

# A study on the compatibility between one-bottle dentin adhesives and composite resins using micro-shear bond strength

Minju Song<sup>1</sup>, Yooseok Shin<sup>2</sup>, Jeong-Won Park<sup>1</sup>,  
Byoung-Duck Roh<sup>2\*</sup>

<sup>1</sup>Department of Conservative Dentistry  
Gangnam Severance Dental Hospital,  
<sup>2</sup>Department of Conservative Dentistry  
and Oral Science Research Center,  
Yonsei University College of Dentistry,  
Seoul, Korea

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<sup>1</sup>Song M; Park JW, Department of  
Conservative Dentistry Gangnam  
Severance Dental Hospital, Yonsei  
University College of Dentistry,  
Seoul, Korea

<sup>2</sup>Shin Y; Roh BD, Department of  
Conservative Dentistry and Oral  
Science Research Center, Yonsei  
University College of Dentistry,  
Seoul, Korea

## \*Correspondence to

Byoung-Duck Roh, DDS, MSD, PhD.  
Professor, Department of  
Conservative Dentistry and Oral  
Science Research Center, Yonsei  
University College of Dentistry,  
50 Yonsei-ro, Seodaemun-gu, Seoul,  
Korea 120-752  
TEL, +82-2-2228-3146; FAX, +82-  
2-313-7575; E-mail, OPERATYS16@  
yuhs.ac

**Objectives:** This study was performed to determine whether the combined use of one-bottle self-etch adhesives and composite resins from same manufacturers have better bond strengths than combinations of adhesive and resins from different manufacturers. **Materials and Methods:** 25 experimental micro-shear bond test groups were made from combinations of five dentin adhesives and five composite resins with extracted human molars stored in saline for 24 hr. Testing was performed using the wire-loop method and a universal testing machine. Bond strength data was statistically analyzed using two way analysis of variance (ANOVA) and Tukey's *post hoc* test. **Results:** Two way ANOVA revealed significant differences for the factors of dentin adhesives and composite resins, and significant interaction effect ( $p < 0.001$ ). All combinations with Xeno V (Dentsply De Trey) and Clearfil S<sup>3</sup> Bond (Kuraray Dental) adhesives showed no significant differences in micro-shear bond strength, but other adhesives showed significant differences depending on the composite resin ( $p < 0.05$ ). Contrary to the other adhesives, Xeno V and BondForce (Tokuyama Dental) had higher bond strengths with the same manufacturer's composite resin than other manufacturer's composite resin. **Conclusions:** Not all combinations of adhesive and composite resin by same manufacturers failed to show significantly higher bond strengths than mixed manufacturer combinations. (*Restor Dent Endod* 2015;40(1):30-36)

**Key words:** Compatibility; Micro-shear bone strength; One bottle self-etch adhesive; One-step self-etch adhesive; Resin composite

## Introduction

Dentin adhesives for dental restorations have been developed for generations with an emphasis on adhesion to dentin.<sup>1,2</sup> In the late 90s, the one-step self-etch adhesive (two bottles) was developed by the functional combination between the self-etching primer and bonding resin, which was suitable for conditioning enamel and dentin as well.<sup>3,4</sup> However, mixing two solutions is a cumbersome process. Today a one-step self-etch adhesive (one bottle) is available that also has the merits of simplifying the adhesion procedure, reducing chair time, reducing differences between operators, and relieving postoperative sensitivity.<sup>5</sup>

The first one-bottle self-etch adhesive, iBond, was introduced in late 2000. It consists of hydrophilic and hydrophobic substances, water, and solvent, and differs from previous dentin adhesives in terms of its chemical composition.<sup>2</sup> iBOND has its own functional monomer, which characterizes chemical bond to dentin and determines the bond strength of the adhesive.<sup>6-8</sup> Furthermore, it is similar to the one-step self-etch

adhesive (two bottles) in bond strength and border seal characteristics.<sup>9</sup>

Bond strengths of dentin adhesives are also affected by mechanical properties of composite resins. Leirskar *et al.* suggested that the bond strengths of dentin adhesives might depend on the choice of restorative material based on an evaluation of shear bond strengths to dentin of five different dentin adhesives in combination with two resin-based composites.<sup>10</sup> They recommended using composite resin and dentin adhesive from the same manufacturer, but little information is available in the literature regarding the adhesive performance of these mixed manufacturer systems. Roh also analyzed the bond strengths of combinations of total etch adhesive systems and two-step self-etching systems made by respective manufacturers, and suggested combinations based on same manufacturers did not unduly increase bond strengths, but that the characteristics of

dentin adhesive and composite resin had greatest effect on bond strength.<sup>11</sup>

This study was performed to determine whether the combined use of one-bottle self-etch adhesives and composite resins from same manufacturers has better bond strength than combinations of adhesive and resins from different manufacturers. We hypothesized that bond strengths would be better for combinations from same manufacturers.

### Materials and Methods

Five dentin adhesives and five composite resins from 5 manufacturers were used (Table 1). Recently produced dentin adhesives were selected, and the composite resins used were recommended by manufacturers or released around the same time.

**Table 1.** Composition of dentin adhesive systems and composite resins used in this study

Products	% of Filler (vol%)	Composition	Manufacturer
BondForce (BF)		Methacryloyloxyalkyl acid phosphate, 2-Hydroxyethyl methacrylate, Bis-GMA, Triethylene glycol dimethacrylate, Camphoroquinone, Alcohol, C2-4 alkyl, Water	Tokuyama Dental, Tokyo, Japan
Estelite Quick (EQ)	71	Silica-zirconia filler, Composite filler, Bis-GMA, Triethylene glycol dimethacrylate, Camphoroquinone	
iBond (IB)		4-META, UDMA, Glutaraldehyde, Camphoroquinone, Aceton, Water	Heraeus Kulzer, Hanau, Germany
Charisma (CH)	64	Bis-GMA, Barium aluminium fluoride glass, highly dispersive siliciumdioxide	
Xeno V (XV)		Acrylamide alkylsulfonic acid, "inverse" functionalized phosphoric acid ester, Acrylic acid, Bifunctional acrylic resin with amide functions, Initiator/Stabilizer, Tertiary butanol, Water	Dentsply De Trey, Radolfzell, Germany
Ceram X Duo (CD)	57	Methacrylate modified polysiloxane, Demethacrylate resin, Fluorescence pigment, UV stabilizer, Stabilizer, Camphoroquinone, Ethyl-4(dimethylamino) benzoate, Barium-aluminium-borosilicate glass, Methacrylate, functionalised silicone dioxide nano filler, Pigments	
Clearfil S <sup>3</sup> Bond (CS)		10-MDP, Bis-GMA, HEMA, Hydrophobic dimethacrylate silanated colloidal silica, Camphoroquinone, Ethanol, Water	Kuraray Dental, Tokyo, Japan
Clearfil Majesty (CM)	-	Silanated barium glass filler, Pre-polymerized organic filler, Bis-GMA, Hydrophobic aromatic dimethacrylate, di-Camphoroquinone	
G-Bond (GB)		4-MET, Phosphoric ester monomer, UDMA, Silica filler Camphoroquinone, Acetone, Water	GC dental product, Tokyo, Japan
Gradia Direct (posterior)(GD)	65	matrix: UDMA, Dimethacrylate co-monomers filler: Silica, Fluoro-Alumino-Silicate Glass, Prepolymerised filler, Pigments, Catalysts	

Bis-GMA, bisphenol-A-diglycidyl methacrylate; 4-META, 4-methacryloyloxyethyl trimellitate anhydride; UDMA, urethanedimethacrylate; UV, ultra-violet; 10-MDP, 10-methacryloxydecyl dihydrogen phosphate; HEMA, 2-hydroxyethyl methacrylate; 4-MET, 4-methacryloxyethyl trimellitic acid.

### Specimen preparation

Human molars, put in saline solution within 1 month after extraction, were used to make tooth slices. Using a low-speed diamond wheel saw (Struers Minitom, DK-2610 Rodøvre, Ballerup, Denmark), molars were cut into 2 mm-thick slices parallel with the occlusal surface under water irrigation. The dentin surface was ground with 220 grit sandpaper (Figure 1a).

Two stacked post-it notes (3M, Seoul, Korea) were punched and then stuck onto each tooth slice to expose the dentin surface through the hole (Figure 1b). In accordance with the manufacturer's manual, dentin adhesive was applied to the slice and polymerized using a light curing unit (Elipar FreeLight, 3M ESPE, St. Paul, MN, USA). After removing one of two stacked post-it notes, a 0.7 mm-diameter and 0.4 mm-high tube (Tygon, Norton Performance Plastic Co., Akron, OH, USA) was situated at the hole (Figures 1c and 1d) and filled with composite resin, which was then polymerized. Each dentin adhesive was used in combination with the 5 composite resins to produce 25 combinations and 15 specimens were prepared per combination (375 specimens in total). After the tube and post-it note had been removed, excessive adhesive was removed using a blade (Figure 1e). Specimens were then placed in saline solution at room temperature for 24 hours.

### Micro-shear bond strength tests

Testing was conducted as follows. A specimen that had been kept in saline solution for 24 hours was hung on a universal testing machine (EZ Test, Shimadzu, Kyoto,

Japan) and its lower part was looped with wire (the wire-loop method). Force was applied at a cross-head speed of 0.5 mm/min until the bond was broken (Figure 1f).<sup>12</sup> Bond strength was measured using the WinAGSLite program (Shimadzu).

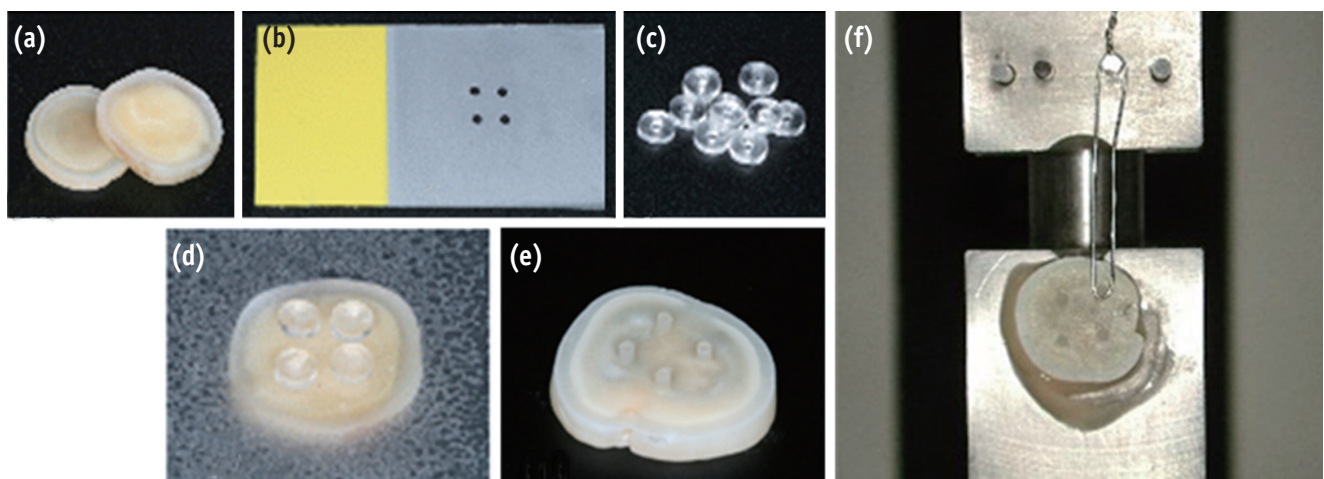
### Statistical Analysis

Data were analyzed using two-way analysis of variance (ANOVA) with Tukey's *post hoc* test. The analysis was conducted using SAS version 9.1 (SAS Institute Inc., Cary, NC, USA).

### Results

ANOVA results are presented in Table 2. Evaluation with two-way ANOVA revealed significant differences for the following factors; dentin adhesives and composite resins, and significant interaction effect ( $p < 0.001$ ). The Tukey test was then applied for dentin adhesives and it showed that Xeno V (XV,  $16.0 \pm 3.5$  MPa), G-Bond (GB,  $14.7 \pm 3.4$  MPa), Clearfil S<sup>3</sup> Bond (CS,  $14.6 \pm 3.5$  MPa) had higher bond strengths than iBond (IB,  $12.0 \pm 4.4$  MPa) and BondForce (BF,  $11.7 \pm 4.2$  MPa,  $p < 0.05$ ). For composite resins, Ceram X Duo (CD,  $16.2 \pm 4.2$  MPa) showed the highest bond strength, and Estelite Quick (EQ,  $14.6 \pm 4.0$  MPa) had the next highest ( $p < 0.05$ ). The bond strengths of each dentin adhesive and composite resin combination are listed in Table 3.

Figure 2 shows the results of the Tukey test for interactions between dentin adhesives and composite resins. All combinations of XV and CS adhesives showed no



**Figure 1.** Preparation of specimens for Micro-Shear Bond Strength Tests. (a) Tooth slice; (b) Post-it note placement; (c) Tube; (d) Composite resin filled in the tube; (e) Specimen for micro-shear bond test; (f) Wire-Loop Method (EZ Test, Shimadzu, Kyoto, Japan).

Table 2. Two-way ANOVA results according to adhesive systems and composite resins

Source	Sum of squares	df	Mean square	F	p value
Corrected model	2569.70	24	107.13	9.27	< 0.001
Adhesives	1048.88	4	262.22	22.72	< 0.001
Resin	751.20	4	187.80	16.27	< 0.001
Adhesive*Resin	768.62	16	48.04	4.16	< 0.001
Error	4040.14	350	11.54		
Corrected total	6608.84	374			

Table 3. Micro-shear bond strength of various combinations of self-etch adhesives and composite resins from same and different manufacturers

	Composite resins					Mean
	Estelite Quick (EQ)	Charisma (CH)	Ceram X Duo (CD)	Clearfil Majesty (CM)	Gradia Direct (GD)	
BondForce (BF)	<u>13.5 ± 5.2</u>	8.5 ± 1.9	12.0 ± 5.0	11.1 ± 1.9	13.4 ± 4.1	11.7 ± 4.2 <sup>B</sup>
iBond (IB)	15.2 ± 4.5	<u>10.5 ± 3.7</u>	15.6 ± 3.2	9.9 ± 3.1	8.9 ± 2.0	12.0 ± 4.4 <sup>B</sup>
Xeno V (XV)	14.4 ± 4.0	16.4 ± 4.2	<u>18.5 ± 2.5</u>	16.1 ± 2.4	14.5 ± 2.7	16.0 ± 3.5 <sup>A</sup>
Clearfil S3 Bond (CS)	16.3 ± 2.5	12.7 ± 4.3	16.8 ± 4.0	<u>14.6 ± 2.0</u>	12.7 ± 2.4	14.6 ± 3.5 <sup>A</sup>
G-Bond (GB)	13.5 ± 2.6	14.6 ± 3.6	18.3 ± 2.8	12.8 ± 3.4	<u>14.5 ± 2.2</u>	14.7 ± 3.4 <sup>A</sup>
Mean	14.6 ± 4.0 <sup>b</sup>	12.5 ± 4.5 <sup>c</sup>	16.2 ± 4.2 <sup>a</sup>	12.9 ± 3.4 <sup>c</sup>	12.8 ± 3.4 <sup>c</sup>	

Means within each group with same superscript letter are not significantly different by Tukey test (small letter, row; capital letter, column); Underlines indicate same manufacturer.

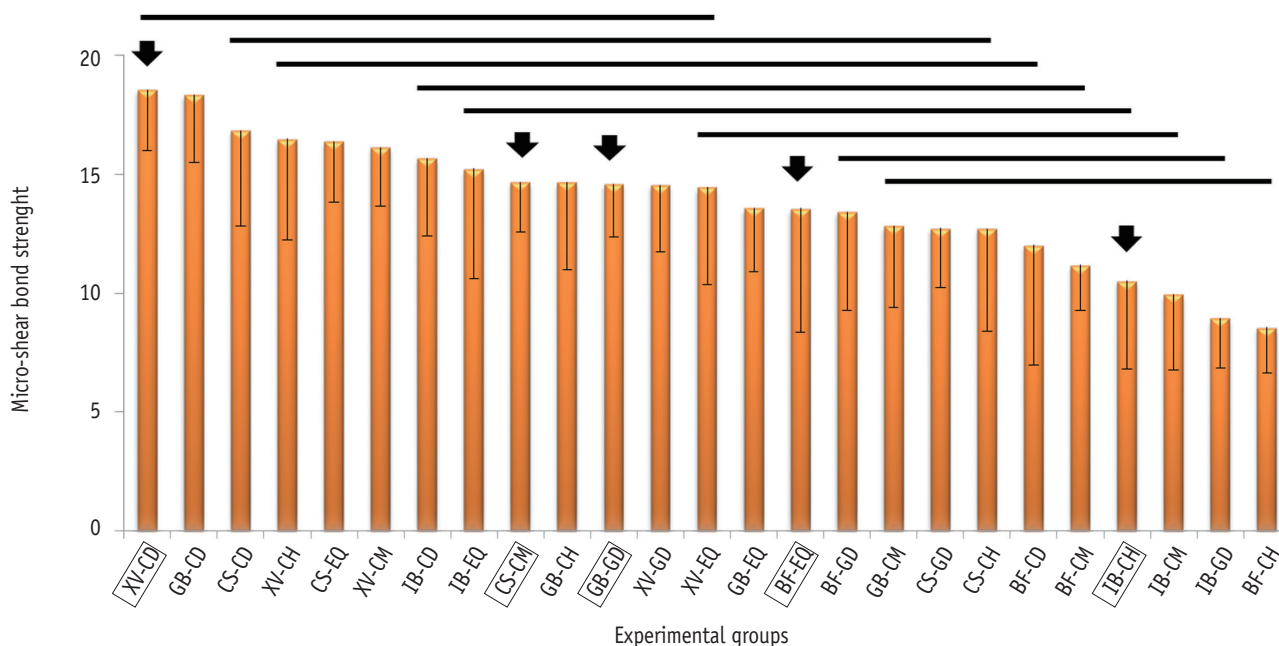


Figure 2. Micro-shear bond strength plots for the experimental groups and Tukey test results for the interaction between dentin adhesives and composite resins. Horizontal bars indicate that there is no significant difference (Black arrow). The material groups designated in the box were the combination of the adhesive and the composite resin supplied by the same manufacturer. XV, Xeno V; CD, Ceram X Duo; GB, G-Bond; CS, Clearfil S3 Bond; CH, Charisma; EQ, Estelite Quick; CM, Clearfil Majesty; IB, iBond; GD, Gradia Direct; BF, BondForce.

significant difference in micro-shear bond strength ( $p < 0.05$ ), while other adhesives showed significant differences depending on the composite resin. Not all combinations of dentin adhesives and composite resins from same manufacturers failed to show significantly higher bond strengths than mixed manufacturer combinations (Figure 2).

XV and BF tended to show higher bond strengths with same manufacturer's composite resins than with other companies' composite resins. On the other hand, the IB-CH combination showed lower bond strength than other combinations, but the IB-EQ and IB-CD combinations showed higher bond strength ( $p < 0.05$ ). Combinations with CD showed the highest bond strength in the all dentin adhesives group, except BF-CD (Figure 2). The EQ composite group did not show significant differences in bond strength when used in combination with any the five dentin adhesives ( $p > 0.05$ ). GD showed similar bond strengths when used in combination with four dentin adhesives, except IB.

## Discussion

Several studies have analyzed the bond strengths of self-etch adhesives/total-etch adhesives and composite resins combinations made by respective manufacturers.<sup>5,11,13</sup> In a previous study, we found that combinations of products of same manufacturers did not exert a crucial influence on bond strength, and that bond strength depended on the characteristics of dentin adhesives and composite resins.<sup>11</sup> Micro-shear bond strength depends on the type of resin-based composites used when a total-etch adhesive is used, whereas the type of adhesive plays an important role in bond strength when a self-etch adhesive is used.<sup>11</sup>

Unlike etch and rinse system, self-etch adhesive systems demineralize the dentin superficially, so that hydroxyapatite rests between collagen fibrils. Hydroxyapatite interacts with dentin adhesive chemically, and thus, improve bond strength.<sup>14</sup> The chemical interaction is dependent on the functional monomers present, and the adhesive strength depends on the hydrolytic stability of  $\text{Ca}^{2+}$ -monomers.<sup>15</sup> Self-etch adhesives contain carboxyl- or phosphate-based monomers, and are characterized by such functional monomers. Yoshida *et al.* analyzed energy changes caused by exposing demineralized hydroxyapatite to functional monomers by using X-ray photoelectron spectroscopy (XPS).<sup>6</sup> Of the functional monomers examined, 10-methacryloxydecyl dihydrogen phosphate (10-MDP) and 4-methacryloxyethyl trimellitic acid (4-MET) showed the highest bond strengths. In particular, in addition to high bond strength, 10-MDP reached a sufficient level of adhesion faster. Thus, it might be suitable for the clinical environment because of a shorter chair time.

Actually in the case of the two-step self-etch adhesive system, bond strength is higher in 10-MDP-bearing Clearfil

SE Bond (Kuraray, Tokyo, Japan) than in 4-MET-bearing Unifil Bond (GC America Inc., Alsip, IL USA).<sup>16</sup> In this study, however, 10-MDP-bearing CS and 4-MET-bearing GB did not show significant differences in shear bond strength. Besides, 4-META (a precursor of 4-MET)-bearing IB showed a significantly lower bond strength in CH, CM, and GD resin composite groups, which suggests that the chemical interaction caused by functional monomers is not enough to explain the bond strength of dentin adhesives.

The characteristics of self-etch adhesive systems might depend upon their acidity. Most one-step self-etch adhesives (one bottle) on the market have low acidity ( $> \text{pH } 2$ ) and form the  $1 \mu\text{m}$ -thick hybrid layer.<sup>17</sup> However, even though a resin can easily permeate into demineralized collagen, nanoleakage is also observed in the hybrid layer. The higher the acidity of dentin adhesive becomes, the less likely resin permeates.<sup>18,19</sup> Also when its pH is below pH 2, chemical adhesion is less likely to be resistant to hydrolytic degradation due to insufficient HA.<sup>19,20</sup> Regarding the dentin adhesives used in this study, only IB showed moderate acidity (pH 1.6), which could have contributed to lower bond strengths.

For combinations of self-etch adhesives and composite resins from same manufacturers, XV-CD (Dentsply, Radolfzell, Germany) and BF-EQ (Tokuyama Dental, Tokyo, Japan) tended to show the highest bond strengths, but these were without statistical significance. Other same manufacturer combinations did not show the compatibility between same manufacturer's products. Combinations with XV dentin adhesive or CD composite resin, for example, showed higher bond strengths than other combinations except BF-CD. The hypothesis of this study was rejected, as some of same manufacturer combinations failed to show significantly higher bond strengths.

In the present study, XV showed the highest bond strength ( $16.0 \pm 3.5 \text{ MPa}$ ) among dentin adhesives. XV contains acrylic amide resin and inverse functionalized phosphoric acid ester as functional monomers. Monomers reserve a polymerizable functional group even after hydrolysis, and thus, dentin adhesives tend to continue the polymerization mechanism during storage, which increases bond strength.<sup>21</sup> All composite resins combined with XV showed high bond strengths.

CD is a nanohybrid-type composite resin and contains organically modified ceramic nano-particles mixed with conventional glass fillers. Nano-particles in CD contain methacrylic group linked to polysiloxane backbone.<sup>22</sup> Siloxanes, which are chemically similar to glass and ceramic, increase bond strengths. In the present study, combination with CD composite resin showed the highest bond strength ( $16.2 \pm 4.2 \text{ MPa}$ ) in the composite resin groups. Furthermore, CD showed the highest bond strength in combination with all dentin adhesives except BF. This is not consistent with the findings of a previous study,

in which it was found that the type of adhesive played an important role in bond strength when combined with self-etch adhesives and composite resins.<sup>11</sup> Other studies reported that the physical characteristics of composite resins depend upon the quantity of filler or monomers and that these significantly dictate the physical characteristics of composite resins and bond strengths.<sup>23</sup> However, in this study, consistent pattern was not observed in relation to the filler content or constituent ingredients.

Remarkably, the IB-CH combination (Heraeus Kulzer) showed lower bond strength in the IB adhesive group and the CH resin composite group. IB when combined with CD or EQ showed high bond strength regardless of manufacturer ( $p < 0.05$ ). Likewise, in the CH composite group, combinations with XV showed the highest bond strengths. It seems that combination of dentin adhesive and composite resin influence each other to some extent and combinations of same manufacturer's products do not produce higher bond strengths.

## Conclusions

In their instructions to users, manufacturers' usually recommend the use of their own dentin adhesives and composites to achieve maximum bond strength because differences in chemical composition might lead to unexpected chemical reactions that adversely affect bonding. However, within the limitations of this study, no specific advantages in the bond strength could be attributed to same manufacturer combinations.

### Orcid numbers:

Minju Song, 0000-0001-7586-2359

Yooseok Shin, 0000-0003-1121-2570

Jeong-Won Park, 0000-0002-2517-8150

Byoung-Duck Roh, 0000-0001-5219-938x

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