



## ASSESSMENT OF BOND PERFORMANCE OF THE CURRENT SELF-ETCH ADHESIVE SYSTEMS TO DENTIN AFTER AGING

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### ABSTRACT

**Objective:** The aim of the investigation was to evaluate effect of water storage on microshear bond strength using two adhesive resin systems. **Material and methods:** A total of sixteen teeth were divided into two groups according to the adhesive system used; Group 1: 3M Single Bond Universal (SBU) and Group 2: G-Premio Bond (GP). Each group was further subdivided into two subgroups; Subgroup A which was stored immersed for 24 hours in distilled water and Subgroup B stored for 2 months. Four micro-cylinders of resin composite (3M ESPE Filtek TM Z250 XT), were placed on the conditioned dentine surfaces. Bonding performance was assessed via microshear bond strength test (mSBS) using universal testing machine after 24 h and 2 months' water storage. The data was registered using computer software in MPa. **Results:** The results revealed that the highest bond strength values were revealed in samples bonded with 3M Single Bond Universal, followed by samples bonded by G-Premio Bond after 24h water storage. After 2 months' water storage, the samples bonded by G-Premio Bond showed higher values, but with no statistical significant difference. **Conclusion:** The water aging resulted in a significant decline in the microshear bond strength values.

**KEYWORDS:** adhesive resin systems, micro-shear bond strength, Resin composite, water storage.

### INTRODUCTION

It is predictable that dentin bonding has less consistent outcomes attributable to its heterogeneous nature and characteristics: type of dentin, collagen content, dentinal tubules structure and direction, besides outward dentinal fluid flow. Bonding to dentin was additionally convoluted by the presence of smear layer. Dentin bonding agents have been introduced to improve adhesion, and to overcome such obstacles and complications <sup>(1,2)</sup>.

In the recent decades, the development of self-etch adhesive resins allowed simpler bonding pro-

ocols, saving the clinical time and effort. Self-etch adhesives are commonly comprised of methacrylate monomers, initiators and solvents. Water is also essential for the acidic monomers ionization, thus permitting substrate demineralization and subsequent infiltration of the monomer <sup>(3)</sup>. Hypothetically, self-etch adhesives reduce the occurrence of non-infiltrated collagen when compared to etch-and-rinse adhesives, based upon their ability to demineralize and infiltrate the substrate simultaneously <sup>(4)</sup>. Praiseworthy, the acidic monomers ionization allows chelation and collagen fibre hybridization, which are essential mechanisms in the dental bonding <sup>(3)</sup>.

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Worth mentioning that, the durability of the adhesive bond is of significant importance for longevity of adhesive restorations. Still, polymerization shrinkage and the resultant stresses of resin composites together with the hydrolysis of the hybrid layer/adhesive material are considered the most common reasons for failure of bonding to the dental structure. What is more, the acidic nature of self-etch primers/adhesives is also among the factors of the hydrolysis phenomena<sup>(5)</sup>.

Hence, long term stability of resin bonded dentin remains debatable. The quality of the adhesion to dentin has been assessed by laboratory tests such as tensile or shear test. Some studies described reduction in bond strength after long water storage<sup>(5-7)</sup>.

Accordingly, the purpose of this study was to evaluate the effect of water storage (24h and 2 months) on the microshear bond strength of contemporary adhesive resin systems to dentin substrate.

## **MATERIALS AND METHOD**

Two commercially available universal adhesive resin systems; Single Bond Universal, (3M Deutschland GmbH, Germany) (SBU) and G-Premio BOND (GC corporation, Tokyo, Japan) (GP) and one resin composite (3M ESPE Filtek™ Z250 XT, USA) were utilized in this study.

### **Preparation of the specimens:**

Sixteen sound human third molars were used in the current study. The teeth were disinfected in 0.2% thymol for 48 hours at 37 °C, then stored at 4 °C in physiologic saline which was changed every week and used within 2 months.

The teeth were embedded in a polyethylene tube (15 mm x 25 mm) with their long axis parallel to that of the tube which was filled with cold-cured acrylic resin to the cemento-enamel junction. The teeth were ground horizontally at the occlusal third using a low-speed diamond disk (Edetal Golden

S.A.W., Switzerland) under water cooling. The exposed dentine surfaces were polished using silicon carbide paper (600 grit) to form standardized smear layer.

### **Grouping of the specimens:**

The teeth were randomly divided into two groups (n=8) according to the adhesive system used; Group 1: SBU and Group 2: GP. Each group was further subdivided into two subgroups according to the storage period; Subgroup A which was immersed for 24 hours in distilled water and Subgroup B was stored for 2 months.

### **Preparation of resin composite micro-cylinders for bond strength testing:**

Each adhesive system was applied according to the manufacturer's instructions and light cured using LED light curing unit (Premium plus light cure C02-S, Premium Plus UK, England) at full mood (1,200 mW/cm<sup>2</sup> for 10 seconds). For each tooth, 4 polyethylene tubes (0.9 mm height and 0.5 mm internal diameter) were filled with resin composite (3M ESPE Filtek™ Z250 XT), placed over the conditioned dentine surface then cured for 20 seconds with the same curing mood. The polyethylene tubes were later removed using sharp lancet. The samples of subgroup 1A and 2A were stored at distilled water for 24 hours before the microshear bond strength testing. Samples of subgroups 1B and 2B were stored for 2 months' time period.

The specimens were checked under a stereomicroscope (Olympus, Tokyo, Japan) at a magnification of 20× prior to bond strength testing. Any specimen that presented a detectable interfacial defect was excluded and replaced.

### **Microshear bond strength testing (µSBS):**

The acrylic resin block with the sample was attached to the lower fixed head of the universal testing machine (Instron® model 3345, England). Each

micro-cylinder was subjected to a  $\mu$ SBS test using a stainless steel wire 0.14-inch diameter attached to the upper movable head of the testing machine, that was placed as close as possible to the composite-dentine interface. The test was applied with tensile mode of force at a crosshead speed of 1.0 mm/min up to specimen failure. The force required for failure (Newton) was divided by the surface area ( $\text{mm}^2$ ) to calculate the shear bond strength in MPa using machine software (BlueHill 3 Instron England). Results of micro-shear bond strength were calculated and subjected to statistical analysis.

### Statistical analysis

Statistical analysis was performed with IBM® SPSS® Statistics Version 25 for Windows. The mean and standard deviation values were calculated for each group. Normality test was performed using Kolmogorov-Smirnov test and revealed normal distribution between values of each group. Homogeneity test was performed using Levene's test and revealed homogenous distribution between all variables. Therefore, 2 independent sample T test was performed between the variables (with significance level was set at  $P \leq 0.05$ ) to reveal the statistical significant difference.

### RESULTS

The highest microshear bond strength values were recorded in samples bonded with SBU after 24h of water storage ( $26.27 \text{ MPa} \pm 4.81$ ), followed by samples bonded by GP ( $17.34 \text{ MPa} \pm 1.98$ ) with no significant difference.

After 2 months' water storage, the samples bonded by GP showed higher, but with no statistical significant difference microshear bond strength ( $9.72 \text{ MPa} \pm 2.85$ ) when compared with SBU ( $9.06 \text{ MPa} \pm 3.06$ ). In both groups, the water storage showed a statistical significant reduction in the microshear bond strength values.

**TABLE (1)** Shear bond strength values of the two adhesive systems after 24 hours and 2 months water storage.

Adhesive Storage Time	SBU Mean (MPa) $\pm$ SD	GP Mean (MPa) $\pm$ SD	P-value
<b>24 hours</b>	$26.27 \pm 4.81$	$17.34 \pm 1.98$	0.014
<b>2 months</b>	$9.06 \pm 3.06$	$9.72 \pm 2.85$	0.61
<b>P-value</b>	0.000 *	0.001 *	

\* Indicates the mean difference is statistically significant at the 0.05 level.

### DISCUSSION

To mimic the clinical scenario thoroughly, artificial saliva can be used for aging, but bond performance diminutions obtained were comparable to those obtained with water degradation. Consequently, the specimens' aging by water storage was designated <sup>(8)</sup>. Being the most frequently used artificial aging technique <sup>(9)</sup>, in the current study distilled water was used as storage medium for the bonded substrates, utilized for 24 h and 2 months at 37 °C.

The microshear bond strength test was designated in the present study to attain regional mapping and depth profiling of the substrate. Further, the small specimens' size allows many assessments to be performed on the same substrate. Wire and loop was nominated for exerting loading forces over specimens not only to the bonding area under evaluation, but even to the near sites of the adhesive interface resulting in the evaluation of the bond strength of the adhesive and its surroundings, with a better expression of stress distribution at the edge of the bonding area <sup>(10)</sup>.

With respect to the results of the current study, the highest microshear bond strength values were recorded in samples bonded with SBU after 24 h water storage, when compared to those bonded by GP with no significant difference between the two subgroups. This might be explicated by the

bonding mechanism of SBU established by simultaneous demineralization and infiltration of tooth substrate. Where, such bonding agent can be categorized with respect to their pH as being: ultra-mild eliminating the smear layer, integrating the smear plug in the resin tag, resulting in a shallow, uniform resin infiltrated dentin layer<sup>(11)</sup>.

In addition, SBU and GP comprise 10- MDP (10-methacryloyloxydecyle dihydrogen phosphate) which is a highly hydrophilic functional monomer dissolved in water. MDP is capable of interacting chemically with tooth minerals, enhancing the micro-mechanical adhesion<sup>(12)</sup>. Besides, when mixed with HEMA (in SBU), it is capable of improving the wettability of dentin, leading to better interaction of the adhesive with hydroxyapatite crystals decreasing the solubility of those crystals<sup>(13)</sup>. In addition, MDP may chemically interact with tooth substrates, increasing the hydrolytic stability of the hybrid layer, hence resulting in long-term bonding<sup>(14)</sup>.

What is more in SBU is the presence of the Vitrebond copolymer, being capable of dissipating the stresses at the adhesive interface owing to its chemical interaction with apatite minerals. In addition, Vitrebond copolymer is recognized to encourage stability against humidity deterioration<sup>(15,16)</sup>.

Moreover, the nanofillers in SBU forming thicker adhesive layer and consequently a flexible interface relieving interfacial stresses between the resin composite undergoing polymerization shrinkage and the rigid dentinal interface<sup>(17)</sup>. This was confirmed in other studies, stating that the addition of fillers reinforces the hybrid layer, improving the bonding and decreasing the leakage<sup>(18)</sup>.

With reference to GP, Papadogiannis et al., reported a decrease in its degree of conversion as a result of presence of three monomers interfering with the conversion [10-MPD, 4-MET (4-methacryloxyethyl trimellitic acid) MDTP Methacryloyloxydecyl dihydrogen thiophosphate)]. They also reported that the adhesive consistency after setting was a soft gel with poor cohesive strength<sup>(19)</sup>.

It is documented generally that the presence of water as a solvent in the composition of self-etch adhesive systems is essential to ionize the acidic monomers and trigger the demineralization process, while the other organic co-solvents like acetone are added to form an azeotropic mixture with water. This mixture accelerates the removal of excess water by means of air-drying, promoting the diffusion of monomers into the dentin<sup>(20,21)</sup>. This could be one reason for the satisfactory results of the GP when compared to SBU.

Ethanol containing adhesive (SBU) has more affinity for chasing water than acetone containing one (GP). Their evaporation increases the concentration of monomers in the adhesives, which lowers the vapour pressure of the remaining residual solvents, making it impossible to evaporate all solvents during the air-drying<sup>(22)</sup>. The residual water and solvents are responsible for producing localized areas of incomplete monomer polymerization, creating porosities in the bonded interfaces, which consequently might allow inward diffusion of water molecules during storage. Additionally, water is able to diffuse freely through the porosities left after the evaporation of solvents/unreacted monomers<sup>(22)</sup>.

After 2 months of water storage, the microshear bond strength of the two adhesive systems displayed dramatic drop. This was in agreement with previous work using a variety of storage periods<sup>(6,23,24)</sup>.

Such decline in the bonding efficacy by time might be elucidated by degradation of interface components by water storage. This may be related to the ability of simplified adhesive systems to absorb water, that plays role in hydrolytic degradation of resin-dentin bonds after long-term storage<sup>(7)</sup>. Water can infiltrate and deteriorate the mechanical properties of the polymer matrix by swelling, reducing the frictional forces between the polymer chains, a process is known as "plasticization"<sup>(25,26)</sup>. This passive hydrolysis and leaching effect of break-down products of previous mechanisms is the most important mode of degradation of resin-dentin bond over time<sup>(26)</sup>.

The behaviour of these materials after polymerization was described as permeable membranes<sup>(27)</sup>. Water sorption and dissolution of the incompletely polymerized resin containing amphiphilic monomers may result in deterioration of the one-step self-etch adhesive. Besides, the higher acidity and hydrophilicity of the acidic monomers raise the hazard of hydrolytic degeneration<sup>(28)</sup>.

Moreover, the antagonistic properties of the hydrophilic and hydrophobic constituents, resulting in a hybrid layer with incomplete adhesive infiltration into dentin. Microscopic water filled channels displayed in such hybrid layer permit water movement from underlying dentin to the adhesive-composite zones. Furthermore, the water can diffuse back from the bonded dentin into hydrophilic adhesive resins after drying since hydrophilic resins attract water. Therefore, increasing the hydrophilic resin monomers (HEMA) in adhesives may negatively affect the durability of resin-dentin bonds<sup>(24, 29,30)</sup>.

### CONCLUSION:

Within the limitations of this study, the following can be concluded:

The highest bond strength was presented in samples bonded with Single Bond Universal after 24h water storage. On the other hand, the samples bonded by G-Premio BOND showed higher, but without significant difference microshear bond strength when compared with Single Bond Universal after 2 months' water storage. In all experimental groups, the bond performance deteriorated significantly due to water storage.

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