeiss, Thornwood, NY, USA). A multi-argon laser emits light at a wavelength of 633 nm, which yields the arithmetic mean SR from a mean plane within the sampling area (245 µm × 245 µm × 60 µm).

Surface free energy and its component parts, namely the non-polar (γ^LW) and polar acid/base component (γ^{AB}) which is divided into acid (γ^+) and base (γ^-) components, were measured by the sessile drop method. Three probe liquids of different polarity were used: 1-bromonaphthalene, formamide, and deionized water. A video camera equipped with an image analyzer (Phoenix 300, Surface electro optics, Seoul, Korea) visualized the shape of the drop and gave the contact angle. The contact angle measurements were performed at room temperature. The volume of drop is controlled by a machine interfaced with a computer and pictures of drop were taken as soon as drop placed on surfaces using a video camera interfaced with the computer. Right and left contact angles of each drop were automatically calculated from the computer and averaged to give one contact angle per drop. The solid SFE and its components were determined from contact angle measurements according to the van Oss model [19]. Each experiment was repeated five times for three specimens of each material.

2.3. Bracket bonding test

Eighty-five freshly extracted, healthy (without caries and restoration-free) human premolars were cleaned with a rotary brush and pumice, and stored in a 1% aqueous solution of chloramines-T (Junsei chemical, Tokyo, Japan) at 4 °C until further use. Written consents were obtained from the subject and the research protocol was approved by the Institutional Review Board of the University Hospital. The premolars were embedded individually in an acrylic mold with the labial surface parallel to the mold base so that their labial surface would be parallel to the force during the shear bond test. The

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