Original Article

The Influence of Orthodontic Adhesive Properties on the Quality of Orthodontic Attachment

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Abstract: The objective of this study was to determine the resilience, glass transition temperature (Tg), ultimate flexural strength (UFS), and penetration coefficient of 3 composite adhesives (Concise, Transbond, and Right On) and a glass ionomer cement (Fuji Ortho LC). For 25 minutes after initial set, the composite materials were significantly more resilient than Fuji Ortho LC ($P = .000$). Resilience values for all materials increased for up to 90 minutes after initial set, reflecting a continuation of their setting reaction. Right On remained the most resilient material at 90 minutes after initial set ($P \leq .01$). At 120 minutes, there was no significant difference in the resilience of Concise, Right On, and Fuji Ortho LC. However, Transbond was significantly less resilient than these materials ($P \leq .01$). The composite materials recorded significantly higher glass transition values (89–123°C) than Fuji Ortho LC (54°C). The composite materials were also significantly stronger (61–68 MPa) than Fuji Ortho LC (35 MPa) in flexion. The penetration coefficients of Concise and Transbond were significantly higher ($P \leq .001$) than Right On and Fuji Ortho LC. However, the relative penetration coefficients of the materials studied did not appear to influence the degree of bracket base penetration achieved by the cements. In conclusion, Fuji Ortho LC offered a reduced energy-absorbing capacity immediately after bracket placement and reduced cohesive and mechanical adhesive strength. In addition, the glass transition temperature of Fuji Ortho LC was low; this may result in compromised attachment at temperatures above 60°C. Significant differences in the penetration coefficient of the materials studied have been recorded. However, the penetration of the 4 cements into a range of bracket base morphologies was uniformly good. Therefore, the differences in penetration coefficient recorded should not influence the quality of the bracket cement interface provided. (*Angle Orthod* 2000;70:241–246.)

Key Words: Orthodontic adhesive; Resilience; Glass transition temperature; Ultimate flexural strength; Penetration coefficient

INTRODUCTION

A dental material can only be properly developed and applied when the physical properties of the material are known. The mechanical properties of many orthodontic cements are strain-rate–sensitive or viscoelastic. If a sinusoidal stress of prescribed frequency is applied and the resultant strain measured, an in-phase modulus (E′), representing energy storage or elastic behavior, and an out-of-phase modulus (E″), representing energy loss or viscous properties, can be calculated. The ratio of out-of-phase to in-phase modulus (E″/E′) is represented as tan δ. Tan δ is closely allied to the resilience of a material—that is, the amount of energy absorbed by a material when it is not stressed beyond its proportional limit.[1] A tan δ value of 0 would indicate perfect elasticity and high resilience. The higher the tan δ value, the higher the energy absorption, and the lower the resilience. It can be appreciated that the resilience of an orthodontic cement will influence the quality of an orthodontic attachment.

The peak of the tan δ output, when measured for a material over a range of temperatures, reflects the glass transition temperature (Tg). This is the temperature at which the molecular chains of a material have obtained sufficient energy to allow intermolecular bond rotation. Under these conditions, a material transforms from a frozen, glasslike condition with limited mobility to a more mobile system

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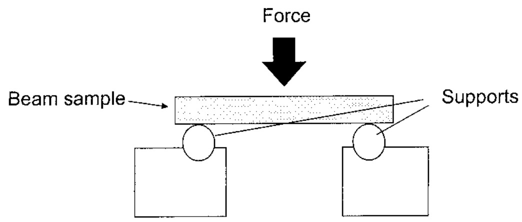

FIGURE 1. Three-point bending test. The span between supports 20 mm.

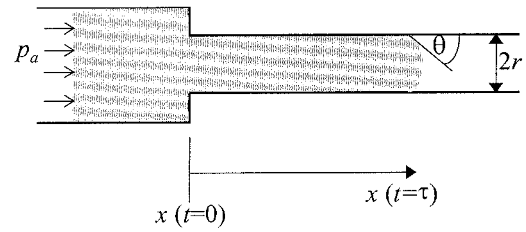

FIGURE 2. Measurement of a penetration coefficient—cement in calibrated tubing.

that achieves thermodynamic equilibrium. The Tg of an orthodontic cement is only of relevance if it lies within the range of intraoral temperatures.[2] Intraoral temperatures that exceed the Tg may result in failure of attachment or attachment movement and appliance deactivation.[3]

The ultimate flexural strength (UFS) is the flexural stress required to induce fracture within a material. The values determined are not a measure of individual interatomic forces of attraction or repulsion but instead a reflection of collective interatomic forces across the material. Mount[4] has suggested that the cohesive strength of an orthodontic cement is the primary determinant of bond strength.

Finally, the quality of orthodontic attachment is influenced by the geometry of the bracket-cement interface. This is primarily determined by the flow of orthodontic cement into the undercuts provided by the bracket base. The degree of penetration will determine the dimensions and physical properties of the resin tags, and any areas of incomplete penetration could lead to stress concentrations

TABLE 1. Tan δ 1 Hour After Initial Set

Temperatures °C	Concise		Transbond		Right On		Fuji Ortho LC	
	Mean	Standard Deviation	Mean	Standard Deviation	Mean	Standard Deviation	Mean	Standard Deviation
30	0.0888	0.0093	0.0889	0.0030	0.0492	0.0040	0.1302	0.0201
35	0.0957	0.0091	0.0977	0.0045	0.0496	0.0047	0.1378	0.0237
37.4	0.1001	0.0096	0.1023	0.0040	0.0529	0.0049	0.1417	0.0257
40	0.1033	0.0107	0.1121	0.0040	0.0554	0.0052	0.1463	0.0264
45	0.1106	0.0086	0.1277	0.0053	0.0598	0.0053	0.1548	0.0287
50	0.1220	0.0113	0.1452	0.0068	0.0647	0.0069	0.1625	0.0282
55	0.1394	0.0080	0.1643	0.0080	0.0716	0.0074	0.1665	0.0276
60	0.1490	0.0052	0.1773	0.0088	0.0816	0.0100	0.1713	0.0242

TABLE 2. Comparison of Mean Tan δ Levels 1 Hour After Initial Set

Cements	Temperature, °C							
	30	35	37.4	40	45	50	55	60
Concise								
Transbond	1.000	0.990	0.998	0.627	0.145	0.033*	0.015*	0.002**
Right On	0.000***	0.000***	0.000***	0.000***	0.000***	0.000***	0.000***	0.000***
Fuji	0.000***	0.000***	0.000***	0.000***	0.000***	0.000***	0.008**	0.018*
Transbond								
Concise	1.000	0.990	0.998	0.627	0.145	0.033*	0.015*	0.002**
Right On	0.000***	0.000***	0.000***	0.000***	0.000***	0.000***	0.000***	0.000***
Fuji	0.000***	0.000***	0.000***	0.000***	0.008**	0.156	0.992	0.832
Right On								
Concise	0.000***	0.000***	0.000***	0.000***	0.000***	0.000***	0.000***	0.000***
Transbond	0.000***	0.000***	0.000***	0.000***	0.000***	0.000***	0.000***	0.000***
Fuji	0.000***	0.000***	0.000***	0.000***	0.000***	0.000***	0.000***	0.000***
Fuji								
Concise	0.000***	0.000***	0.000***	0.000***	0.000***	0.000***	0.008**	0.018*
Transbond	0.000***	0.000***	0.000***	0.000***	0.008**	0.156	0.992	0.832
Right On	0.000***	0.000***	0.000***	0.000***	0.000***	0.000***	0.000***	0.000***

* Significant at $P < .05$ (Tukey's post hoc test).
** Significant at $P < .01$ (Tukey's post hoc test).
*** Significant at $P < .001$ (Tukey's post hoc test).

and reduced interfacial strength. The primary determinants of cement flow are the penetration coefficient of the cement, determined by surface chemistry and the pressure of application. In addition, employing Poiseille's law for the flow of a Newtonian fluid, it can be demonstrated that fluid penetration is proportional to the square root of time, implying that low viscosity cements with long working times will penetrate into pores more readily than high-viscosity cements with short working times. Fan et al[5] reported that the time required for a sealant to penetrate a specific distance is highly dependent on the penetration coefficient of the sealant. The penetration coefficient of the orthodontic cement is therefore an important parameter to determine together with a quantification of cement penetration of bracket base relief.

MATERIALS AND METHODS

Sample preparation for resilience, UFS, and Tg evaluation

Four different orthodontic cements were evaluated: Concise (3M Company, St Paul, Minn), Transbond (3M Unitek, Monrovia, Calif), Right On (TP Orthodontics Inc, La Porte, Ind), and Fuji Ortho LC (GC Corporation, Tokyo, Japan). Beam specimens of each material were prepared in stainless steel molds with internal dimensions of 2.0 mm $\times$ 2.0 mm $\times$ 25.0 mm (ISO 4049). A separating medium was applied to the mold, and the cement was packed to excess. A glass slide was then applied under pressure to create the fourth surface of the chamber.

Equal volumes of the 2 constituent Concise pastes were mixed according to the manufacturer's instructions and allowed to cure until completely set. The light-cured materials (Transbond and Fuji Ortho LC) were cured by exposure to a visible blue-light unit (Luxor, ICI light unit, Macclesfield, UK). We used 3 overlapping exposures, each of 60 seconds' duration. The beam samples were then removed from the mold and the light-cured samples were exposed to an additional 60 seconds of blue light on their deep surface.

The preparation of beam samples of Right On no-mix cement proved difficult. The clinical application of this material involves surface contact of thin sections of the base paste with an activator painted onto the tooth surface and bracket base. The action of the activator is therefore superficial and inadequate for the preparation of 2-mm-thick beams. To ensure complete cure of the cement, paste and activator were mixed in 5:1 proportions that provided appropriate working and setting times.

After the removal of all flash, each sample was stored in distilled water at 37°C for 1 hour, where appropriate. Each of the samples was then accurately measured with an Electronic Digital Micrometer (CADAR Measurement and Control Systems, Sheffield, UK).

Resilience determination

When a stress is applied to a perfectly elastic solid, the deformation (strain) occurs in phase with the applied stress. A completely viscous material would, however, respond with a deformation that lagged behind the applied stress by 90°. When a sinusoidal stress is applied to a viscoelastic material, the material will respond in a way which lies somewhere between these extremes, with the resultant strain lagging behind the applied stress by some angle θ (where $\theta < 90°$).

The Dynamic Mechanical Thermal Analyser (DMTA; Polymer Laboratories, Epsom Surrey, UK) was employed to determine resilience. The DMTA applies a sinusoidal stress of prescribed frequency and measures the resultant strain determining E' modulus, representing energy storage or elastic behavior and E' modulus, representing energy loss or viscous properties. The ratio of E'' to E' moduli is presented as tan δ. Tan δ was determined for each material at 1 hour after initial set over a temperature range of 30–160°C (encompassing extremes of intraoral temperature), at a constant frequency of 2 Hz. The frequency selected represented an average chewing frequency.[6] In addition, tan δ was recorded over the first 2 hours after initial set at a frequency of 2 Hz and a constant temperature of 37°C.

Flexural strength determination

Each sample was mounted in a 3-point loading jig (Figure 1). The Lloyds testing machine (Lloyds Instruments LR10K, Lloyds Instruments, Fareham, Hants, UK) was used to deliver the applied load and measure the sample deflection and UFS in this study. The Lloyds machine was fitted with a 1000-N load cell and programmed to deliver force at a crosshead speed of 0.75 mm/min (ISO 4049). Each sample was tested to destruction, and the UFS was recorded.

Penetration coefficient

In the study of Fan et al[5] that evaluated fissure sealants and a similar study by Faust et al[7] that investigated orthodontic cements, the penetration of resins along horizontal capillary tubes of varying diameter against time was recorded. In both studies, there was a suggestion that solvents[5] or altered mixing proportions[7] were introduced to alter resin viscosity before testing. In the present investigation, rather than alter the cement consistency, a known pressure was prescribed to assist cement flow and penetration distance against time plotted for each of the cements evaluated. The apparatus, schematically shown in Figure 2, was used to measure the penetration over time for the orthodontic cements studied. For each sample, 0.01-mL glass capillary tubing was attached to the end of a 1.0-mL syringe. The cement to be evaluated was placed into the chamber of the syringe, and the plunger was advanced until the cement entered the capillary tubing.

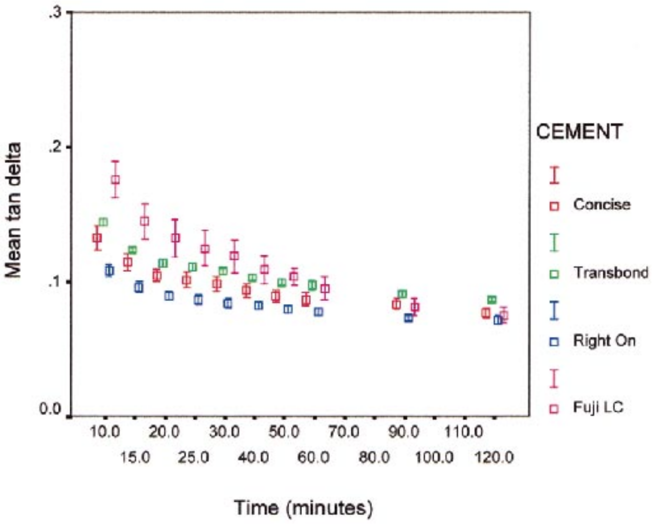

FIGURE 3. Mean tan δ 0–2 hours after initial set showing 95% confidence interval (37°C).

To prevent the setting reaction from altering the viscosity of the cement, the light-cured materials (Transbond, Fuji Ortho LC) were shielded with blue-light filters. The constituent Concise pastes were evaluated separately and the activator was not added to the Right On cement. The apparatus was aligned in the Lloyds testing apparatus, and the machine was programmed to deliver an accurately prescribed constant force (range, 5–12 N) for 30 seconds at a cross-head speed of 50 mm/min. Two different forces were prescribed for each cement, the displacement of the cement over time was recorded, and the penetration coefficient was calculated.

Cement penetration

The extent of cement penetration into the bracket base undercut was evaluated in this study by microscopic evaluation of transverse sections through the bracket cement interface. The penetration of the same 4 orthodontic cements into 6 different bracket base designs was compared. The bracket bases studied included 60, 80, and 100 mesh discs (American Orthodontics Ltd, Sheboygan, Wis), the Omni double-mesh base (GC Corporation), and the Dynalock and Mini Twin bases (3M Unitek). The Mini Twin and Dynalock brackets are commercially available precoated with Transbond cement. The application of cement by the manufacturer could theoretically influence the geometry of the bracket-cement interface. A direct comparison was therefore made between the resin penetration of precoated and conventionally loaded Mini Twin and Dynalock brackets.

For each of the 60, 80, and 100 mesh bases, 3-mm discs were prepared. Lower incisor brackets represented the remaining base morphologies. For each cement, 10 discs/brackets were cemented to 100 × 6 mm stainless steel strips by use of normal clinical practice for bracket location. Each cement was prepared according to the manufacturers' instructions, and the chemically cured cements (Concise, Right On) were allowed to set. The light activated materials (Transbond, Fuji Ortho LC) were cured with a 60-second exposure to blue light.

When fully set, the samples were invested in Stycast 1266 polymer (Grace, NV Nijverheidsstraat 7, Belgium) that was allowed to set for 24 hours. The resultant 100 × 7 × 3–mm acrylic strips, each containing 10 disc/bracket samples, were sectioned with a Microslice (Malvern Instruments, Malvern, UK), dividing each disc/bracket into 2 halves. This process generated 20 transverse sections of the bracket-cement interface. However, to ensure that individual samples represented a distinct area of the bracket-cement interface, the cut surface of each section was reduced by approximately 50 μm with 500 μm abrasive paper. The samples were finally polished with 5–20 μm abrasive paper and examined under the light microscope with incident light at 40× magnification.

The extent of cement penetration could be clearly evaluated by use of this technique. Where mesh wires running in the plane of the section obscured vision, further reduction was performed to allow evaluation of the complete mesh

TABLE 3. Tan δ 0 to 2 Hours After Initial Set

	Concise		Transbond		Right On		Fuji Ortho LC	
Time, min	Mean	Standard Deviation	Mean	Standard Deviation	Mean	Standard Deviation	Mean	Standard Deviation
10	0.1323	1.07E–02	0.1440	3.06E–03	0.1082	5.60E–03	0.1757	1.63E–02
15	0.1141	7.38E–03	0.1237	2.25E–03	0.0956	4.86E–03	0.1446	1.60E–02
20	0.1043	5.67E–03	0.1138	3.24E–03	0.0894	3.71E–03	0.1321	1.67E–02
25	0.1011	6.99E–03	0.1109	3.15E–03	0.0867	4.59E–03	0.1245	1.55E–02
30	0.0985	6.38E–03	0.1079	2.73E–03	0.0839	3.95E–03	0.1185	1.48E–02
40	0.0935	5.80E–03	0.1029	2.85E–03	0.0820	2.83E–03	0.1086	1.26E–02
50	0.0893	5.30E–03	0.0992	2.66E–03	0.0793	2.94E–03	0.1033	7.49E–03
60	0.0868	5.89E–03	0.0972	4.16E–03	0.0773	2.95E–03	0.0948	1.03E–02
90	0.0832	4.84E–03	0.906	3.07E–03	0.0730	2.42E–03	0.0812	7.48E–03
120	0.0768	4.13E–03	0.865	2.47E–03	0.0716	3.80E–03	0.0702	7.02E–03

TABLE 4. Summary of Variables Analyzed[a]

Cement	Tg, °C		UFS, MPa		PC, $\times 10^{-5}$	
	Mean	Standard Deviation	Mean	Standard Deviation	Mean	Standard Deviation
Concise	89.76	13.14	68.02	9.29	5.132	1.49
Transbond	96.11	3.12	61.11	9.64	5.649	1.51
Right On	123.58	4.95	67.79	7.63	2.507	0.83
Fuji Ortho LC	54.06	9.04	35.17	4.97	2.745	1.04

[a] Tg indicates glass transition temperature; UFS, ultimate flexural strength; and PC, penetration coefficient.

depth. Difficulty was experienced when the double-mesh base was evaluated. No individual section demonstrated the lower of the 2 meshes across the complete bracket length. These samples were evaluated by examining serial sections of the mesh until the lower mesh could be investigated in its entirety. Those samples in which incomplete cement penetration was suspected were gold sputtered and examined under a scanning electron microscope. The area of any voids present was measured by Seescan software (Seescan Plc, Level 3, Cambridge, UK) and presented as a percentage of the total area of the bracket-cement interface.

RESULTS

We used descriptive statistics, 1-way and general factorial analysis of variance, and Kruskal-Wallis nonparametric tests, where appropriate, to analyze the data. Tan δ values at 1 hour after initial set, between 30°C and 160°C, are presented in Table 1. Mean values are compared in Table 2. Tan δ values at 37°C, over the first 2 hours after initial set, are presented in Figure 3 and Table 3. Tg, UFS, and penetration coefficient values are presented in Table 4. Mean values are compared in Table 5. The maximum area of incomplete resin penetration recorded for any cement/bracket base studied was 0.02%. For most materials, the incidence of voids at the bracket cement interface was 0. Therefore, no meaningful comparisons could be made.

TABLE 5. Comparison of Mean Values Analyzed[a]

Cement	Tg	UFS	PC
Concise			
Transbond	0.435	0.243	0.884
Right On	0.000***	1.000	0.000***
Fuji Ortho LC	0.000***	0.000***	0.000***
Transbond			
Concise	0.435	0.243	0.884
Right On	0.000***	0.270	0.000***
Fuji Ortho LC	0.000***	0.000***	0.000***
Right On			
Concise	0.000***	1.000	0.000***
Transbond	0.000***	0.270	0.000***
Fuji Ortho LC	0.000***	0.000***	0.970
Fuji Ortho LC			
Concise	0.000***	0.000***	0.000***
Transbond	0.000***	0.000***	0.000***
Right On	0.000***	0.000***	0.970

[a] Tg indicates glass transition temperature, UFS, ultimate flexural strength; and PC, penetration coefficient.
* Significant at $P < .05$ (Tukey's post hoc test).
** Significant at $P < .01$ (Tukey's post hoc test).
*** Significant at $P < .001$ (Tukey's post hoc test).

DISCUSSION

Flexural strength

Mount[4] has suggested that the cohesive strength of orthodontic cement is the primary determinant of bond strength. The differences demonstrated in UFS may therefore have a large influence on the quality of attachment. The composite materials (Concise, Transbond, and Right On) recorded UFS values of 61–68 MPa. This is in contrast to Fuji Ortho LC, which recorded 21–35 MPa.

The strength of the orthodontic cement influences the quality of orthodontic attachment by determining the load at which failure will occur cohesively within the body of the cement or within the areas of cement that penetrate into bracket base undercuts and etched enamel porosities. The relatively low cohesive strength of the glass ionomer cement might be one of the reasons that it provides a weaker orthodontic attachment. However, within the bracket-cement-enamel system, the adhesion of the cements to bracket base and enamel also contribute to the quality of attachment. It will be the weakest link in the system under load that determines the strength of the attachment.

Resilience

Tan δ is closely allied to the resilience of a material—that is, the amount of energy absorbed by a material when it is not stressed beyond its proportional limit.[1] A tan δ value of 0 would indicate perfect elasticity and high resilience. The higher the tan δ value, the higher the energy absorption and the lower the resilience.

Of interest in this study were the comparative resilience of the materials and the change of resilience with time. Tan δ was recorded initially as a function of temperature. For all materials, an increase in tan δ (decreased resilience) was recorded with increased temperature.

When measured at a constant temperature of 37°C, the composite materials were significantly more resilient than Fuji Ortho LC during the first 25 minutes after initial set ($P = .000$). Resilience values for all materials were seen to increase for up to 90 minutes after initial set, reflecting a continuation of their setting reaction. Right On remained the most resilient material at 90 minutes after initial set ($P \leq .01$). At 120 minutes, there was no significant difference in the resilience of Concise, Right On, and Fuji Ortho LC. However, Transbond was significantly less resilient than these materials ($P \leq .01$).

The continued change in tan δ over the 2-hour period after initial set is demonstrated in Figure 3. All materials showed an increase in resilience that, in most cases, reduced in rate after 60–90 minutes. Concise, Transbond, Right On, and Fuji all showed a significant increase in resilience between 30 and 60 minutes after initial set ($P \leq .003$). These materials demonstrated a further increase in resilience between 60 and 120 minutes ($P \leq .003$), but no material recorded any significant change between 90 and 120 minutes.

The changes in resilience reflect the materials cure evolution. Whether chemically or light activated, these materials undergo an immediate polymerization that is followed by a cross-linking process. The increased resilience with time recorded in this study suggests that the cross-linking process is continuing for at least 60 minutes after initial set.

An interesting feature of the tan δ measurements is the altered resilience of Fuji Ortho LC when stored in water. In both studies, the samples were evaluated at 1 hour after initial set and at 37°C. However, storage in water for 1 hour after initial set appears to result in Fuji Ortho LC offering a reduced resilience. This has obvious clinical implications.

Glass transition temperature

The peak of the tan δ output, when measured for a material over a range of temperatures, reflects the Tg. Right On recorded a Tg that was significantly higher ($P = .000$) than all other materials. The other composite materials (Concise and Transbond) recorded significantly higher Tg values than Fuji Ortho LC ($P = .000$). However, it is not the relative transition temperatures of the cements studied that are of major clinical importance. The low Tg of Fuji Ortho LC lies within the range of intraoral temperature and temperatures of 50°C to 60°C could result in softening of the cement and attachment failure.

Penetration coefficient

The measurement of penetration coefficient suggested that the orthodontic cements studied might perform differently when penetrating bracket base or etched enamel undercuts. An interesting outcome of this study was the relatively low penetration coefficients recorded by the low viscosity materials (Right On and Fuji Ortho LC). The penetration coefficient of a material is a product of the material's surface tension, contact angle (which influences wetability), and viscosity. It must be assumed that the penetration coefficient recorded for these materials is largely determined by physical properties other than their viscosity.

However, the methodology employed in this section was novel, and there was no mechanism for calibration of the apparatus. Relative values have been provided for each material, but how these relate to reality is difficult to assess without evaluating a material with established values for viscosity and surface tension.

Cement penetration

Analysis of the results of the cement penetration study indicated that all cements achieved exceptionally good penetration into the bracket bases studied. The incidence of voids at the bracket cement interface was too low for any comparison to be made. This would suggest that the recorded differences in penetration coefficient were not large enough to influence cement penetration into the bases studied. Alternatively, the applied force overcame any differences in penetration coefficient.

The study suggests that differences in cement viscosity and penetration coefficient are not of sufficient magnitude to prevent good penetration of the cements studied into the undercuts of the bracket bases evaluated.

CONCLUSIONS

Significant differences in the physical properties of the composite materials and Fuji Ortho LC have been recorded. The composite materials were generally stronger and more resilient; this characteristic should offer increased cohesive and mechanical adhesive strength.

The low Tg of Fuji Ortho LC is of clinical concern because intraoral temperatures above 50°C to 60°C could possibly compromise attachment.

The penetration coefficients of the materials studied differed significantly. However, the difference in penetration coefficient did not appear to influence the degree of cement penetration into the bracket bases studied.

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