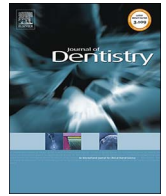




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Antibacterial and remineralizing orthodontic adhesive containing quaternary ammonium resin monomer and amorphous calcium phosphate nanoparticles

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ABSTRACT

Objectives: To evaluate the bonding performance, antibacterial activity, and remineralization effect on enamel of the orthodontic adhesive containing MAE-DB and NACP.

Methods: Eighty non-carious human premolars were divided into 3 groups: Transbond XT (TB), PEHB + 5% MAE-DB (PD), and PEHB + 40% NACP + 5% MAE-DB (PND). Premolars were bonded with orthodontic brackets, the first subgroup (n = 10) and the second subgroup (n = 10) were subjected to shear bond strength testing after immersed in water for 1 day and in demineralization solution for 28 days respectively and then tested surface roughness, while the third subgroup (n = 6) was used for microhardness evaluation after aged in demineralization solution for 28 days. For each adhesive, fifty disk samples were prepared for antibacterial study. Specimens measuring 12 mm × 2 mm × 2 mm were fabricated for ion release test.

Results: Bond strengths were in the order TB = PND > PND = PD for “1-day in water”, and in the order TB = PND > PD for “28-days in pH 4 solution”. No significant difference in the ARI scores for the three adhesive. Numerous bacteria adhered to TB surface, while PD and PND had minimal bacterial growth and activity. PND showed high levels of Ca and P ions release and enamel hardness. The surface roughness of enamel in PND was much lower than TB and PD and showed no significant difference with the sound, control enamel.

Conclusion: PND adhesive with 5% MAE-DB and 40% NACP exhibits antibacterial and remineralizing capabilities, and did not adversely affect bond strength compared to commercial adhesive.

Clinical significance: Novel adhesive containing quaternary ammonium monomer and nano-amorphous calcium phosphate represents a promising candidate in combating enamel white spot lesions and even dental caries.

1. Introduction

Formation of enamel white spot lesions around bonded orthodontic brackets is a serious and common complication during orthodontic treatment [1–3]. The prevalence of white spot lesions in patients with fixed appliance therapy has been reported to be 73–95% [2,4]. Previous studies have shown that the rate of enamel demineralization in

orthodontic patients is higher than those without orthodontic treatment [5–7]. Placement of fixed appliances increases plaque accumulation around the brackets and colonization of cariogenic bacteria [4,8,9]. Organic acids produced by those bacteria such as lactic, propionic and butyric acids result in surface and subsurface demineralization of the tooth enamel [10].

Because of the esthetic concerns associated with peri-bracket white

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Table 1
Composition (in wt%) of the experimental adhesives used in the present study.

Adhesives	PMGDM	EBPADMA	HEMA	Bis-GMA	BPO	CQ	NACP	MAE-DB
	44.5 (100%)	39.5	10	5	0.8	0.2		
PD	95						0	5
PND	55						40	5

PMGDM: pyromellitic glycerol dimethacrylate; EBPADMA: ethoxylated bisphenol A dimethacrylate; Bis-GMA: bisphenol A glycidyl dimethacrylate; HEMA: 2-hydroxyethyl methacrylate; BPO: benzoyl peroxide; CQ: camphorquinone; NACP: amorphous calcium phosphate nanoparticles; MAE-DB: 2-methacryloxyethyl dodecyl methyl ammonium bromide.

spot lesions, continuing efforts have been made to manage these lesions by preventing enamel demineralization or promoting enamel remineralization [11–13]. Topical fluoride or fluoride-releasing adhesives and cements have been used to prevent enamel demineralization around orthodontic brackets [12,13]. However, fluoride application to enamel prior to the placement of orthodontic brackets renders the enamel more resistant to phosphoric acid-etching. This reduces bonding efficacy and leads to premature bond failure [11]. Prescription of topical fluoride destined for home-use is usually inefficient due to low patient compliance [14]. An alternative preventive approach is based on calcium phosphate (CaP) remineralization [15–18]. Calcium (Ca) and phosphate (P) ions released from CaP biomaterials create an ionic reservoir in dental plaques. This reservoir of supersaturated Ca and P ions helps to prevent demineralization and facilitates enamel remineralization [19].

Resin-based materials containing nanoparticles of amorphous calcium phosphate (NACP) have been developed recently. The NACP-containing materials are capable of inducing enamel remineralization by releasing high levels of Ca and P ions [20–23]. Because of the small particle size and high surface area of these nanoparticles, resins that contain NACP have much higher Ca and P ion release than conventional CaP-filled resins [24,25]. Compared with a fluoride-releasing composite, NACP-containing resin composite possesses a 4-fold capacity for remineralization of incipient enamel lesions [26]. The NACP nanocomposite rapidly neutralizes a pH 4 demineralization solution and raises its pH to 5.5 and higher, thereby reducing the rate of enamel demineralization [27]. Incorporation of up to 40% NACP in a bonding agent has no adverse effect on bond strength [19–22]. However, the Ca and P ions release of the conventional CaP and NACP containing resin materials lasted for weeks to months, and then the release would decrease over time [25,28]. In our previous studies, novel dental resins containing NACP and PMGDM/EBPADMA were developed with the high capabilities of substantial recharge and sustained long-term release of Ca and P ions [19,23,29]. The rechargeable NACP orthodontic adhesive showed high bracket shear bond strength and Ca and P ion release capability. However, whether the new NACP orthodontic adhesive can inhibit the enamel demineralization as expectation is still unknown and need to be studied.

Polymerizable quaternary ammonium salt (QAS) possesses remarkable antimicrobial activities against a wide range of bacteria, fungi and viruses, and is a broad spectrum cationic biocide [30]. Cationic QAS disrupts the integrity of bacteria by attracting and puncturing negatively-charged bacterial cell membranes [30,31]. 2-methacryloxyethyl dodecyl methyl ammonium bromide (MAE-DB) is an example of a polymerizable QAS. The MAE-DB molecule contains two polymerizable methacrylate groups that enables it to co-polymerize with additional MAE-DB monomers and other resin monomers to produce a resin matrix with stable and long-lasting bactericidal effect [30,32]. Incorporation of this QAS resin monomer into a resin comonomer blend does not significantly compromise the biocompatibility, mechanical and bonding properties of the polymerized resin matrix [30,32,33].

Because bacteria-derived acids generate an acidic environment that hampers enamel remineralization, MAE-DB was used in conjunction with NACP in the present study to develop an orthodontic adhesive for

exerting antibacterial activity and promote remineralization simultaneously, without compromising the quality and longevity of the bonded interface. Accordingly, the objectives of the study were to test the null hypotheses that: 1) there is no difference in resin-enamel bond strength between the experimental MAE-DB and NACP-containing orthodontic adhesive and a commercial orthodontic adhesive that does not contain these components, and 2) the experimental MAE-DB and NACP-containing orthodontic adhesive does not possess significantly better bactericidal properties against *Streptococcus mutans* biofilms, or significantly better enamel remineralization potential compared with a commercial orthodontic adhesive that does not contain these components.

2. Materials and methods

2.1. Synthesis of NACP

A spray-drying technique was used to fabricate NACP, as previously described [26,28]. Calcium carbonate (CaCO₃) and dicalcium phosphate anhydrous (CaHPO₄) were dissolved in an acetic acid solution to obtain final Ca and PO₄ ionic concentrations of 8 and 5.333 mmol/L, respectively. Formation of NACP was initiated by atomizing a suspension of CaP micro-droplets followed by drying in a heated chamber to generate solid particles. The resulted NACP has a mean particle size of 116 nm, as reported previously [34].

2.2. Orthodontic adhesives

Two experimental orthodontic adhesives were formulated. The first consists of 44.5 wt percent (wt%) pyromellitic glycerol dimethacrylate (PMGDM) (International Laboratory USA, South San Francisco, CA, USA) and 39.5 wt% ethoxylated bisphenol A dimethacrylate (EBPADMA; MilliporeSigma, St. Louis, MO, USA; Table 1). Being an acidic adhesive monomer [35,36], PMGDM possesses recharging capability by chelating calcium ions from a recharging solution [23,29]. Ten wt% 2-hydroxyethyl methacrylate (HEMA; MilliporeSigma) and 5 wt% bisphenol A glycidyl dimethacrylate (BisGMA) (MilliporeSigma) were added to the PMGDM-EBPADMA mixture. These weight fractions were selected because previous studies showed that addition of 10% HEMA and 5% BisGMA into the PMGDM-EBPADMA resin comonomer blend increased its bond strength to dentin [29]. Benzoyl peroxide (BPO, 0.8 wt%; Alfa Aesar, Heysham, Lancashire, United Kingdom) was added to enable chemical-cure, and camphorquinone (CQ, 0.2 wt%; MilliporeSigma) was added as photoinitiator to provide the capacity for the resin blend to be light-cured. The resin was designated as PEHB. The polymerizable QAS, MAE-DB (Fig. 1), was added to the PEHB at a weight fraction of 5% (Table 1) according to the previous study [32].

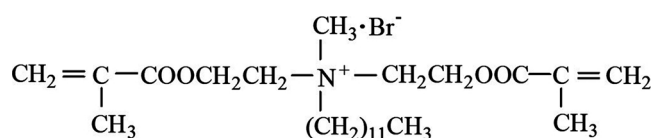


Fig. 1. Structure of 2-methacryloxyethyl dodecyl methyl ammonium bromide (MAE-DB).

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