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Bonding ability of 4-META self-etching primer used with 4-META/MMA-TBB resin to enamel and dentine: Primary *vs* permanent teeth

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ABSTRACT

Objectives: The present study compared the efficacies of the self-etching Teeth Primer (TP: 4-META), and the etchants Red Activator (RA; 65% phosphoric acid) and Green Activator (GA; 10% citric acid with 3% ferric chloride), for bonding to enamel and dentine of human primary and permanent teeth, when used with 4-META/MMA-TBB resin (Bondfill SB).

Methods: Forty-eight non-carious primary canines and third molars were used. Eight groups were prepared: Group 1 (primary enamel with RA), Group 2 (permanent enamel with RA), Group 3 (primary enamel with TP), Group 4 (permanent enamel with TP), Group 5 (primary dentine with GA), Group 6 (permanent dentine with GA), Group 7 (primary dentine with TP) and Group 8 (permanent dentine with TP). Micro-tensile bond strengths (MTBS) were measured and analyzed statistically using ANOVAs and Tukey HSD tests at $\alpha = 0.05$. Efficacy of etching/priming and the morphology of bonded interfaces were observed with SEM.

Results: Etching/priming efficacy of TP on enamel was low. The MTBS of Group 2 was significantly higher than the other groups (Groups 1, 3 and 4). For dentine, significant differences in MTBS were observed, in the order of Groups $6 > 8 > 7 = 5$ ($p < 0.05$). The MTBSs of permanent dentine were significantly higher than primary dentine. For primary teeth, there was no significant difference in the MTBSs between enamel and dentine, irrespective of primer or etchant ($p > 0.05$).

Conclusion: TP primer/Bondfill SB may be used as an alternative to other adhesive/resin composite systems for bonding to enamel and dentine of primary teeth.

1. Introduction

Attrition and wear of enamel and dentine, gingival recession and formation of wedge-shaped defects occur throughout life due to clenching, grinding, mastication, tooth brushing and other factors. For restoration of these defects, it is beneficial for the wear resistance of dental materials to approximate those of tooth substrates, particularly for areas that are in constant occlusal contact. An adhesive resin chemically initiated with tri-*n*-butylborane (TBB) has been used for bonding between tooth structure and restorative materials. One of the problems associated with the TBB-initiated resin system is its insufficient wear resistance, due to the absence of inorganic fillers.[1,2]

A modified TBB resin containing pre-polymerized organic filler particles (Bondfill SB, Sun Medical Co., Ltd., Moriyama, Japan) was recently developed for filling, luting, or sealing

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procedures employed in preventive and restorative dentistry.[3] This modified resin demonstrated better wear characteristics when compared with conventional unfilled TBB-initiated resins.[4] Bondfill SB is a resin composite that utilizes the 4-methacryloyloxyethyl trimellitate anhydride/methyl methacrylate-TBB (4-META/MMA-TBB) system for adhesion to tooth substrates. For practitioners who are using the etch-and-rinse bonding technique, the manufacturer has originally developed an etchant (65% phosphoric acid; Red Activator) for bonding to enamel, and a conditioner consisting of 10% citric acid with 3% ferric chloride (Green Activator) for bonding to dentine. Because enamel and dentine in primary teeth (especially for dentine) are more susceptible to primers and etchants than permanent teeth,[5–9] over-etching may occur when the same etching time employed for permanent teeth is adopted for etching primary teeth. To alleviate this problem, a 4-META containing self-etching primer (Teeth Primer, Sun Medical Co., Ltd.) was developed and included in Bondfill SB for practitioners who prefer the self-etching approach, and for bonding to tooth substrates in primary teeth. As the pH of Teeth Primer is 3.0, the bonding technique has been termed the ultra-mild self-etching approach.[10,11] Although the use of mild self-etching primers/adhesives is not ideal for bonding to permanent tooth enamel,[12,13] limited information is available for their bonding efficacy to primary tooth enamel. Thus, the objective of the present study was to compare the use of the self-etching Teeth Primer (TP) in Bondfill SB, with the 65% phosphoric acid-containing Red Activator (RA) for bonding to primary and permanent human enamel, and with the Green Activator (GA) for bonding to primary and permanent human dentine. The null hypotheses tested were: (1) there is no difference in the micro-tensile bond strength of Bondfill SB to enamel or dentine treated with TP, RD or GA; and (2) there is no difference in the micro-tensile bond strength of TP-primed specimens between primary teeth and permanent teeth for either enamel or dentine.

2. Materials and methods

Forty-eight non-carious primary canines that were extracted to expedite eruption of the succedaneous teeth or for orthodontic reasons were used as primary tooth specimens. Forty-eight extracted non-carious upper third molars were used as permanent tooth specimens. Informed consent for tooth collection was obtained from parents and subjects according to the regulations of ethics committees of Nagasaki University. The extracted teeth were frozen in physiologic saline within 10 min after extraction. Table 1 shows the components and protocol of the materials used in the present study.

The primary or permanent teeth were each divided into 4 groups according to whether enamel or dentine was bonded, and the primer or activator employed for substrate treatment. Group 1 (primary tooth enamel etched with Red Activator: RA); Group 2 (permanent tooth enamel etched with RA); Group 3 (primary tooth enamel primed with Teeth Primer: TP); Group 4 (permanent tooth enamel primed with TP); Group 5 (primary tooth dentine etched with Green Activator: GA); Group 6 (permanent tooth dentine etched with GA); Group 7 (primary tooth dentine primed with TP); and Group 8 (permanent tooth dentine primed with TP).

2.1. *Scanning electron microscopy (SEM)*

Flat enamel and dentine surfaces were prepared using silicon carbide papers under copious water-cooling on the labial surfaces of primary canines and the occlusal surfaces of third molars ($N = 12$ for each of 8 groups). Final polishing was achieved using 1200-grit silicon carbide paper. In Groups 1 and 2, 65% phosphoric acid (RA) was applied on enamel for 30 s, rinsed with water for 15 s and dried with an air-water syringe. In Groups 5 and 6, 10% citric acid with 3% ferric chloride (GA) was applied on dentine for 10 s, rinsed for 15 s and dried with an air–water syringe. For Groups 3, 4, 7 and 8, Teeth Primer (TP) was applied on enamel or dentine for 20 s and air-dried for 20 s. For Groups 3, 4, 7 and 8, the specimens were immersed in acetone for 10 s to eliminate resinous material from the primed tooth substrates. All specimens were dried in a desiccator and sputter-coated with platinum (Superfine Coater ESC-101; Elionix Co., Tokyo, Japan) prior to SEM observation (JSM-5610LV, JOEL, Tokyo, Japan).

2.2. *Micro-tensile bond strength (MTBS) evaluation*

Flat enamel or dentine surfaces were prepared in the manner previously described using 180-grit silicon carbide paper for final polishing. Enamel or dentine surfaces were etched or primed in the same manner as for the SEM observation. After etching or priming, a mixture of the polymer and monomer-catalyst of Bondfill SB was applied and built up using the brush-on technique. For primary teeth, Bondfill SB was applied

Table 1 – Components of the materials and protocols utilised in the preset study.

Material (Batch no.)	Components	Protocol
Teeth Primer: TP (0056)	4-META, water, acetone, reducing agent (pH 3.0)	Apply primer to enamel and dentine for 20 sec and air dry
Red Activator: RA (TE1)	65% phosphoric acid (pH 0.3)	Apply activator to enamel for 30 sec, wash with water and air dry
Green Activator: GA (SF2)	10% citric acid with 3% ferric chloride (pH 0.6)	Apply activator to dentine for 10 sec, wash with water and air dry
Bondfill SB		
Liquid (10F0629)	4-META, MMA, poly-functional methacrylate	Apply mixture of the polymer powder and monomer catalyst, using the brush-on technique to the bonded tooth surface
Powder (10K03173)	PolyMMA, TMPT pre-polymerized filler, pigment	
Catalyst V (TT1)	TBB, TBB-O, hydrocarbon	

Abbreviations: 4-META: 4-methacryloxyethyl trimellitate anhydride; MMA: methyl methacrylate; TMPT: trimethylopropane trimethacrylate; TBB: tri-n-butylborane; TBB-O: partially oxidized tri-n-butylborane.