

## Influence of Filler Level on the Bond Strength of Orthodontic Adhesives

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### ABSTRACT

**Objective:** To evaluate the effect of different filler contents of orthodontic adhesives on shear bond strength and Adhesive Remnant Index (ARI).

**Materials and Methods:** Four experimental adhesive groups were created: group 1 was an unfilled urethane-dimethacrylate (UDMA) adhesive, group 2 consisted of UDMA and a filler content of 30 vol%, group 3 consisted of UDMA and a filler content of 50 vol%, and group 4 was manufactured with a filler level of 70 vol% in a UDMA matrix. The embedded filler was silicon dioxide. After etching and priming, stainless steel brackets were bonded to extracted human third molars ( $n = 60$ ) with the experimental adhesives (15 teeth per group). After storage in distilled water for 72 hours, shear bond strength and ARI scores were evaluated. Kruskal-Wallis and post hoc test were performed for statistical analysis.

**Results:** Shear bond strength of UDMA-based adhesives depends on filler content. Higher adhesive filler levels reveal greater bond strength between enamel and stainless steel brackets. ARI scores showed no significant difference among the groups. However, higher filled adhesives (filler content 70 vol%) seem to present higher ARI scores.

**Conclusion:** Highly filled UDMA adhesives offer greater bond strength than do lower filled or unfilled resins.

**KEY WORDS:** Orthodontic adhesive; Filler level

### INTRODUCTION

The major fraction of orthodontic adhesives is made of resin composites. Adhesive composites are complex materials composed of synthetic polymers, particulate ceramic reinforcing fillers, and silane coupling agents that bond the reinforcing fillers to the polymer matrix.<sup>1</sup> In addition, molecules that promote or modify the polymerization reaction are necessary.<sup>1</sup> Over the years the evolution of orthodontic adhesives has en-

compassed several areas, including modifications of filler components, filler levels, and the polymeric part of the resins.<sup>2-6</sup>

As an organic material, Bis-GMA (2,2-bis[4-(2-hydroxy-3-methacryloyloxypropoxy)-phenyl] propane), which was developed by Bowen in 1956,<sup>7</sup> is often in use. However, Bis-GMA reveals a very high viscosity because of the hydrogen bonding interactions that occur between the hydroxyl groups on the monomer molecules. Therefore, Bis-GMA has to be diluted with a more fluid resin, for instance, triethyleneglycol-dimethacrylate.<sup>1</sup> Nowadays, alternative monomer systems such as urethane-dimethacrylate (UDMA) are offered. UDMA presents lower viscosity, more effective light curing, lower water sorption, and greater toughness in comparison with Bis-GMA.<sup>8</sup>

Fillers are added to the polymeric part of the adhesives to provide strengthening, increase stiffness, reduce dimensional changes, and improve handling.<sup>9-13</sup> Most current composites are filled with silicate particles based on oxides of barium, strontium, zinc, aluminum, or zirconium.<sup>14</sup> To provide a strong bond between the organic monomer and the inorganic filler particles, silanes with functional methacrylate groups are necessary. The most commonly used silane is 3-methacryloyloxypropyl trimethoxysilane.

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To start polymerization, adhesives need to be activated by an initiator system. Camphorquinone, an  $\alpha$ -diketone, is part of the initiator system in light-curing adhesives and instigates polymerization after absorbing energy in the presence of visible blue light with a wavelength of 440–480 nm.<sup>6</sup>

In orthodontics, a lot of literature<sup>15–19</sup> about shear bond strength (SBS) of brackets is obtainable. The problem of orthodontic bracket bonding is its semipermanence. Bond strength should be high enough to resist accidental debonding during treatment but also low enough so that the bracket can be removed from the tooth without generating excessive force that might damage the tooth.<sup>17</sup> Several studies<sup>15–18</sup> demonstrated that clinically adequate bond strength for a stainless steel bracket to enamel should be 6–8 MPa. Nevertheless, the influence of adhesive filler level on SBS of stainless steel brackets has not been sufficiently investigated so far.

The purpose of the present investigation was to evaluate the effect of different filler contents of orthodontic adhesives on SBS and the Adhesive Remnant Index (ARI). The experimental adhesives were manufactured of the monomer UDMA and functional silane-treated silicon dioxide ( $\text{SiO}_2$ ) fillers of varying filler level. The SBS and ARI of stainless steel brackets with the experimental adhesives were measured.

## MATERIALS AND METHODS

The experimental adhesives consisted of UDMA (initiator: camphorquinone) as a monomer matrix and functional silane-treated experimental  $\text{SiO}_2$  fillers (Ivoclar-Vivadent, Schaan, Liechtenstein; filler size: 0.01  $\mu\text{m}$ ).

Four different adhesives were produced: group 1 was an unfilled UDMA polymer (control group), group 2 consisted of UDMA and a filler content of 30 vol%, group 3 was an adhesive made of UDMA and a filler level of 50 vol%, and group 4 consisted of UDMA and a filler content of 70 vol%.

The adhesive mixtures were produced by hand mixing the monomer and filler in correct portions. The adhesive matrix was mixed in a Speed mixer device (Speed Mixer DAC 150FVZ, Hauschild Engineering, Hamm, Germany) for 60 seconds (1800 rpm) to attain a homogenous mixture. After preparation the manufactured adhesives were stored in opaque receptacles to avoid premature polymerization.

A total of 60 recently extracted third molars were stored in 0.5% chloramines-T. The roots were removed and the crowns were embedded in autopolymerization acrylic resin so that the facial surface of each tooth was parallel to the base of the polymer. The teeth were cleaned with a nonfluoridated pumice



**Figure 1.** Overall view of the shearing rod used for performing shear bond strength tests.

paste and rubber cups. The enamel surface of each tooth was etched with 20% phosphoric acid (Gluma Etch 20 Gel, Heraeus Kulzer, Hanau, Germany) for 30 seconds. A frosted appearance indicated a successful etch. After this procedure the primer Transbond XT Primer (3M Unitek, Monrovia, Calif) was applied, gently thinned with air, and light cured for 20 seconds (Ortholux LED, 3M Unitek). Afterwards, stainless steel brackets (Ormesh, Ormco Corporation, Glendora, Calif) were bonded to the teeth with the four described experimental adhesives (15 teeth per group). All brackets were placed centrally on the flat buccal surfaces of the teeth. The samples were then light cured with a light-emitting-diode curing device (Ortholux LED, 3M Unitek) for 20 seconds. All brackets were bonded by the same operator.

All samples were stored for 72 hours in distilled water before SBS tests were performed with a universal testing machine, Zwick 1446 (Zwick, Ulm, Germany; volume = 1 mm/min). A knife-edge shearing rod was used to deliver the shear force at the interface of the bracket base and enamel (Figure 1). The embedded tooth and the adhesively fixed bracket were positioned in the testing machine so that the bracket slot was placed horizontally. All brackets were shear tested to failure. The surface area of the bracket bases was determined by measuring their length and width and computing the mean area. The SBS was evaluated by the formula  $\sigma_{\text{shear}} = F_{\text{max}}/A_{\text{bracket base surface}}$  (MPa). Furthermore, ARI scores were assessed on a 0–3 scale as follows: 0 = no adhesive on enamel, 1 = <50% adhesive on enamel, 2 = >50% adhesive on the enamel, 3 = 100% adhesive on enamel.

Statistical analysis was performed by the program

**Table 1.** Statistical analysis of shear bond strength (SBS) and Adhesive Remnant Index (ARI) compared with the unfilled control group (significance level  $P = .05$ )<sup>a</sup>

Experimental adhesive	SBS	ARI Index
30 vol% SiO <sub>2</sub> -filled UDMA-based adhesive	NS	NS
50 vol% SiO <sub>2</sub> -filled UDMA-based adhesive	.005	NS
70 vol% SiO <sub>2</sub> -filled UDMA-based adhesive	.005	NS

<sup>a</sup> SiO<sub>2</sub> indicates silicon dioxide; UDMA, urethane-dimethacrylate; and NS, not significant.

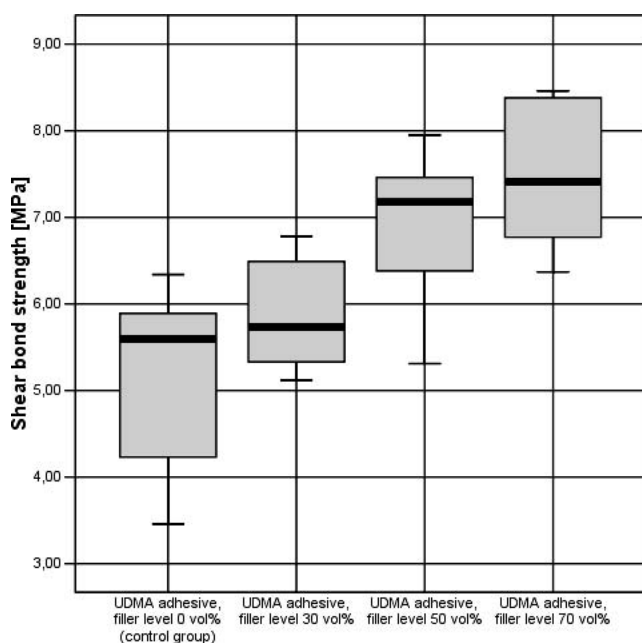
SPSS for Windows 12.0 (SPSS Inc, Chicago, Ill) with the Kruskal-Wallis and post hoc test. Means and standard deviations were calculated. The significance level was set at  $\alpha = .05$ .

## RESULTS

When the 30 vol% filled adhesive was compared with the unfilled control group, no significant enhancement of SBS could be measured (Table 1). The 50 vol% and 70 vol% SiO<sub>2</sub>-filled adhesives showed a significant ( $P = .005$ ) increase of SBS in comparison with the control group (Figure 2). The highest median values of SBS were measured when using a UDMA-filled adhesive with a filler content of 70 vol% ( $7.41 \pm 0.79$  MPa). ARI scores did not reveal a significant change among the groups (Table 2).

## DISCUSSION

In the present study a UDMA matrix was preferred for the experimental adhesive because UDMA-based

**Figure 2.** Shear bond strength (MPa) of urethane-dimethacrylate-based adhesives (median: 25%/75% percentiles: minimum, maximum).**Table 2.** Adhesive Remnant Index (ARI) of urethane-dimethacrylate (UDMA)-based adhesive groups<sup>a</sup>

Group	ARI score			
	0	1	2	3
Unfilled UDMA-based adhesive (control group)	0	1	3	6
30 vol% SiO <sub>2</sub> -filled UDMA-based adhesive	0	0	3	7
50 vol% SiO <sub>2</sub> -filled UDMA-based adhesive	0	0	3	7
70 vol% SiO <sub>2</sub> -filled UDMA-based adhesive	0	0	2	8

<sup>a</sup> SiO<sub>2</sub> indicates silicon dioxide.

composites reveal some advantages (eg, lower viscosity, lower water sorption, and greater toughness) in comparison with Bis-GMA.<sup>8</sup> In addition, Ferracane and Condon<sup>20</sup> stated that a UDMA matrix may cure more extensively than Bis-GMA-based composites. Nevertheless, Söderholm and Shang<sup>21</sup> described the depth of cure of some UDMA-based composites as less in comparison with Bis-GMA composites because of a greater mismatch in the refractive index between monomer and filler. Peutzfeld<sup>2</sup> stated that it might be difficult to compare results of existing in vitro and in vivo studies about UDMA and Bis-GMA composites because actual differences caused by dissimilar monomer systems are often camouflaged by the fact that the materials differ in many aspects (eg, type and amount of initiators and fillers and the quality of silanization of the filler particles). It has been reported that the filler concentration and the nature of bonding between filler particle and resinous matrix play a more prominent role in influencing the properties of a composite than the choice of monomer type.<sup>22</sup> However, Peutzfeld<sup>2</sup> described the choice of monomer system as a relevant factor in influencing the composite properties.

In the current investigation the filler level was 0 vol% (control group), 30 vol%, 50 vol%, and 70 vol%. According to previous reports<sup>1,3,7</sup> the best mechanical properties could be achieved by incorporating high concentrations of filler particles of various sizes into the resin. Brantley and Eliades<sup>8</sup> illustrated that the volume fraction of orthodontic adhesives may range from 30% to 75%. Fillers are added to the polymeric part of the adhesives to provide strengthening, increase stiffness, reduce dimensional changes, and improve handling.<sup>9-13</sup> In orthodontics, the polishing qualities and surface smoothness of the filled adhesives are important features during cleanup procedures after debonding.<sup>8</sup>

Current composites are filled with silicate particles based on oxides of barium, strontium, zinc, aluminum, or zirconium.<sup>14</sup> Several composites obtain a uniform distribution of small filler sizes, whereas others utilize a broader distribution of particles.<sup>13,14</sup> Many different types of fillers are utilized in orthodontic adhesives.

Nevertheless, according to Ferracane<sup>1</sup> there is no superiority of any specific filler because every type of filler reveals advantages and disadvantages during its clinical use.

To offer a strong bond between the organic monomer and the inorganic filler particles, silanes with functional methacrylate groups are required. Silane coupling agents were applied in dentistry for bonding organic to inorganic materials. A silane coupling agent is able to connect SiO<sub>2</sub> groups with an adhesive consisting of a UDMA or Bis-GMA system because of its bipolar structure. Studies in restorative dentistry demonstrated that filler content in composites does influence polymerization shrinkage and that decreased filler levels cause greater polymerization shrinkage.<sup>23–26</sup> James et al<sup>23</sup> compared microleakage of two orthodontic adhesives. They found out that the lower filled adhesive revealed more microleakage than the higher filled counterpart. However, they used commercially available adhesives that differed in only 3% filler level. Orthodontic adhesives have to present good handling to the operator, that is, during bracket positioning on the tooth the adhesive must offer enough stiffness and should not permit the bracket to slide away. To reach this goal, an orthodontic adhesive should have a sufficient filler level. During bracket debonding, residual adhesive remnants have to be easy to remove. Orthodontic adhesives must offer biocompatibility and adequate bond strength.

Analysis of the data obtained in the present study offers some insight into the influence of filler level of adhesives on bond strength of stainless steel brackets. It could be summarized that bond strength of UDMA-based adhesives depends on filler content. Higher adhesive filler levels reveal greater bond strength between enamel and stainless steel brackets. ARI scores showed no significant difference among the groups. However, more adhesive remnants seem to remain on the bracket base during debonding after applying the unfilled adhesive. An explanation for these findings could be that filler-reinforced adhesives reveal greater strength in comparison with unfilled adhesive. Hence, filler reinforcement of adhesives increased bond strength, and debonding occurred on the bracket-adhesive interface (adhesive fracture). The unfilled adhesive showed less bond strength because of reduced stiffness of the resin in comparison with the filled adhesive, and debonding occurred within the adhesive resin (cohesive fracture) and not on the bracket-adhesive interface. Consequently, unfilled adhesives tend to lower ARI scores.

Another explanation for lower ARI scores of the unfilled adhesive might be that unfilled resins reveal lower viscosity than their filled counterparts and are more effective in coating undercuts of bracket bases. There-

fore, debonding of stainless steel brackets with an unfilled adhesive appears within the polymer and not on the bracket-adhesive interface. Ostertag et al<sup>27</sup> evaluated the influence of adhesive filler concentrations on shear, torsional, and tensile bond strengths of ceramic brackets. They supposed an enhancement of bond strength with increased filler level with ceramic brackets. It was described earlier that filled adhesives reduce polymerization shrinkage,<sup>24–26</sup> improve handling during bonding, and reduce microleakage.<sup>23</sup>

The results of the present study confirm the positive effect of filled orthodontic adhesives because the bond strength of stainless steel brackets was increased with a UDMA-based and SiO<sub>2</sub>-filled experimental adhesive. It is a common belief<sup>17,28,29</sup> that clinically adequate bond strength for a stainless steel bracket to enamel should be 6–8 N/mm<sup>2</sup>. In the present investigation the bond strength values of the unfilled and filled adhesives are approximately within this range.

## CONCLUSIONS

- Bond strength of stainless steel brackets depends on the filler level of orthodontic adhesives.
- Higher filled UDMA adhesives seem to provide greater bond strength than do lower filled or unfilled resins.
- Additionally, filled adhesives seem to provide more adhesive fractures on the bracket-adhesive interface, whereas unfilled resins tend to cohesive fractures.

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