Novel test to evaluate the bond strength of a luting cement

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A luting cement can be defined as a gap-filling mortar used to join two solid objects together, such as a crown and a tooth [1]. The luting applications of Glass Polyalkenoate Cements (GPCs) are limited by their lack of command set [2]. This can be addressed to some extent by the incorporation of a light curable resin [3] but these resin-modified GPCs (RMGPCs) have inherent problems, mainly due to the presence of the resin, in that they swell in aqueous media, they have poor long-term mechanical properties [4, 5], and there are toxicological concerns with microleakage of the monomer [6]. There is also a logistical problem in transmitting light through a brace or bracket to initiate polymerization. The authors have previously shown that ultrasound can be used to command set conventional, resin-free GPCs [7–11]. This is likely to be of major benefit in orthodontics as ultrasound can be transmitted through metal brackets to command set the luting GPC. However, there are currently no trials indicating whether this command set influences the strength of the bond in the tooth/GPC/bracket construct. The work contained herein evaluates the bond strength between the constituent components by developing a novel test method.

Bond strength is defined as the force per unit area required to break a bonded assembly with failure occurring in or near the adhesive interface [12]. The literature is devoid of any *in vivo* studies for determining what force or stress causes a bracket to de-bond and so any *in vitro* tests are unlikely to directly replicate the clinical situation.

The type of GPC used also affects bond strength. Chemical setting GPCs tend to fail cohesively whereas RMGPCs fail adhesively, the reason being that there are minimal defects within the resin phase leading to a higher bond strength [13].

This letter suggests a novel method of evaluating the bond strength between a GPC and both teeth and orthodontic brackets by using hydroxyapatite (HA) and hardened steel disks to mimic the tooth and bracket, respectively.

Four GPCs were employed for the test and these were set both conventionally and by ultrasound. GPCs were formulated from the following glasses:

Glass A: $4.5SiO_2{\cdot}3Al_2O_3{\cdot}1.5P_2O_5{\cdot}3SrO{\cdot}2CaF_2$
Glass B: $4.5SiO_2{\cdot}3Al_2O_3{\cdot}1.5P_2O_5{\cdot}3SrO{\cdot}2SrF_2$

These glasses were mixed with two different PAAs, E7 and E8 (Advanced Healthcare Limited, Kent, UK), to produce four GPCs; A/E7, A/E8, B/E7, and B/E8. The molar mass details of the PAAs are included in Table I. Tartaric acid (TA) was incorporated at 10 wt.%. The powder:acid:liquid (P:A:L) mixing ratio (glass:acid:water/TA solution) used was 9:2:4; designed to mimic the handling properties of commercial luting GPCs.

All GPCs were handmixed with a spatula on a glass slab. The ultrasonic equipment employed for command setting was a Piezon® Master 400 dental scaler (EMS, Nyon, Switzerland), with a frequency of 25–30 kHz. The insert used (DS-003) was developed for scaling applications.
In order to ensure that the GPCs used were suitable for luting purposes, the film thicknesses of the cements were measured in line with ISO 9917 [14]. In order for a GPC to pass the ISO standard for film thickness, four of the five measurements recorded should be below 25 μm.
120 HA disks, (32 mm Ø × 3 mm ht), were produced by mixing 4.5 g HA powder (Stryker Howmedica Osteonics, Cork, Ireland) with 0.7 ml deionized water. The slurries were then cold pressed (10 tonnes, 20 s) and subsequently sintered to 1220 °C by an accepted regime [15].
Hardened steel disks (50 mm Ø × 3.5 mm ht), each with a hole (13 mm Ø) in the center, were machined from steel sheet (Engineering Steels Ltd., Limerick, Ireland).
Samples ($n = 5$) were prepared from each of the cements. Immediately after mixing, the GPC was placed onto the steel disk. The HA disk was placed on top of the cement, parallel to the steel disk and the construct was clamped and stored in an oven (37 °C, 15 min). The samples were subsequently removed from the oven, wrapped in tissue paper saturated with distilled water, and placed back in the oven for 1 and 7 days. Fig. 1a shows a test specimen.
The HA/cement/steel sandwich was produced as above. Ultrasound was then applied through the steel disk for

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TABLE I Molar mass details of the poly(acrylic) acids

CODE	Mw	Mn	PD
E7	25 700	8140	3.2
E8	51 900	21 900	2.4

TABLE II Film thickness of GPCs

	A/E7	A/E8	B/E7	B/E8
Mean FT μm (St. Dev.)	21 (3)	23 (1)	22 (2)	23 (1)

45 s. The samples were subsequently wrapped in tissue paper saturated with distilled water and placed in an oven (37 °C) for 1 and 7 days.

Fig. 1b shows the experimental setup that was used to measure the bond strength of the constructs. The diameter of the punch is 12 mm. The HA disk is under the steel disk and the maximum load required to push the HA disk from the steel disk was recorded for each sample. Bond strengths were measured after 1 and 7 days.

The bond strength was calculated using the following formula:

$$\sigma = \frac{F}{A}$$

where σ is the bond strength (MPa), F is the force applied (N), and A is the area (mm).

The area was calculated as follows:

$$\text{Area of HA disk} = \pi r^2, \quad \text{where } r = 16\,\text{mm} \quad \text{(A)}$$
$$\text{Area of whole in steel disc} = 6.5\,\text{mm} \quad \text{(B)}$$
$$\text{Test area} = \text{A} - \text{B}$$

Table II shows the film thickness of each GPC.

All GPCs passed the film thickness requirement.

Fig. 2 shows the bond strength of each of the cements with respect to setting technique. Although the results are dependent upon the specific GPC employed, the graph indicates:

- Bond strength increases with maturation time.
- The application of ultrasound results in a greater bond than chemical setting.

Figure 1 (a) Method used to measure bond strength. HA disk is pushed off the steel disk using the loaded piston. (b) Test specimen prior to bond strength measurement. HA disk is beneath the steel disk.

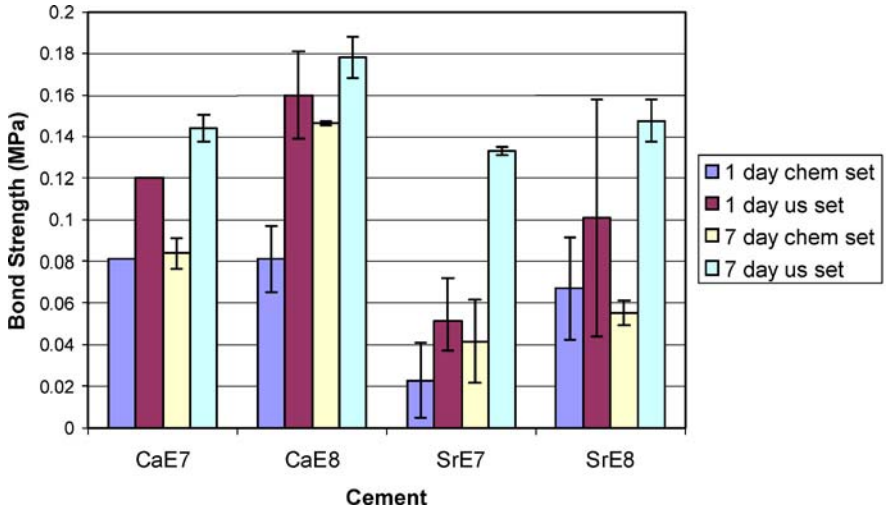

Figure 2 Bond strength for the GPCs after 1 and 7 days.

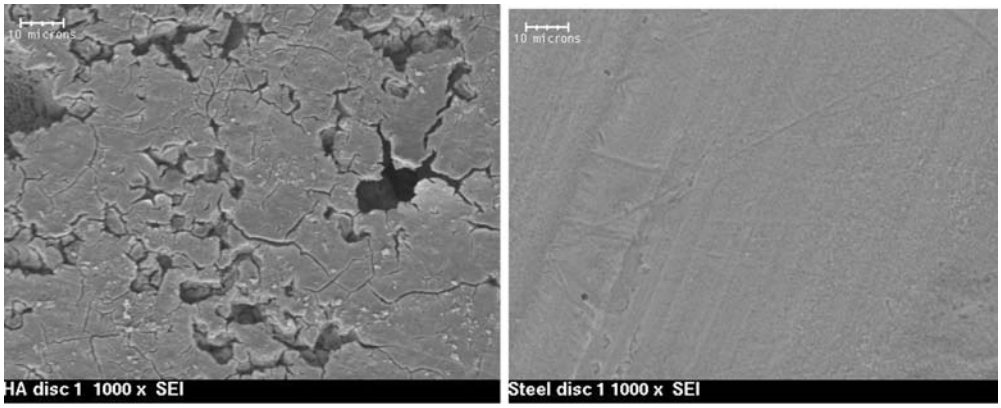

Figure 3 SEM micrograph of (a) HA disk with cement attached after de-bonding, (b) steel disk after de-bonding.

• For both maturation times, the ultrasonically set GPCs produced a stronger bond than those set chemically.

Failure in all cases was adhesive at the interface with the HA. Fig. 3 shows SEM micrographs of the HA and the steel after de-bonding.

This paper shows how a novel testing rig can be designed to evaluate the bond strength between luting GPCs and substrates.

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