Microleakage beneath brackets bonded with flowable materials: effect of thermocycling

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SUMMARY The purpose of this study was to evaluate the effects of thermocycling on microleakage beneath brackets bonded with an orthodontic composite and different flowable materials. Brackets were bonded to 200 bovine incisors divided into five groups: (1) Transbond XT, (2) X-Flow, (3) Dyract-Flow, (4) Admira-Flow, and (5) Beautiful-Flow. Half the teeth in each group were thermocycled. The specimens were dyed with 1 per cent methylene blue for 24 hours to determine the percentage of microleakage into the enamel–adhesive and adhesive–bracket interfaces using image analysis equipment. Data were analysed using the Kruskal–Wallis and Mann–Whitney *U*-tests (P < 0.05), applying Bonferroni correction when required (P < 0.005).

Without thermocycling, microleakage at the enamel-adhesive interface was significantly greater for Admira-Flow than for X-Flow (P < 0.005). At the adhesive-bracket interface, there were no significant differences (P > 0.005). After thermocycling, microleakage of Beautiful-Flow at the enamel-adhesive interface was significantly less than for the other materials tested (P < 0.005), while at the adhesive-bracket interface, Admira-Flow and X-Flow showed significantly more microleakage than Beautiful-Flow and Transbond XT (P < 0.005). Analysis of the effect of thermocycling on each material showed that microleakage increased significantly at the enamel-adhesive interface with Transbond XT (P < 0.05), decreased with Beautiful-Flow (P < 0.05), increased significantly at both interfaces with X-Flow, but not to a statistically significant level with Dyract-Flow and Admira-Flow (P > 0.05). The giomer, Beautiful-Flow, demonstrated the best performance after thermocycling, while composite resins and, in particular, the flowables showed a poorer performance.

Introduction

Microleakage can be defined as the filtration of bacteria, fluids, molecules, ions, and even air between the walls of a cavity, previously prepared in readiness for restoration and the restoration material (Kidd, 1976). In conservative dentistry, the majority of restorative materials show variable levels of marginal microleakage due to changes in dimension and a lack of good adaptation to cavity walls (Mali *et al.*, 2006). Among the clinical consequences of marginal microleakage are secondary carious lesions, pulpal pathology, post-operative pain and sensitivity, and, consequently, potential failure of the restoration (Youssef *et al.*, 2006). In orthodontics, microleakage may play a part in the formation of decalcification lesions at and under the adhesive–enamel interface.

Studies of orthodontic bracket (James *et al.*, 2003; Arhun *et al.*, 2006; Arikan *et al.*, 2006) and band (Gillgrass *et al.*, 1999) microleakage are few but in all of them some degree of microleakage has been observed. Therefore, although the area around a bracket is critical to the development of decalcification, the area beneath the bracket also requires investigation (Arhun *et al.*, 2006).

Because bond materials are routinely subjected to changes of temperature in the oral cavity, it is important to determine if these temperature variations cause stress to the adhesive which might affect levels of microleakage (Bishara *et al.*, 2003). For this reason, thermocycling is a method widely used for the assessment of bond materials. It aims at thermally stressing the adhesive joint at the tooth restoration interface by subjecting the restored teeth to extreme temperatures compatible with those encountered intraorally. This process may highlight the mismatch in thermal expansion between the adhesive material and tooth structure, resulting in different volumetric changes during temperature fluctuation and causing fatigue of the adhesive joint with subsequent microleakage (Wahab *et al.*, 2003). Several studies have shown that thermocycling significantly reduces bond strength (Arici and Arici 2003; Bishara *et al.*, 2003; Daub *et al.*, 2006) and increases microleakage beneath the bond materials (Hakimeh *et al.*, 2000; Wahab *et al.*, 2003; Helvatjoglu-Antoniades *et al.*, 2004).

Composite resins are materials which have traditionally been used both in conservative dentistry and for bracket bonding. Composite resins are made up of two main components: an organic resin matrix and inorganic mineral filling. Flowable composites maintain the same particle size as traditional composites but reduce the proportion of filling to allow for a higher proportion of resin, in this way reducing the viscosity of the mixture (Tecco *et al.*, 2005). The filling content affects polymerization shrinkage (James *et al.*, 2003), which in turn increases the chance of marginal leakage. Various studies have evaluated the use of flowable composites for bracket bonding (Uysal *et al.*, 2004; Tecco *et al.*, 2005). Several other new flowable materials have also been introduced onto the market for orthodontic use, such as ormocers (Vicente and Bravo, 2007), compomers (Bishara *et al.*, 2001; Tecco *et al.*, 2005; Vicente *et al.*, 2006), and giomers. Although some investigations of microleakage in conservative dentistry have been carried out for these materials (Ferdianakis, 1998; Payne, 1999; Estafan and Stafan, 2000; Yazici *et al.*, 2003a,b, 2004; Pardi *et al.*, 2006), no study appears to have been undertaken into microleakage when these materials are used for bracket bonding.

The aim of this research was therefore to evaluate the effect of thermocycling on microleakage under brackets bonded with an orthodontic composite resin (Transbond XT), a flowable composite (X-Flow), a flowable compomer (Dryact-Flow), a flowable ormocer (Admira-Flow), and a flowable giomer (Beautiful-Flow).

Materials and methods

Teeth

Two hundred bovine incisor teeth were placed in 0.1 per cent thymol solution for 1 week to prevent bacteria growth (the solution was changed daily). They were then stored in distilled water, which was changed daily. In no case was one tooth stored for more than 1 month after extraction.

Brackets

Two hundred upper central incisor brackets were used (Victory Series, 3M Unitek Dental Products, Monrovia, California, USA). The base area of the bracket (10.25 mm²) was calculated using image analysis equipment and MIP 4 software (Microm Image Processing Software, Digital Image Systems, Barcelona, Spain).

Bonding procedure

The teeth were divided into five equal groups and the brackets bonded on the labial surfaces according to the manufacturers' instructions. The chemical compositions of the products are shown in Table 1. For all groups, the labial surfaces were polished with a rubber cup and polishing paste (Détartrine, Septodont, Saint-Maur, France) and etched with 37 per cent *o*-phosphoric acid gel (Total Etch, Ivoclar Vivadent, Schaan, Liechtenstein) for 30 seconds. The enamel was then washed with water and dried with compressed air.

Group I (n = 40): Transbond XT (3M Unitek Dental Products). A layer of Transbond XT primer was applied to the tooth and Transbond XT paste to the base of the bracket and pressed firmly onto the tooth. Excess adhesive was removed from around the base of the bracket with a probe

Fable 1	Composition	and lot numbers	of the tested	adhesives.
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Adhesive	Composition	Lot number		
Transbond XT	Silane-treated quartz Bisphenol A diglycidyl ether dimethacrylate Bisphenol A bis(2, hudroxyathyl	6XA/6EB		
	ether)dimethacrylate Dichlorodimethylsilane reaction product with silica	0<10001002		
Dyract-Flow	Strontium-alumino-fluoro-silicate glass	0610001903		
	Highly dispersed silicon dioxide Dipentaerythritol penta acrylate monophosphate			
	N,N-dimethyl aminoethyl methacrylate Carboxylic acid modified			
	methacrylate macromonomers Diethylene glycol dimethacrylate Camphorquinone			
	Ethyl-4-dimethylaminobenzoate 2-Hydroxymethoxybenzophenone Butylated hydroxy toluene (BHT)			
	and other stabilizers Iron pigments Titanium dioxide			
Beautiful-Flow	Bis-GMA/TEGDMA resin Multifunctional glass fillers, S-PRG fillers based on fluoro-	21600BZZ00147		
X-Flow	boroaluminosilicate glass Strontium-alumino-sodium- fluoro-phosphor-silicate glass	0610000912		
	and methacrylate resins Diethylene glycol dimethacrylate Highly dispersed silicon dioxide			
	UV stabilizer Ethyl-4-dimethylaminobenzoate Camphorquinone			
	BHT Iron pigments Titanium dioxide			
Admira Flow	Barium-aluminium-boro-silicate glass, silicone dioxide ormocers, Bis-GMA, urethane-dimethacrylate triethylene-dimethacrylate	441124		

and the adhesive was light cured positioning the light guide of an Ortholux XT lamp (3M Unitek Dental Products) on each interproximal side for 10 seconds.

Group II (n = 40): Dyract-Flow (Dentsply DeTrey GmbH, Konstanz, Germany). A layer of Prime and Bond NT (Dentsply DeTrey) was applied to the enamel leaving it undisturbed for 30 seconds. A moisture-free air source was then used to deliver a gentle burst of air to the enamel and the adhesive, Prime and Bond NT, was light cured for 20 seconds. Afterwards, Dyract-Flow was applied to the base of the bracket, the bracket was placed on the tooth, and light cured on each interproximal side for 20 seconds.

Group III (n = 40): Beautiful-Flow F10 (Shofu Inc., Kyoto, Japan). Fl-Bond primer (primer A + B) (Shofu Inc.)

was applied to the enamel and left for 10 seconds. It was then dried with compressed air. Afterwards, the bonding agent was applied and light cured for 10 seconds. Beautiful-Flow was applied to the base of the bracket and pressed firmly onto the tooth. Excess adhesive was removed from around the base of the bracket with a probe and the adhesive was light cured positioning the light guide of an Ortholux XT lamp on each interproximal side for 20 seconds.

Group IV (n = 40): X-Flow (Dentsply DeTrey GmbH). Prime and Bond NT was applied as in group II. X-Flow was applied to the base of the bracket and then the bracket was placed on the tooth and light cured on each interproximal side for 20 seconds.

Group V (n = 40): Admira-Flow (Voco, Cuxhaven, Germany). Admira bond was applied to the tooth surface and left for 30 seconds. It was then lightly dispersed with an air jet and polymerized for 20 seconds. Admira-Flow was then applied to the bracket base and the bracket placed on the tooth and light cured with an Ortholux XT lamp on each interproximal side for 20 seconds.

Storage of test specimens

The 40 specimens in each group were stored for 24 hours in distilled water at 37°C [International Organization for Standardization (ISO), 1994].

Thermocycling test

Twenty specimens from each group were subjected to thermocycling following the ISO 11405 recommendations (ISO, 1994). The thermocycling test comprised 500 cycles in distilled water between 5 and 55°C. The exposure to each bath lasted 20 seconds, and the transfer time between baths was between 5 and 10 seconds.

Microleakage testing

The teeth were dried with a dental air jet and covered with two coats of nail varnish (Resist and Shine, L'Oréal, Paris, France), leaving 1 mm around the edges of the bracket base uncovered. The specimens were then submerged in a solution of 1 per cent methylene blue for 24 hours. In order to avoid penetration by the methylene blue through the apical foramen, the teeth were placed vertically in a container with the roots in a metal grid so that the methylene blue only covered the crown of the tooth and the gingival third of the root.

Microscopic observations

The teeth were sectioned longitudinally in an inciso-cervical direction with a water-cooled diamond saw (Horico, Berlin, Germany), thus providing three sections per tooth.

Each section was examined on both sides, so that each specimen underwent six examinations. The percentage of microleakage for each face was determined using image analysis equipment (Sony dxc 151-ap video camera, connected to an Olympus SZ11 microscope) and MIP 4 software (Digital Image Systems) on the enamel–adhesive and adhesive–bracket interfaces, both on the gingival and incisal edge at \times 100 magnification. The total percentage of microleakage for enamel–adhesive and adhesive–bracket interfaces was obtained by summing the percentages of microleakage observed at the incisal and gingival edges of each interface. The mean percentage of microleakage for each of the six observations per tooth was calculated.

All observations were carried out by the same author (AV). Twenty five sections were examined with an interval of 1 month between the examinations. No significant differences for the total percentage of microleakage at the enamel–adhesive and adhesive–bracket interfaces between the first and second examination were found (P = 0.12 and P = 0.09, respectively).

Statistical analyses

As the data did not fulfil the criteria for normality or homogeneity of variance, non-parametric tests were used.

The existence of significant differences between the groups in total microleakage at each interface, with and without thermocycling, was analyzed using the Kruskal-Wallis test (P < 0.05), finding those groups which were significantly different with the Mann-Whitney U-test for two independent samples. In order to avoid accumulation of errors due to multiple comparisons, the significance level was modified dividing it (P < 0.05) by the number of comparisons made (Bonferroni correction) with P < 0.005considered as significant. For a single material, total microleakage for the enamel-adhesive interface without thermocycling was compared with the total for the same interface after thermocycling by means of the Mann-Whitney U-test (P < 0.05); the same comparison was made for the adhesive-bracket interface with and without thermocycling.

Results

The results are shown in Table 2. Comparison of total microleakage at the enamel–adhesive interface for the different materials without thermocycling showed significantly higher levels of microleakage for Admira-Flow than X-Flow (P = 0.004). At the adhesive–bracket interface, without thermocycling, no significant differences between materials were observed (P = 0.06).

When the materials were subjected to thermocycling, Beautiful-Flow showed significantly less microleakage than the other materials tested at the enamel–adhesive interface (Transbond XT P = 0.004, X-Flow P = 0.000, Dyract-Flow P = 0.000, and Admira-Flow P = 0.000); while at the adhesive–bracket interface, Admira-Flow and

Materials	Non-thermocycled					Thermocycled						
	Enamel-adhesive			Adhesive-bracket		Enamel-adhesive		Adhesive-bracket				
	Mean ± SD	Median	Range	Mean \pm SD	Median	Range	Mean \pm SD	Median	Range	Mean ± SD	Median	Range
Transbond XT	$1.12 \pm 20.26^+$	0.00	8.92	1.42 ± 2.02	0.11	5.46	1.54 ± 1.20	1.39	4.00	$1.64 \pm 1.41^{\circ}$	1.54	5.01
X-Flow	$0.77 \pm 1.63^{+}$	0.02	5.90	$1.86 \pm 2.16^{+}$	1.29	7.68	3.10 ± 2.70	2.67	10.16	4.74 ± 4.97^{d}	3.12	16.73
Dyract-Flow	1.78 ± 2.59	0.85	10.46	2.50 ± 1.99	2.28	7.40	2.36 ± 2.93	1.45	12.80	2.73 ± 3.75	1.53	15.01
Admira-Flow	2.51 ± 2.79 ^a	1.20	8.92	4.40 ± 6.68	2.85	28.61	4.35 ± 3.72	4.24	14.60	$4.45\pm3.87^{\text{d}}$	3.51	15.24
Beautifil-Flow	$2.01\pm3.17^{\scriptscriptstyle +}$	0.78	13.38	1.39 ± 1.35	1.22	4.00	$0.43\pm0.80^{\text{b}}$	0.00	2.84	$0.79 \pm 1.37^{\texttt{c}}$	0.02	5.34

 Table 2
 Percentage of total microleakage at the enamel–adhesive and adhesive–bracket interfaces.

Significant differences within the same column are indicated by superscript letters. For each material significant differences between thermocycled and non-thermocycled specimens, at each interface, are indicated by a plus (+) sign. + versus thermocycled (P < 0.05); * versus X-Flow (P < 0.005); b versus Transbond-XT, X-Flow, Dyract-Flow, and Admira-Flow (P < 0.005); c versus groups marked with d (P < 0.005); SD, standard deviation.

X-Flow showed significantly more microleakage than Beautiful-Flow (P = 0.000 and P = 0.000, respectively) and Transbond XT (P = 0.002 and P = 0.004, respectively).

When analyzing the effect of thermocycling on each material, with Transbond XT, total microleakage at the enamel–adhesive interface was significantly greater after thermocycling (P = 0.02); for X-Flow, thermocycling produced a significant increase in microleakage at both interfaces (enamel–adhesive P = 0.00 and adhesive–bracket P = 0.01); Beautiful-Flow showed total microleakage at the enamel–adhesive interface which was significantly less after thermocycling (P = 0.01). The values for total microleakage with Dyract-Flow (enamel–adhesive P = 0.23 and adhesive–bracket P = 0.39) and Admira-Flow (enamel–adhesive P = 0.07 and adhesive–bracket P = 0.39) were not significantly affected by thermocycling.

Figure 1a,b show the microleakage at the enameladhesive and adhesive-bracket interfaces, respectively.

Discussion

This study set out to determine the effect of thermocycling on microleakage beneath brackets bonded with an orthodontic composite resin, a flowable resin, compomer, ormocer, and giomer.

Bovine teeth were used since it is becoming more difficult to obtain extracted human teeth because of the increase in preventative dentistry. However, the microstructure and orientation of hydroxyapatite crystals in bovine enamel is similar to that of humans (Gomes, 2004). Many authors have used bovine teeth for bond testing of restorative materials (Yamamoto *et al.*, 2003; Fonseca *et al.*, 2005; Hayakawa *et al.*, 2005) and microleakage (Amaral *et al.*, 2004; Costa *et al.*, 2006; da Silva *et al.*, 2006; Ritter *et al.*, 2006) and bracket bond research (Cacciafesta *et al.*, 2006; Cozza *et al.*, 2006; Godoy-Bezerra *et al.*, 2006; Soderquist *et al.*, 2006).



Figure 1 Microleakage at the enamel–adhesive (a) and adhesive–bracket (b) interface.

Similar to the studies of Arhun *et al.* (2006) and Arikan *et al.* (2006), the microleakage at the two adhesive interfaces, which form the bracket–enamel union, was measured, given that the clinical consequences of microleakage differ at each

interface. While the enamel–adhesive interface is critical with regard to the development of white spots, the adhesive– bracket interface can play a part in bracket failure caused by bond degradation (Arikan *et al.*, 2006). Both Arhun *et al.* (2006) and Arikan *et al.* (2006) evaluated microleakage using an index, while in the present study, the percentage of microleakage was used, providing a quantitative variable which was therefore more precise with regard to the extent of microleakage.

Comparing total microleakage at the enamel-adhesive interface for the different materials without thermocycling, Admira-Flow showed a significantly higher percentage of microleakage than X-Flow. The volume of X-Flow's filling content is 38 per cent (Lohbauer et al., 2006) while that of Admira-Flow is 50.5 per cent (Üctasli et al., 2004). It is to be expected that the lower filling content of X-Flow contracts more under polymerization than Admira-Flow. However, when these materials are used for bracket bonding rather than in restorative dentistry, different factors play various roles. First, the adhesive layer is very fine and pressed between the bracket and tooth. Second, there is usually an excess of resin at the edges of the bond area which can absorb some of the shrinkage. Last, the bracket is free floating and shrinkage would pull the bracket closer to the enamel, which is probably more of an advantage than a disadvantage (Oesterle et al., 2001). Thus, resin shrinkage is probably not a concern (Oesterle et al., 2001) and the lower rigidity of the flowable composites may be a counteracting factor (Labella et al., 1999) because the elastic modulus plays an important role in preventing microleakage (Yap et al., 2004).

When the materials investigated were subjected to thermocycling, Beautiful-Flow showed significantly less microleakage at the enamel–adhesive interface than the other adhesives tested. This might be due to the dimensional change (swelling) that giomers undergo because of uptake of water into the structure of the material when exposed to fluids (McCabe and Rusby, 2004). The loss of volume caused by polymerization is greater for the flowable composites due to their higher proportion of resin. This loss of volume will produce empty gaps in the indentations of the grid on the bracket base, decreasing the retentive capacity and therefore increasing microleakage at this interface.

No significant differences were observed at the adhesive– bracket interface without thermocycling, whereas after thermocycling, Beautiful-Flow and Transbond XT showed significantly less microleakage than Admira-Flow and X-Flow. The improved performance of Beautiful-Flow at this interface can also be explained by its water uptake (McCabe and Rusby, 2004). With regard to Transbond XT, it has been shown that this system bonds better at the adhesive–bracket interface than flowable composites (Uysal *et al.*, 2004).

When the effect of thermocycling on each material was analyzed individually, the composite resins were more affected, and of the two resins tested, the flowable composite underwent more microleakage than the more traditional material: for Transbond XT, thermocycling caused a significant increase in total microleakage at the enameladhesive interface, while for X-Flow, total microleakage increased significantly at both interfaces. Thermal expansion is a factor affecting adhesion between the bond material and tooth difference in the thermal expansion coefficient between the bracket-adhesive-enamel strata leads to different dimensional changes when they are subjected to changes of temperature (Sideridou et al., 2004). Transbond XT performed better than X-Flow because it has a greater percentage volume of inorganic filling which diminishes the thermal expansion coefficient, bringing it closer to that of tooth enamel, and preventing long-term microleakage (Uysal et al., 2004).

The fact that Admira-Flow did not show greater microleakage after thermocycling could also be related to the thermal expansion coefficient, as the coefficient for ormocers, according to the manufacturers approximates that of the natural tooth structure (Voco Scientific Circular, 2001).

With Beautiful-Flow, microleakage at the enameladhesive interface decreased significantly after thermocycling. The absorption of water by restorative materials is a factor in the reduction of microleakage (Attin *et al.*, 1995). Giomers undergo a significant degree of dimensional change (swelling) when exposed to fluids. A positive effect of water take-up for restorative materials is that it provides a mechanism for compensating shrinkage resulting from polymerization.

With Dyract-Flow, total microleakage was not affected by thermocycling. The main difference in microstructure between giomers and compomers is the presence of prereacted glass-polyacid zones which make up part of the filling in the structure of giomers (McCabe and Rusby, 2004). Compomers are similar to composite resins in that they are fundamentally hydrophobic, although less so than composites. Compomers are set by a polymerization reaction, and it is only once they are set that the minority hydrophilic constituents draw in a limited amount of water to promote a secondary neutralization reaction. Polymerization is associated with contraction and the development of measurable stresses, and it may be that the absorption of water plays some part in reducing these stresses (Nicholson, 2007).

Although microleakage-orientated caries has been well documented in restorative dentistry (Gladwin and Bagby, 2004), as stated by Arikan *et al.* (2006) 'the potential of caries adjacent to and beneath orthodontic brackets still remains as an underestimated threat to the permanent tooth, especially with regard to long term fixed appliance therapy'. Studies of microleakage can contribute to knowledge of the efficacy of orthodontic adhesives. Some degree of microleakage was found in all the groups tested, a fact which highlights the importance of evaluating microleakage beneath brackets.

Conclusions

Some degree of microleakage was found in all the groups investigated. The giomer presented the best performance after thermocycling, while composite, and, in particular, the flowable resins showed a poorer performance.

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