

# Orthodontic resin under water immersion

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Newman<sup>1</sup> introduced direct bonding with composite resin to the orthodontic field in 1965 and it has since become a widely accepted procedure for attaching brackets directly to enamel.<sup>2,3</sup> Composite resin has also been used successfully in restoring areas of anterior teeth, in place of an amalgam restoration in posterior teeth, and for sealing pits and fissures in posterior occlusal surfaces.<sup>4,6</sup> The most important shortcoming of dental composite is its relatively poor resistance to wear when used clinically.<sup>7,8</sup> Some investigators have shown that increased quantities of inorganic fillers improve a resin's mechanical properties<sup>9</sup> and resistance.<sup>9,10</sup> However, the absorption and desorption of water by the composite resin's polymer matrix could cause filler-matrix debonding<sup>11,12</sup> or even hydrolytic degradation of the fillers.<sup>13-15</sup>

Orthodontic composite consists of 24% to 72% inorganic fillers.<sup>16</sup> During a 2- to 3-year orthodontic treatment period, the composite is essentially immersed in water – saliva – where it may desorb and absorb water. This could cause filler-matrix debonding or hydrolytic degradation of the filler and affect bond strength, hardness, and wear resistance. However, few researchers have paid much attention to this point in orthodontics.<sup>17-22</sup> Changes in bond strength and the debonding interface distributions of composite resin following water immersion depend on the duration of the immersion. Hence, the purposes of this study were to: (1) compare the bond strength and debonding interface distribution of orthodontic composite resin after water immersion for various times; and (2) analyze the relationships among bond strength, debonding

## Abstract

The absorption and desorption of water by a polymer matrix of composite orthodontic resin could cause debonding of the filler-matrix or hydrolytic degradation of fillers and loss of bond strength. In this study, the bond strength of brackets directly bonded with orthodontic composite to the enamel surface of premolars was measured with an Instron machine; the debonding interface distribution was analyzed by scanning electron microscope and energy dispersive x-ray spectrometry following water immersion for 1, 2, and 3 days, and 1, 2, 4, 8, 16, 24, and 32 weeks, respectively. The results show that, under water immersion, bond strength may gradually weaken over time. The greatest loss occurs initially, followed by a period of relative stabilization, and then a weaker reduction after 24 weeks. The greater the time in water immersion, the less the bond strength and the greater the destruction of the composite resin. The debonding interface occurs between bracket and resin.

## Key words

Bond strength • Debonding interface • Orthodontic resin • Water immersion

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	Mean (kg/mm <sup>2</sup> )	S.D. (kg/mm <sup>2</sup> )
Day 1	0.73	0.14
Day 2	0.72	0.02
Day 3	0.72	0.01
Week 1	0.69	0.14
Week 2	0.67	0.11
Week 4	0.58	0.06
Week 8	0.62	0.09
Week 16	0.60	0.14
Week 24	0.48	0.13
Week 32	0.46	0.24

Note: 10 samples in each group  
F=5.10                      P=0.0001<0.05

	Bracket-resin		Within the resin		Resin-enamel	
	Mean	SD	Mean	SD	Mean	SD
Day 1	42	17	24	11	34	11
Day 2	42	16	26	11	32	12
Day 3	44	13	23	10	33	16
Week 1	40	18	26	14	35	12
Week 2	40	13	27	11	33	15
Week 4	39	19	30	16	31	14
Week 8	40	13	29	13	31	10
Week 16	42	10	31	12	27	7
Week 24	39	13	33	12	28	16
Week 32	40	8	36	13	24	11

SD: standard deviation

interface within the resin itself, the amount of water, and the duration of immersion.

**Materials and methods**

One hundred premolars were extracted from young patients (9 to 16 years old) for the purpose of orthodontic treatment. After extraction, the teeth were washed, immersed in physiological saline, and stored in a closed plastic box until testing. The criteria of tooth selection were as follows: the crowns were grossly perfect, without caries, and had not been treated with chemical agents, such as hydrogen peroxide, alcohol or formalin. The teeth were randomly divided into 10 groups of 10 teeth each.

Dyna-Lock maxillary premolar brackets (batch No. 018-503, 3M/Unitek Corp., Monrovia, Calif) were selected for the study. The bracket base was arced with an area approximately 3.1 mm x 3.4 mm (10.54 mm<sup>2</sup>). Concise orthodontic composite resin (3M Corp., St. Paul, Minn) was used to bond the brackets to the teeth.

To prepare the teeth for bonding, the buccal surface of each crown was polished with a pumice powder (Prophycol fine particle, Moyco Industries Inc., Philadelphia, Penn) water paste containing no fluoride or oil for 10 seconds, then washed with water spray and dried with air spray. The Concise acid etchant (batch No. 2BC, pH: 0.21) was applied for 15 seconds,<sup>23</sup> then rinsed with an abundant water spray for 10 seconds and dried again with air spray. The etched surface became chalky-white in appearance. The

bracket base was outlined in pencil on the center of the enamel. The surface outside the marked area was coated with red nail polish to standardize the bonding area. The bonding agent (batch No. paste A:2EU1, paste B:2HA1) was then applied to the central chalky-white surface of the enamel and bracket base. Orthodontic composite resin (batch No. paste A:2HE1, paste B: 2HC1) was also applied to the bracket base and the bracket was pressed onto the demarcated etched buccal enamel with a placement scaler. Once the bracket was in the correct position, the scaler was removed. Excess composite resin was removed before setting and the bracket was left undisturbed until set.

Ten groups of specimens were incubated in a 37°C water bath for 1, 2, and 3 days, and 1, 2, 4, 8, 16, 24, and 32 weeks, respectively. Tensile bond strength was tested with a universal testing machine (Model 1000, Instron Corp., Boston, Mass) at a cross-head speed of 2 mm/min. The distributive percentages of the debonding interfaces were examined and calculated, using a scanning electron microscope (Canscan Corp., Serial 4, Cambridge, England), and mapping of energy dispersive x-ray spectrometry (Philips Corp., EDAX, SW9100, Hillegon, Holland). Bond strengths and debonding interface percentages were recorded. Means and standard deviations were determined. Data were analyzed with SAS software (SAS Institute Inc., Cary, NC) by one-way and two-way ANOVA.<sup>24</sup> Scheffe's test was then used to identify significant differences be-

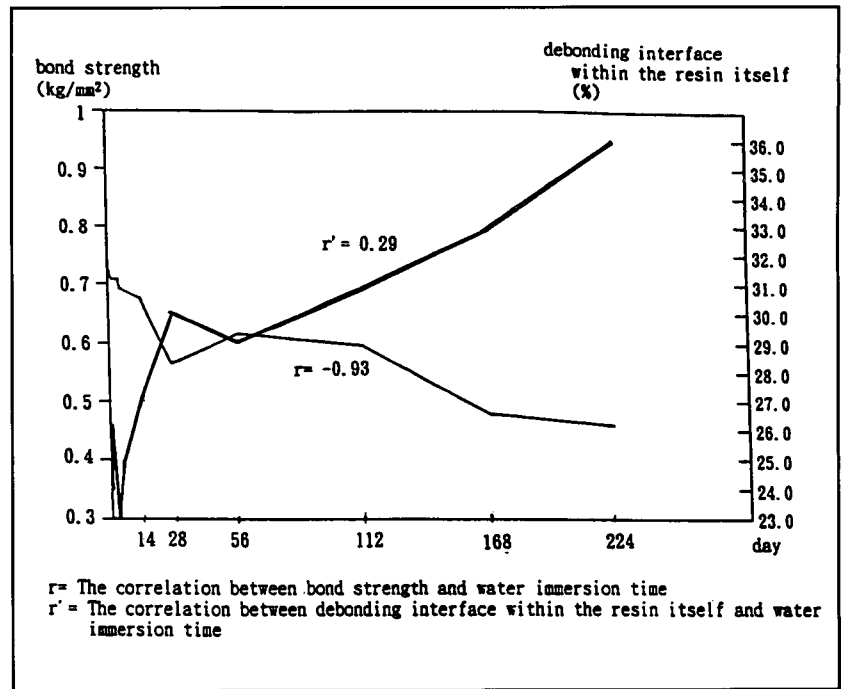
tween the levels of the bond strength and debonding interface distribution under investigation. Detailed procedures of bond strength detection and debonding interface analysis have been described in a previous study.<sup>23</sup>

### Results

Bond strengths are shown in Table 1. Statistical analysis of bond strength with one-way ANOVA showed the F value to be 5.10, which was statistically significant ( $P=0.0001$ ). Bond strength was further analyzed with Scheffe's test, with an  $\alpha$  value of 0.05. This showed that bond strength on the first day was greater than bond strength at any time from the second day to the 32nd week, and bond strengths from the first day to the 24th week were greater than bond strength at the 32nd week. The differences were statistically significant; however, there were no statistically significant differences between each successive time interval. The correlation value ( $r$ ) between the time of water immersion and bond strength was  $-0.93$  with a statistically significant difference ( $P=0.0001$ ) (Figure 1).

A greater decrease in bond strength occurred during the first four weeks (from  $0.73 \text{ Kg/mm}^2$  to  $0.58 \text{ Kg/mm}^2$ , difference of  $-0.15 \text{ Kg/mm}^2$ ) than during the remainder of the study (from  $0.58 \text{ Kg/mm}^2$  to  $0.46 \text{ Kg/mm}^2$ , difference of  $-0.12 \text{ Kg/mm}^2$ ).

In the debonding interface analysis, these debonding interfaces were found: (1) between bracket base and resin, (2) between resin and enamel, and (3) cohesive failure within the resin itself. There was no enamel detachment. The distributive percentages of the various debonding interfaces are shown in Table 2. The statistical relationships between 10 water immersion times and three types of debonding interface distributions were analyzed by using two-way ANOVA. The F value of comparison between 10 water immersion times and percentages of bond failure distribution interaction was 4.35, which was statistically significant ( $P=0.0001$ ). The F value of comparison among 10 immersion times was 0.01 and was not statistically significant ( $P=1.00$ ). The F value of comparison among three debonding interface distributions was 23.92, which was statistically significant ( $P=0.0001$ ). The  $\alpha$  value of 0.05 was chosen for the post hoc treatment with the Scheffe's test of three types of debonding interface. The results showed that the debonding interface between metal and resin was greater than that within the resin itself or between resin and enamel with a statistically significant difference. However, there was no difference between



the within-resin and resin-enamel interfaces. The F value of the debonding interface within the resin itself among 10 immersion times was also analyzed with one-way ANOVA and was found to be 1.06, which was not statistically significant ( $P=0.40$ ).

The correlation value ( $r'$ ) between time of water immersion and debonding interface within the resin itself was 0.29 with a statistically significant difference ( $P=0.004 < 0.05$ ) (Figure 1).

The correlation value between bond strength and debonding interface within the resin itself was 0.30 with a statistically significant difference ( $P=0.003$ ).

### Discussion

Bond strength on the first day was greater than that at any time from the second day to the 32nd week, and the difference was statistically significant. Differences among the successive time intervals from the second day to the 32nd week were not statistically significant. After a relatively large initial loss, bond strength stabilized. There were no statistically significant differences in bond strength of Concise within 24 weeks of immersion. However, bond strength by the 32nd week had decreased significantly, indicating that immersion times greater than 24 weeks will result in diminished bond strength. Statistical analysis of the bond strength showed no differences within the first 24 weeks of water immersion. The value of correlation ( $r$ ) between water immersion and bond strength was  $-0.93$  with a

**Figure 1**  
The correlation between bond strength, debonding interface within the resin itself and water immersion time

statistically significant difference ( $P=0.0001$ ), indicating that the greater the time of immersion, the lower the bond strength.

Although there were no statistically significant differences in debonding interfaces within the resin itself among 10 water immersion times ( $P=0.40$ ), the value of correlation between debonding interface within the resin itself and immersion time was 0.29, with a statistically significant difference, indicating that the greater the immersion time, the greater the debonding interface within the resin itself. This indirectly suggested that the absorption and desorption of water by the polymer matrix of composite could cause filler-matrix debonding or even hydrolytic degradation of the fillers. The value of correlation between bond strength and debonding interface within the resin itself of 10 various times of water immersion was 0.30 with a statistically significant difference ( $P=0.003$ ), indicating that the value of the bond strength is related to the debonding interface within the resin itself. The lower the bond strength, the greater the debonding interface within the resin itself.

The rate of debonding at the bracket-resin interface remained nearly constant (39% to 42%) during 32 weeks of immersion. However, the data of the debonding interface within the resin itself and between the resin and the enamel displayed a linear relationship: the longer the immersion, the more the debonding interface occurred within the resin itself and the less the debonding interface occurred between enamel and resin.

Concise orthodontic composite produces a good bond strength.<sup>25,26</sup> It does, however, contain inorganic fillers<sup>20</sup> that may desorb and absorb water, which could cause filler-matrix debonding or hydrolytic degradation of the filler

and may affect the bond strength and debonding interface distribution following long-term water immersion.

Soderholm<sup>20</sup> investigated the leakage of filler elements from Concise after storage in water. Filler elements are the main component of Concise—76.8% by weight fraction and 0.61 by volume fraction. Microfilled composite contains only about 0.39 volume fraction. The main composition by weight was Si with additions of 0.1% to 1% Al, 0.01% to 0.1% Sb, Ti and Mg, as well as 0.001% to 0.01% F and B. Differences in ordered bonds may be responsible for the leakage of filler elements from Concise.<sup>20</sup> Quartz has a crystalline structure with regularly distributed long-range ordered bonds, while the microfill particles have irregularly distributed short-range ordered Si-O-Si bonds. When these different fillers are bonded to resin matrices and immersed in water, the matrices swell and radial tensile stresses are induced on the filler interfaces, thereby straining the Si-O-Si bonds inside the fillers. Because the amorphous fillers had initially strained Si-O-Si bonds, these fillers will reach still higher energy levels than will the quartz fillers, making the amorphous fillers more susceptible to stress corrosion. Similar reactions can also occur in the stressed quartz particles. The induced stress, caused by water swelling of the matrix, would also influence the stress corrosion attack of the filler. The greater the water immersion time, the greater the leakage of composite.

Newman et al.,<sup>18</sup> Williams et al.<sup>19</sup> and Muria et al.<sup>27</sup> reported that the bond strength of acrylic cement showed a tendency to decrease over time with water immersion. Cohl et al.<sup>21</sup> showed slight increases in bond strength after 30 days and 60 days of water immersion. Beach and Jalaly<sup>17</sup> reported increases in bond strength with primer

and decreases in bond strength without primer for water immersion after 6 months. The causative mechanism may be the use of sealant primer which decreases the tensile bond strength.<sup>17,22</sup> The primer reduces the susceptibility of the bond to attack by water. It is possible that the effect of a primer depends on the thickness of the layer. If it becomes too thick, it may act as a separating agent and the bond strength decreases. In this study, bond strength after 32 weeks of water immersion was the same as the bond strengths Beech and Jalaly<sup>17</sup> and Rock<sup>22</sup> found using a sealant as the bonding material.

In an earlier study,<sup>23</sup> no statistically significant differences were found in bonding strength following etching times of 15 seconds and 60 seconds. However, enamel detachment was found with an etching time of 60 seconds. Hence, the etchant was applied 15 seconds in this study.

The large variation in standard deviations of debonding interface between groups may be due to uneven filler distributions within the composite resin and various particle size of filler which may affect the result of immersion for various times.

From the view point of debonding interfaces, greater debonding interfaces were located between metal and resin. Methods for improving the mechanical retention or for choosing the best type of bracket base will be analyzed in a future study.

### Conclusion

The bond strength of Concise orthodontic composite resin gradually fell over time under water immersion. The initial loss of bond strength is the greatest, and may be followed by a period of relative stabilization, with a notable diminishment after 24 weeks. The greater the time of im-

mersion, the greater the destruction of composite resin itself and the less the bond strength. The debonding phenomenon of orthodontic composite resin may gradually and automatically occur under water immersion due to characteristics of the composite resin.

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