Effect of contamination and etching on enamel bond strength of new light-cured glass ionomer cements

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Abstract: The effect of water and saliva contamination on the bond strength of metal orthodontic brackets cemented to etched (10% polyacrylic acid) and unetched human premolar enamel was investigated. Two bonding agents were used: one commercially available product (LC) and one experimental (EX) light-cured glass ionomer. Shear bond strength was measured after aging for 5 minutes, 15 minutes, and 24 hours. The results were compared by ANOVA and Scheffe's tests at $p = 0.05$. For LC, the bond strength of brackets bonded to etched enamel, with and without contamination, was statistically higher than that of brackets bonded to unetched enamel for all aging times. An exception was the bond strength to unetched enamel with saliva contamination after 24 hours; for EX, this value was statistically higher than that measured on unetched enamel with water contamination. Contamination by saliva did not reduce bond strength to unetched enamel. For both etched and unetched enamel, there was no significant difference between LC and EX after 24 hours for all contamination conditions.

Key words: Light-cured glass ionomer cements, Shear bond strength, Contamination

Direct bonding of orthodontic brackets has become common for esthetic reasons and for its ease of handling. Most conventional chemical and light-cured orthodontic bonding resins have BIS-GMA as a prime constituent. Use of these direct-bond adhesives requires that the enamel at the bond site be etched with a phosphoric acid solution, and the field be completely dry throughout the procedure. These direct bonding adhesives have been successful in providing clinically acceptable bond strength. However, acid etching has been blamed for decalcification and the development of white spot lesions around bonded orthodontic appliances.[1,2] It is also difficult to completely prevent moisture from contaminating etched enamel.

Glass ionomer cements, which set by an acid-base reaction, were developed in 1972 for use in clinical restorative dentistry. These cements have the unique properties of being able to bond chemically to enamel and dentin, and to release fluoride ions. Research evaluating glass ionomer cements for use in orthodontic treatment has shown that bond strength to unetched and etched enamel is significantly lower than that of composite resin to etched enamel.[3-5] Further, glass ionomer cements exhibit a prolonged setting reaction that results in poor initial bond strength.[6]

Newly developed light-cured glass ionomer cements for orthodontic bonding have produced improved working and setting times. Fricker,[6] in a clinical trial of 10 cases, reported no significant difference in failure rates of direct-bonded orthodontic brackets cemented with light-cured reinforced glass ionomer cement (Fuji II LC, GC Dental Industrials, Tokyo, Japan) and those cemented using composite resin (System 1+, Ormco Corp, Glendora, Calif). Silverman et al.[7] tested an advanced formula-

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Table 1
Materials

Direct bonding adhesives	Code	Manufacturer	Composition	HEMA in liquid (%)	Batch No.
Fuji Ortho LC	LC	GC Dental Industrial Corp, Tokyo, Japan	Powder: Fluoro-aluminosilicate glass Liquid: HEMA (Hydroxyethyl methacrylate) Maleic/acrylic acid copolymer camphoroquinone	30	290651
Experimental glass ionomer bonding adhesive	EX	3M Unitek Dental Products, Monovia, Calif, USA	Powder: Fluoro-aluminosilicate glass Liquid: HEMA, Polyacrylic/ itaconic acid/methacrylate copolymer, camphoroquinone	19	Experimental

tion of a light-activated glass ionomer (Fuji Ortho LC, GC Dental) and concluded that it exhibited all the qualities needed to bond brackets without etching and in the presence of saliva. However, it is not clear whether the bond strength of this light-cured glass ionomer cement is affected by etching or by contamination by saliva or water.

The purpose of this investigation was to study the interactive effects of etching, water and saliva contamination, and aging on the bond strength of one commercially available and one experimental orthodontic light-cured glass ionomer cement.

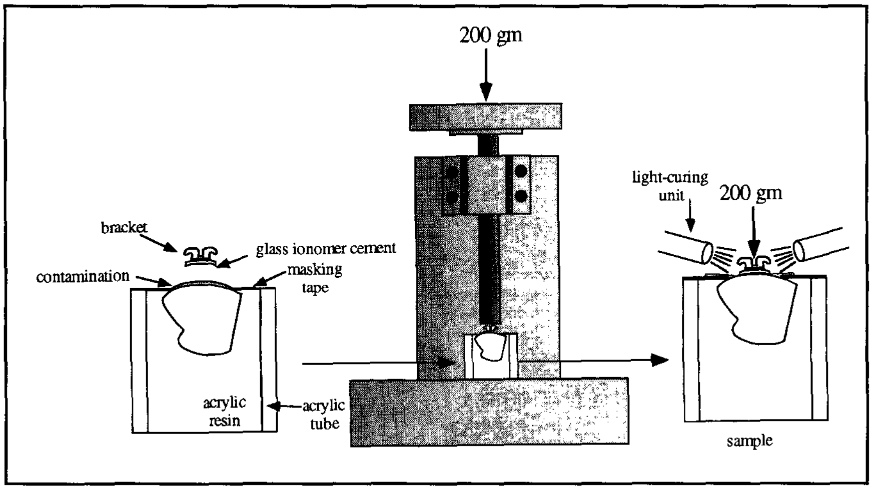

Figure 1
Schematic representation of preparation of specimen for shear bond test

Materials and methods

Bond strength test

Three hundred sixty human premolars, extracted for orthodontic reasons, were used in this research. The roots were removed, leaving the crowns to be embedded in plaster. Two light-cured glass ionomer cements were used in this study, as shown in Table 1. Fuji ORTHO LC light-cured resin-reinforced glass ionomer cement (GC Co, Japan) contains maleic acid/ polyacrylic acid copolymer and hydroxyethyl methacrylate (HEMA); the experimental glass ionomer bonding adhesive (EX: 3M/Unitek Dental Products, Monrovia, Calif) contains polyacrylic acid/methacrylate copolymer and HEMA. Enamel surfaces were polished with polishing cream (Polishing Paste 1, Beebland Medico Dental, Inc, Japan), washed with water for 10 seconds, and dried with oil-free compressed air for 10 seconds. The teeth were divided into two equal groups. Teeth in one group were conditioned (etched) for 20 seconds using 10% polyacrylic acid, washed with water for 10 seconds, and dried with oil-free compressed air for 10 seconds. Teeth in the other group remained unetched. The etched and unetched groups for each cement were further divided into two groups: the teeth in one group were contaminated by exposure to either 20 ml of deionized water or 20 ml of human saliva, and teeth in the other group remained uncontaminated.

Since the exact content of the saliva was not intended to be a variable of the investigation, one person (a healthy 16-year-old female) was selected to provide all the saliva used. The subject chewed paraffin wax and the saliva was collected. It was immediately filtered using 190 mesh nylon filter (Sofar Ine, St. Gallenv, Switzerland) to ensure the absence of particulate matter, then stored in sterile vials. The saliva was used for the experiments immediately after filtration.

The cements were mixed according to the manufacturers' instructions. The mixture was applied to the mesh surface of metal brackets having a base dimension of 3.35 × 3.35 mm (Tomy Int, Japan). The brackets with mixed cement were pressed onto the enamel surface under a load of 200 gm, and excess cement was removed with an explorer before curing (Figure 1). This load was selected because it corresponded to the approximate force measured when performing the operation manually on a testing machine load cell. While under load, the bonding cement was illuminated for 60 seconds on each side of the bracket with a visible light-curing unit (Luxor: Model 4000, ICI, Macclesfield, England). The light intensity was calibrated to 1000 W/m^2. All surface treatment and bonding procedures were carried out in a room with a controlled environment (21°C, 40% relative humidity). After the cement was cured, the specimens were immersed in 37°C water for 5 minutes, 15 minutes, or 24 hours. The shear bond strength experimental design is summarized in Table 2. Shear bond tests were performed on a universal testing machine at a crosshead speed of 1 mm/min (Figure 2). A 0.25 mm- spacer was used to ensure a constant distance between the loading head and the tooth surface. The mean bond strength under each condition was calculated from the results of 10 specimens.

After bond strength testing, the enamel and bracket surfaces were examined under scanning electron microscopy (JSM-330, JEOL Ltd, Tokyo, Japan). All specimens for SEM observation were dried in a desiccator under vacuum for 24 hours. Then the surface was coated with a thin layer of gold-palladium alloy and observed. Fracture patterns were divided into two groups according to location: Type A, cement-enamel interface failure; type B, cement cohesive fracture and cement-enamel interface failure; and type C, bracket-cement interface failure.

Table 2
Shear bond strength experimental design summary

Material (2):
  LC and EX
Surface pretreatment (2):
  non-etched, etched
Surface contamination (3):
  dry, water, saliva
Aging time (3):
  5 min, 15 min, 24 hr

Total number of experimental combinations = 2 x 2 x 3 x 3 = 36. Each experimental combination was replicated 10 times for a total of 360 teeth.

Flexural strength test

The flexural strength of the cement was tested after each of the aging times in order to ascertain whether changes in flexural strength play a role in shear bond strength.

Each mixed cement was inserted into a silicone mold with the ends covered by a glass slide. Five rectangular specimens (2 × 2 × 30 mm) of each cement were illuminated for 60 sec from the superior surface and for an additional 60 sec from the inferior surface in a light-curing unit. The specimens were stored in 37°C water until the tests were performed. The flexural strength was measured 5 minutes, 15 minutes, or 24 hours after illumination began.

The data obtained in this study were analyzed statistically using two-way and three-way analyses of variance (ANOVA) performed across the groups in each test, and F-values were obtained. If the F-value corresponded to $p<0.05$, the groups in each test were subjected to Scheffe's multiple comparison test for the detection of statistical differences.

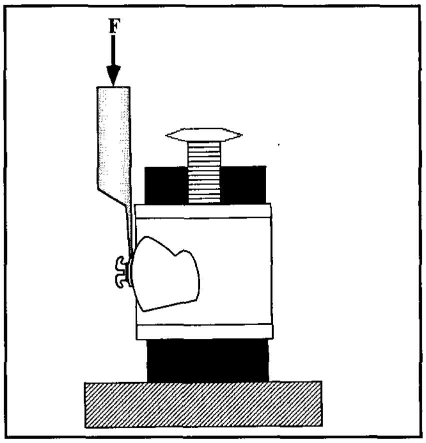

Figure 2
Schematic representation of shear bond test apparatus

Results

The shear bond strength of specimens bonded to unetched and etched enamel with and without contamination are summarized in Tables 3 and 4. The three-way ANOVA showed significant differences in bond strength among contaminant conditions (dry, water, and saliva), among aging times, and between etched and unetched enamel for LC and EX ($p<0.05$). No three-way interactions were found among type of contaminant, aging time, and etching for either cement ($p>0.05$). For LC, bond strengths to etched enamel with and without contamination were statistically higher ($p<0.05$) than those to unetched enamel for all aging times, except for unetched enamel with saliva contamination after 24 hours. For EX, the bond strengths to etched enamel with water contamination were statistically higher ($p<0.05$) than those to unetched enamel with water contamination, regardless of aging time.

The interactions between aging time and type of contamination for LC on unetched enamel are shown in Figure 3, and for EX in Figure 4. The effects of aging and contamination were similar for the two cements. A significant ($p<0.05$) reduction in bond strength is seen with water contamination after

Table 3
Bond strength of LC (MPa ± standard deviation)

	5 minutes		15 minutes		24 hours	
	Unetched	Etched	Unetched	Etched	Unetched	Etched
Dry	7.0±2.0	11.6±2.0	7.2±1.1	11.5±1.4	8.4±1.7	12.4±2.8
Water	4.5±1.0	8.2±1.7	5.2±0.7	10.7±3.4	6.9±1.4	12.6±2.2
Saliva	6.0±1.3	7.6±1.5	6.3±0.8	7.9±2.3	7.2±1.7	8.2±1.8

Bond strengths connected by vertical and horizontal bars and brackets were not significantly different ($p>0.05$)

Table 4
Bond strength of EX (MPa ± standard deviation)

	5 minutes		15 minutes		24 hours	
	Unetched	Etched	Unetched	Etched	Unetched	Etched
Dry	7.1±1.6	8.3±1.0	7.1±1.3	8.5±2.1	8.9±2.3	10.4±1.6
Water	3.6±0.9	5.5±0.4	4.7±1.0	6.7±1.2	7.8±1.8	10.4±3.2
Saliva	6.5±1.8	6.0±1.0	6.6±2.0	6.8±1.1	7.9±2.7	9.2±1.6

Bond strengths connected by vertical and horizontal bars and brackets were not significantly different ($p>0.05$)

short aging times. No bond strength difference remained after 24 hours. There was no significant aging effect observed in the saliva-contaminated groups. After 24 hours, there were no significant bond strength differences among the contaminated groups compared with the dry groups for either cement. Further, neither cement provided greater bond strength.

The interactions between aging time and type of contamination for LC on etched enamel are shown in Figure 5. While no significant aging effect was seen for the dry and saliva-contaminated conditions, a significant increase in bond strength with aging time was noted for the water-contamination group. However, after 24 hours, the values of the dry and water-contaminated groups were the same ($p<0.05$). The 24-hour bond strength for the saliva-contaminated group was significantly lower.

The interactions between aging time and type of contamination for EX on etched enamel are shown in Figure 6. This cement showed a more pronounced aging effect on dry enamel than observed for LC. The bond strength values for 24 hours aging were similar, but were significantly ($p<0.05$) lower than the small aging time value. A similar trend was seen for water- and saliva-contaminated enamel, with the 5-minute and 15-minute values significantly ($p<0.05$) lower than the corresponding dry enamel values. However, the values for the dry and water- and saliva-contaminated conditions were not significantly different after 24 hours. Furthermore, after 24 hours, there were no statistically significant differences in bond strengths between EX and LC for each of the enamel conditions.

SEM micrographs of typical fracture patterns for both cements after bonding tests are shown in Figure 7. The area covered with residual cement after the bond tests are shown in Tables 5 and 6. In the unetched group, samples without and with contamination after 24 hours aging showed mainly mixed cohesive and cement-enamel interface failures (type B). While failures of samples that remained dry showed mixed cohesive and cement-enamel interface failures, those with water-contamination after 5 and 15 minutes aging showed cement-enamel interface failures (type A). For etched enamel, LC specimens, regardless of contamination or aging time, showed mainly type B failures. Failures of EX samples with and without contamination after 24 hours aging, and those that remained dry after 5 and 15 minutes aging, showed type B fractures. However, EX samples with water- and saliva-contamination after 5 and 15 minutes aging showed type A fractures. No type C (bracket-cement interface) failures were observed.

The flexural strengths of both cements are shown in Figure 8. For LC, flexural strengths after 15 minutes and 24 hours were statistically higher ($p<0.05$) than after 5 minutes. For EX, flexural strength after 24 hours was statistically highest ($p<0.05$). For LC, flexural strength at each immersion time was significantly higher than the value for EX at the corresponding conditions.

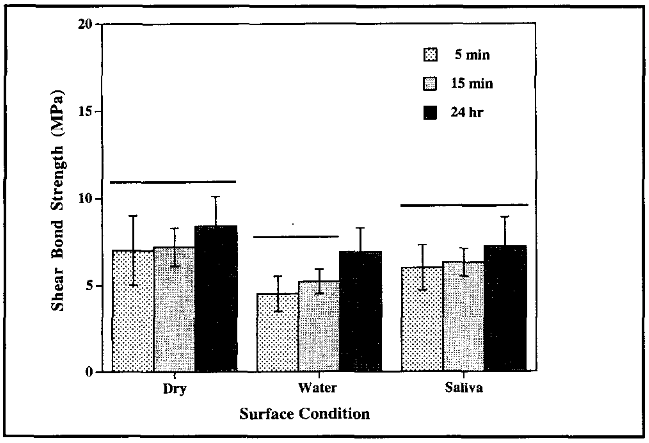

Figure 3
Effect of aging time on shear bond strength of LC to unetched enamel. Groups connected by horizontal line are not different (p<0.05).

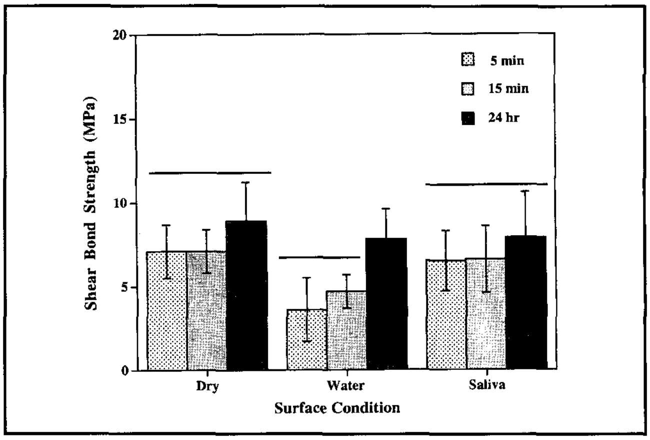

Figure 4
Effect of aging time on shear bond strength of EX to unetched enamel. Groups connected by horizontal line are not different (p<0.05).

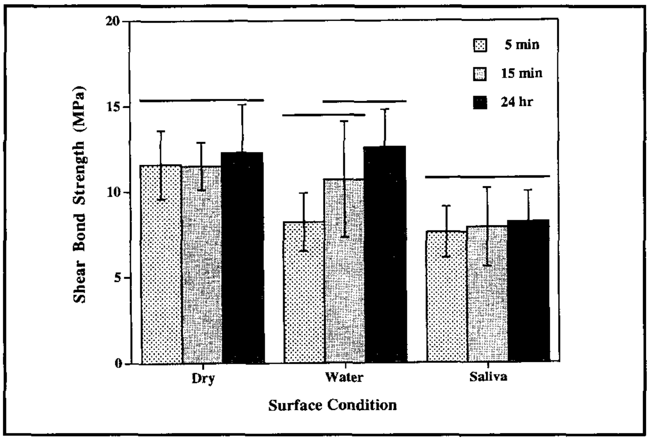

Figure 5
Effect of aging time on shear bond strength of LC to etched enamel. Groups connected by horizontal line are not different (p<0.05).

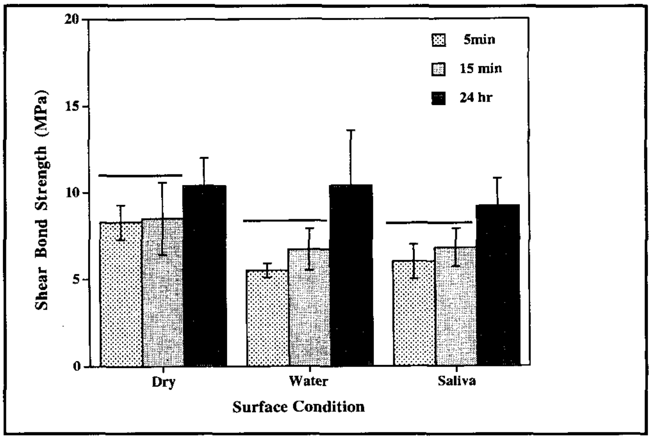

Figure 6
Effect of aging time on shear bond strength of EX to etched enamel. Groups connected by horizontal line are not different (p<0.05).

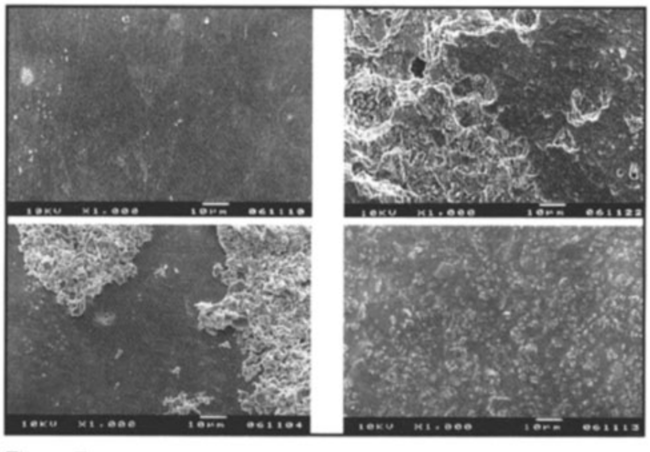

Figure 7
Fracture patterns after shear bond strength tests. Above, type A mode: left, enamel surface; right, bracket surface. Below, type B mode: left, enamel surface; right, bracket surface.

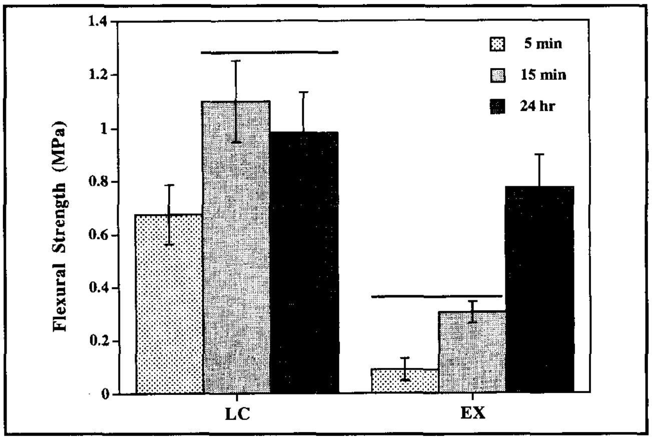

Figure 8
Flexural strength of LC and EX. Groups connected by horizontal line are not different (p<0.05).

Discussion

A major difference in the composition of the two light-cured glass ionomer cements evaluated is in the concentration of HEMA (Table 1). The Fuji ORTHO LC contains nearly twice the amount of HEMA as the experimental material, which may explain some of the observed bond strength differences. For example, LC demonstrated greater bond strength to etched enamel than to unetched enamel. Exceptions were for specimens contaminated with saliva and aged for 24 hours, and for EX after short aging times. These observations are consistent with the SEM micrographs, which revealed that after bond strength testing, more cement remained on etched enamel than on unetched. Because of the higher HEMA concentration, resinous tags of polyHEMA are more readily formed in etched enamel by photoinitiated free radical polymerization. The nature of the mechanical properties of the resinous tags is another critical factor affecting the strength of the bond to etched enamel. Flexural testing revealed that LC was stronger than EX at each aging time, which led to increased bond strength. Since the HEMA concentration of EX is lower than that of LC, the polymerization setting reaction by visible light is delayed. This delay may explain the lower bond strength of EX compared with LC after 5 and 15 minutes aging.

Water contamination had a deleterious effect on the bond strength of both cements after short aging times. Mojon et al.[8] reported that water contamination reduces the mechanical properties of glass ionomer cement. Higashino[9] described how bond strength can be reduced by dissolving the surface layer of cements close to enamel in water. These observations explain the weaker bond strength and the enamel-cement interface failure observed in the sample with early water contamination.

Table 5
Tooth bonding area (%) covered with residual glass ionomer cement (LC)

	5 minutes		15 minutes		24 hours	
	Unetched	Etched	Unetched	Etched	Unetched	Etched
Dry	20	50	20	50	20	50
Water	0	20	0	50	20	50
Saliva	0	20	0	20	20	20

Table 6
Tooth bonding area (%) covered with residual glass ionomer cement (EX)

	5 minutes		15 minutes		24 hours	
	Unetched	Etched	Unetched	Etched	Unetched	Etched
Dry	20	20	20	20	20	50
Water	0	0	0	0	20	50
Saliva	0	0	0	0	20	20

It was expected that the detrimental effect of saliva contamination on bonding would be more of a problem than water contamination. This expectation is based on the postulation that the setting of the cements would be compromised by the presence of proteins and minerals in the saliva that are not soluble in HEMA. Perhaps more significantly, the higher viscosity of saliva may occlude the microscopic roughness produced by etching, as well as lower the surface energy, thereby inhibiting proper resinous tag formation. However, for unetched enamel, saliva was less disturbing than water to both cements. Mojon et al.[8] noted that, although water in saliva disturbs the cement during the early stages, some components of saliva protect the cement or counteract the effects of water. This finding helps to explain why the bond strength to unetched enamel with saliva contamination in early stages was higher than that to unetched enamel with water contamination. It should be noted that the bond strength differences between unetched and etched enamel were observed because the amount of residual saliva may be higher on etched enamel than on unetched enamel.

These results indicate that the light-cured glass ionomer cements used in this study (LC, EX) are capable of offering clinically acceptable bond strength to unetched and etched enamel. Etched enamel surfaces produced higher bond strengths. Excessive residual water, especially on unetched enamel, reduces the initial bond strength. Therefore, better results could be obtained when the teeth are lightly dried with a cotton roll and an oil-free air syringe. When etching is considered necessary, saliva contamination should be avoided because of its negative effects on bond strength. This study also indicates that acid etching is recommended in cases where heavy forces are to be applied to the tooth or teeth, especially when such application is to occur immediately after bonding.

Conclusions

The effect of water and saliva contamination on the shear bond strength of metal brackets with mesh base bonded to unetched or etched human enamel using commercially available and experimental orthodontic light-cured glass ionomer cements was investigated.

The results of this study indicate the following:

1. For LC, bond strength to etched enamel, with or without contamination, was statistically higher than to unetched enamel for all aging times except for unetched enamel with saliva contamination after aging 24 hours.
2. For EX, the bond strength of brackets bonded to etched enamel contaminated with water were statistically higher than that of brackets bonded to unetched enamel and contaminated with water, regardless of aging time.
3. Water contamination had a deleterious effect on the bond strength of both cements to etched and unetched enamel for short aging times.
4. For unetched enamel, saliva was less deleterious to bond strength than water for both cements.
5. For etched and unetched enamel, there was no significant difference in bond strength between LC and EX after 24 hours for all surface conditions.

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