# The effect of moisture on the shear bond strength of gold alloy rods bonded to enamel with a self-adhesive and a hydrophobic resin cement

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SUMMARY The aim of this *in vitro* study was to investigate the influence of enamel moisture on the shear bond strength (SBS) of a hydrophobic resin cement, Maximum Cure® (MC), and a self-adhesive resin cement, Multilink Sprint® (MLS), after etching of the enamel. Forty cylindrical gold alloy rods were used to simulate the Incognito® lingual bracket system. They were bonded to the enamel of 40 human teeth embedded in self-cured acrylic resin. Twenty were bonded with MC (10 on dry and 10 on wet enamel) and 20 with MLS (10 on dry and 10 on wet enamel). The SBS of MC and MLS was determined in a universal testing machine and the site of bond failure was defined by the adhesive remnant index (ARI). A Kruskal-Wallis test was performed followed by Games–Howell *post hoc* pairwise comparison tests on the SBS results (P < 0.05) and a chi-square test was used for the analysis of ARI scores (P < 0.05).

On dry enamel, no significant differences between MC ( $58 \pm 5$  MPa) and MLS ( $64 \pm 13$  MPa) were noted. On wet enamel, the adherence of MC ( $6 \pm 8$  MPa) and MLS ( $37 \pm 13$  MPa) significantly decreased but to a lesser extent for MLS. The ARI scores corroborated these results.

In conclusion, MC did not tolerate moisture. MLS was also affected but maintained sufficient adherence.

## Introduction

Bonding of orthodontic brackets is a technique-sensitive procedure and moisture is cited as the most common cause for bond failure (Zachrisson, 1977; Hormati et al., 1980; Xie et al., 1993; Bishara et al., 1998; Grandhi et al. 2001; Hobson et al., 2001). The manufacturers of one of the most used lingual bracket systems, Incognito®, recommend bonding the brackets with a hydrophobic system, Maximum Cure® (MC), (Wiechmann, 2002). For clinical success, this material requires dry and isolated fields and enamel conditioning (Hormati et al., 1980; Grandhi et al., 2001; Rajagopal et al., 2004). The practitioner must use spacers, salivary cotton rolls, and aspiration to decrease the humidity of the bonding environment and the risk of salivary contamination (Cacciafesta et al., 1998, 2003). However, enamel contamination is difficult to control, especially in hard-to-reach areas such as the lingual surfaces of molars (Bishara et al., 1998). Thus, it would be advantageous to be able to bond to enamel in a wet environment with less moisture-sensitive materials. Because of their composition, self-adhesive resin cements are potentially hydrophilic systems.

In a previous study within the field of restorative dentistry, it has been shown, under dry conditions, that acid etching of enamel with phosphoric acid prior to application of the self-adhesive resin cement significantly increased bond strength (De Munck *et al.*, 2004). No reported study has compared the shear bond strengths (SBS) of these materials under both dry and wet conditions.

The purpose of this research was to investigate the influence of enamel moisture on the adherence of a hydrophobic adhesive, MC commonly used in lingual orthodontics and Multilink Sprint® (MLS). It was hypothesized that the adherence to wet enamel of the self-adhesive resin cement would be reduced as compared with that to dry enamel but would be less affected than hydrophobic resin cement.

## Materials and methods

Forty cylindrical gold alloy rods, 5 mm in length and with a plane base of 3 mm in diameter, were used to simulate Incognito® brackets. They were cast and covered, similar to Incognito® brackets, with the resin composite Phase II®, 'Reliance' after surface treatment of their base with Rocatec® (3M Espe, St Paul, Minnesota, USA). They were prepared in the Incognito® laboratory (3M Unitek, Bad Essen, Germany) and used within 15 days of fabrication. The composite was cleaned before bonding with an acetone-soaked cotton pledget. Forty freshly extracted human maxillary central incisors were collected, cleaned of soft tissue, and stored at 4°C in a solution of 1 per cent chloramine and used within 3 months. The criteria for tooth selection included intact buccal enamel, no pre-treatment with chemical agents, no cracks caused by extraction forceps, and no decay. The teeth had been extracted for reasons unrelated to the objectives of this study and with the patients' informed consent. The project was approved by the scientific council of the Faculty of Dental Surgery, University of Paris-Descartes. These selected teeth had the greater portion of the roots removed using sandpaper (80 grit). The crowns were then roughened on their buccal surface with water-cooled sandpaper (800 grit) to expose the enamel in order to obtain a plane enamel surface (greater than 7 mm<sup>2</sup>) on which the gold alloy rod could be bonded. Finally, the residual crowns were embedded in self-cured acrylic resin (Plexcil-Escil, Chassieu, France) in plastic cylinders with the flat enamel surface exposed. The flat surfaces were inspected under ×40 magnification to ensure that the enamel was intact and free of debris. The enamel surfaces were sandblasted for 5 seconds each from a distance of 1 cm, with 50 µm aluminum oxide powder (Al<sub>2</sub>O<sub>3</sub>), according to the manufacturer's protocol (Wiechmann, 2000; D'Arcangelo and Vanini, 2007), then rinsed and dried. The samples were randomly assigned to four groups, each consisting of 10 specimens.

Two adhesive systems were evaluated in the current study: the chemical compositions of which are detailed in Table 1.

MC: (control group) the hydrophobic resin cement recommended by the manufacturer;

MLS: a dual-curing, self-adhesive resin cement. The chemical compositions of the two cements are detailed in Table 1.

For each adhesive, two enamel surface conditions were studied: dry and wet. The various groups tested were group 1 for MC on dry enamel, group 2 for MC on wet enamel, group 3 for MLS on dry enamel, and group 4 for MLS on wet enamel. The teeth in groups 1 and 3 were conditioned with 37 per cent phosphoric acid (Scotchbond Etching, 3M Espe) for 30 seconds, followed by thorough washing for 10 seconds, and gentle drying with compressed air until a chalky white enamel appearance was obtained. The teeth in groups 2 and 4 were conditioned with 37 per cent phosphoric acid (Scotchbond Etching) for 30 seconds, followed by thorough washing for 10 seconds, and drying until a slightly shining, wet-appearing enamel surface was obtained. In groups 1 and 2, the two components of MC (part A and part B) were squeezed together and a thin coat of the resulting mix was applied on the tooth surface and on the rod. The rod was positioned on the enamel surface with sufficient pressure to express excess adhesive, which was removed from around the rod base with a cotton pledget. In groups 3 and 4, MLS was applied with an automix syringe on the tooth surface and on the rod. The rod was positioned on the enamel surface with sufficient pressure to express excess adhesive, which was removed from around the rod base with a probe and the material was light cured. The light source was a Demetron LC curing light (Kerr Corporation, Orange, California, USA) activated for 10 seconds of exposure on four areas around the sample to ensure sufficient irradiation of the cement with a total of 40 seconds exposure time. After bonding, all samples were set under a weight in a device that allowed the stabilization of the bonded rod during 5 minutes. The specimens were then stored in distilled water at 37°C for 24 hours and subsequently tested in shear mode.

The SBS was determined in a universal testing machine (LRX, Lloyd Instruments, Fareham, Hants, UK). The sample was immobilized in the device that has a sliding blade acting like a guillotine, giving a shearing fracture at the enamel-rod junction. A crosshead speed of 0.5 mm/minutes was chosen. The debonded specimens were observed using a binocular microscope (Olympus Europe SZH10, Hamburg, Germany) under ×40 magnification, and scoring was undertaken according to the adhesive remnant index (ARI; Årtun and Bergland, 1984). The ARI scores were used to define the site of bond failure between the enamel, the adhesive, and the rod base. ARI scores range from 0 to 3, that is score 0 = noadhesive remained on the tooth surface, score 1 = less than half of the adhesive remained on the tooth surface, score 2 =more than half of the adhesive remained on the tooth, and score 3 = all the adhesive remained on the tooth with a distinct impression of the rod base.

## Statistical analysis

Each series of tests was carried out on 10 samples. The means and standard deviations for SBS were calculated. A Kruskal–Wallis test was performed followed by Games–Howell *post hoc* pairwise comparison tests on the SBS results. A chi-square test was used to determine significant differences in the ARI scores between the groups. Significance for all these tests was predetermined at P < 0.05. Statistical calculations were performed using the StatView® software for Windows Version 5.0 (SAS® Institute Inc, Cary, North Carolina, USA).

 Table 1
 Materials, manufacturers, batch number, and chemical composition.

Material	Manufacturer	Batch number	Composition (manufacturers' data)
Maximum Cure® (MC)	Reliance Orthodontic Products, Itasca, Illinois,	Part A 0610714; part B 0610713	MC part A: bisGMA, MMA, morpholinoethylmethacrylatehydrofluoride, amine; MC part B: bisGMA, MMA, benzoylperoxide
Multilink Sprint® (MLS)	Ivoclar Vivadent, Schaan, Liechtenstein	J22739	Base and catalyst: paste of dimethacrylates (24–26%), methacrylated phosphoric acid ester (<5%), inorganic fillers, ytterbiumtrifluoride, benzoylperoxide(<1%), stabilizers, pigments

## Results

#### Shear bond strength

Table 2 presents the values of adherence obtained for the MC and MLS adhesive systems on dry and wet enamel. On dry enamel, there was no significant difference between MC ( $58 \pm 5$  MPa) and MLS ( $64 \pm 13$  MPa). On wet enamel, the adherence of MC ( $6 \pm 8$  MPa) and MLS ( $37 \pm 13$  MPa) decreased significantly, 90 (P < 0.001) and 41 (P < 0.05), per cent respectively.

#### Adhesive remnant index

The ARI scores for the four groups are listed in Table 3. The chi-square test indicated significant differences among the various groups. For groups bonded with MC, a lower frequency of failure at the enamel–adhesive interface was observed under dry than under wet conditions. No significant differences in debond location were found among the groups bonded with MLS under dry or wet enamel conditions. No significant differences in debond location were found between the two groups bonded with MLS and the group with MC under dry condition.

## Discussion

The introduction of bonding materials less sensitive to moisture would be a welcome improvement because clinical

 Table 2
 Means and standard deviations for shear bond strength (SBS) on dry and wet enamel of rods bonded with Maximum Cure® (MC) and Multilink Sprint® (MLS).

	SBS (MPa)				
	Dry enamel	Wet enamel			
MC MLS	$\begin{array}{l} 58\pm5^a\\ 64\pm13^a\end{array}$	$\begin{array}{c} 6\pm8^{c}\\ 37\pm13^{b} \end{array}$			

Values with the same superscript letter are not significantly different at P < 0.05.

 Table 3
 Adhesive remnant index (ARI) scores for Maximum Cure® (MC) and Multilink Sprint® (MLS).

Group			ARI				
				0	1	2	3
1	МС	Dry	10 <sup>a</sup>	4	2	2	2
2	MC	Wet	10 <sup>b</sup>	10	0	0	0
3	MLS	Dry	10 <sup>a</sup>	5	3	2	0
4	MLS	Wet	10 <sup>a</sup>	6	2	2	0

Values with the same superscript letter indicates the classes to which each group was significantly associated (P < 0.05)

conditions do not permit ideal isolation, in particular for lingual orthodontics.

This study intended to compare the bond strengths of a conventional hydrophobic resin cement and a self-adhesive resin cement after etching of the enamel under both dry and wet conditions. For both investigated systems, the adherence was reduced on wet enamel as compared with that on dry enamel but to a lesser extent for MLS. Thus, the hypothesis was confirmed.

On dry enamel, the MC adherence was high (58 MPa). This result is in agreement with the adherence value obtained in a previous study in restorative dentistry (De Munck *et al.*, 2004). The mechanism that explains this good bonding is well known. Etching of the enamel surface creates a superficial etching zone with an underlying porous zone. The inflow of the bonding agent into the porous zone results in the formation of resin tags, and micromechanical retention to etched enamel is established (Buonocore, 1955; Retief, 1978; Hitmi, 2004).

On wet enamel, a decrease of 90 per cent of MC adherence to dry enamel was observed. This reduction in bonding is also well known. It has been reported that the bond strength of resin composites to etched enamel is adversely affected by water contamination (Hormati et al., 1980). Water contamination will prevent the bonding agent from sufficiently contacting the etched enamel surface, resulting in reduced bonding. Furthermore, hydrophobic monomers are unable to drive out water occupying the zones of demineralization of enamel and unable to infiltrate the surface zone of etched enamel (Hormati et al., 1980). The weak diffusion of the monomer into the threedimensional network of etched enamel results in a weak adhesion. This phenomenon was highlighted by Hitmi (2004) who observed, in an scanning electron microscopy study, infiltration defects of the hydrophobic resin, Concise<sup>®</sup>, into the stained thickness of enamel as well as the presence of a hiatus at the interface.

The range of ARI scores observed in the present study demonstrated that MC, used in a dry field, showed a significantly lower frequency of failure at the enamel– adhesive interface than on wet enamel. This finding corroborates the preceding explanations and is in agreement with the results of previous studies (Webster *et al.*, 2001; Cacciafesta *et al.*, 2003). On dry enamel, MLS adherence presented no significant difference as compared with MC. On wet enamel, the adherence values obtained with MLS decreased from 64 MPa to 37 MPa, that is relatively less than found for MC.

The results of the present study cannot be compared with those in the literature because self-adhesive resin cements have only recently been introduced (Hikita *et al.*, 2007; De Munck *et al.*, 2004) and no study has reported the influence of moisture on their adherence. The presence of hydrophilic groups (phosphate groups) in these self-adhesive resins may explain the moderate sensitivity to moisture. Thus, the use of MLS in a wet environment is a possible option because its adherence value of 37 MPa may be regarded as sufficient.

No significant difference in debond locations (ARI values) was found among the groups bonded with the selfadhesive cement under dry or wet enamel conditions and with MC on dry enamel. This confirms the moderate influence of moisture on MLS performance.

#### Limitations of the study

Investigation of hydrophilic adhesives is difficult (Klocke *et al.*, 2003) because the effectiveness of the materials may vary with the degree of moisture contamination (Grandhi *et al.*, 2001). There seems to be a limit as to how much of a wet field is acceptable, after which excessive surface moisture can result in a decrease in bond strength (Tay *et al.*, 1996). Furthermore, the present study does not truly reflect the oral environment because only the influence of water was tested. In the oral environment, saliva is more complex than water (Littlewood *et al.*, 2000). Many of its constituents may have additional effects on the resulting bond strength.

## Conclusions

The findings of the present study have lead to the following conclusions:

- 1. MC is unable to tolerate moisture, which confirms the need to find alternative adhesives less sensitive to a moist environment.
- 2. MLS is also affected by moisture but maintains sufficient adherence in a moist environment; this should be clinically useful.

Clinical investigations are necessary to confirm the results obtained in this *in vitro* study.

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