

Relationship between Fracture Toughness and Tensile Peel Strength of Different Types of Luting Cements

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ABSTRACT

Objectives: To study the possible correlation between the fractures toughness of different luting cements with their tensile peel strength.

Methods: Six cements were investigated; two chemically adhesive resin cements (Super-Bond C&B and Panavia 21), one compomer cement (Dyract Cem), two resin-modified glass ionomer cements (Fuji Plus and RelyX Luting), and one conventional glass ionomer cement (Ketac Cem). The tensile peel strength was investigated by bonding grit-blasted Ni/Cr alloy beams to a block of the same alloy using the different types of luting cements (n = 20 for each cement), leaving half the length of the beam free. Beams were pulled off the block with a peeling action by applying a tensile load to the free end of the beam and load at failure point recorded. The values of the tensile peel strength were compared with the known fracture toughness values of these cements from the dental literature that were measured after 24 hours and after seven days.

Results: Direct relationship was found between the fracture toughness of the luting cements and their tensile peel strength. The linear regression analysis showed that the correlation coefficient (r) is equal to 0.94 and 0.98 when compared with 24 hours and 7days fracture toughness data respectively.

Conclusion: Luting cement with higher fracture toughness has higher tensile peel strength and thus better potential for retention of resin bonded bridge.

Key words: Tensile peel strength, Fracture toughness, Luting cement, Resin bonded bridge

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Introduction

The primary disadvantage of the resin bonded bridge (RBB) is that the longevity of the prosthesis is less than that for conventional prosthesis.^(1,2) Improvements in the prosthesis design, preparation design and adhesive bond strength enhance the survival rates of RBB but there are still an unacceptable number of clinical failures mostly because of debonding.⁽³⁻⁵⁾ The tensile peel strength (TPS) test as a means of assessment of bonding of RBB was explored by

Northeast *et al.*⁽⁶⁾ where Ni/Cr beams were bonded to Ni/Cr blocks by adhesive luting cement. The loading conditions result in a peeling action at the adhesive interface, providing a possible explanation for failure of RBB rather than failure being attributable to poor clinical or laboratory technique.⁽⁶⁾ In this study, it was shown that the TPS is a function of the thickness of the retainer *i.e.* the thicker the retainer, the higher tensile peel force required to cause failure.⁽⁶⁾ With thicker retainer the level of

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stress within the adhesive layer was reduced, suggesting that the stress the adhesive layer has to withstand is an important contributory factor to the clinical outcome and is governed by enhancing the mechanical properties of the luting cements. It is therefore proposed that the fracture toughness (KIC) of the luting cement has a major role in determining the retention of RBB. KIC is defined as the amount of energy required to propagate a surface flaw or a pre-existing crack through a material, causing catastrophic fracture.⁽⁷⁾ It is a measure of the critical stress at the tip of a flaw that allows propagation of a crack under tension.⁽⁸⁾ KIC is the lowest stress at which catastrophic crack propagation can occur.^(8,9)

Although there were some investigations that reported on the KIC of luting cements, little information is available on the relationship between the KIC of the cements and their adhesive bond quality. The purpose of this study was to measure the TPS of different types of luting cements bonded to Ni/Cr alloy. Then compare the values of TPS of these cements with their known KIC values from the dental literature^(10,11) to examine the possible correlation between them.

Methods

Six cements were investigated; two chemically adhesive resin cements (Super-Bond C&B and Panavia 21), one compomer cement (Dyract Cem), two resin-modified glass ionomer cements (Fuji Plus and RelyX Luting), and one conventional glass ionomer cement (Ketac Cem). Twenty Ni/Cr alloy beams (Talladium-V, Talladium, Bucks, UK) 22mm long, 5 mm wide and 0.5 mm thick were used. A 1mm diameter central hole was drilled 1.5mm from one end of each beam. A 15 mm brass block (20 blocks) with a Ni/Cr alloy base bonded to one of its surfaces was also used in this study. Ni /Cr beams and blocks were blasted with fresh 50µm alumina grit, washed in distilled water in an ultrasonic cleaner for 5 minutes and then air-dried before bonding with the luting cements. Manufacturers' specifications as to the correct mixing time, paste-to-paste and powder-to-liquid ratios were carefully followed during mixing of luting cement. After mixing the luting cement, it was applied to the fitting surface of the beam.

The beam was aligned perpendicular to the centre of the free edge of the Ni-Cr block such that a 10 mm length of the beam was bonded to the block with the aid of an alignment jig. A compressive load of 40 N was applied vertically to the beam (at about the middle of the 10 mm bonded to the block) during setting of the cement using a Lloyd universal testing machine to produce consistent cementation procedure. Excess cement was removed. Twenty samples were made for each type of the tested cements. The samples were stored in the dry air at room temperature and tested after 24 hours. The prepared specimens were mounted in a Lloyd tensile machine (Lloyds Instruments, UK) with the free end of the beam perpendicular to, and in line with, the load cell (100N). Each beam was pulled off the block with a peeling action by applying a tensile load using a small hook that engaged the hole on the free end of the beam at a crosshead speed of 1mm/min and the force at failure point was recorded. Statistical analysis for the TPS values was carried out using one-way analysis of variance and Tukey's pair wise comparisons. Regression analysis was undertaken to compare the TPS of luting cements with their known KIC values.

Results

The mean TPS values and the standard deviations (in Newton) are as follows: Super-Bond C&B 7.7 +/- 1.4, Panavia 6.1 +/- 1.3, GC Fuji plus 5.1 +/- 0.7, RelyX Luting 4.5 +/- 0.8, Dyract Cem 4.2 +/- 1.3, Ketac Cem 2.4 +/- 0.4. All the fractured surfaces of the tested samples were examined under a stereo zoom microscope. The mode of failure of all the tested cements was cohesive in nature. One-way analysis of variance (ANOVA) showed significant differences between the mean TPS of the cements ($P < 0.05$) (Table I). Tukey's pairwise comparisons showed that the mean TPS of Super-Bond was significantly greater than Panavia 21 as well as all other luting cements. The mean TPS of Dyract Cem, Fuji Plus and RelyX Luting were not significantly different. The mean TPS of Ketac Cem was significantly the lowest. The KIC values for those cements that are used in this study were obtained from literature and were measured after 24 hours and after 7 days.^(10,11) The mean KIC values and the standard deviation

Table I: One-way Analysis of Variance for Tensile peel strength of all tested cements (Twenty specimens for each type of cement)

Source	DF	SS	MS	F	P-value
Resin	5	319.90	63.98	55.96	0.000
Error	114	130.34	1.14		
Total	119	450.25			

Table II: The mean fracture toughness of the different luting cements with their standard deviation ($\text{MPa}\cdot\text{m}^{1/2}$)^(10,11)

Luting cements	KIC after 24 hour	KIC after 7 Days
Super-Bond C&B	1.42±0.2	1.07±0.1
Panavia 21	0.81±0.46	0.83±0.08
GC Fuji Plus	0.72±0.06	0.73±0.08
RelyX Luting	0.36±0.07	0.68±0.06
Dyract Cem	0.48±0.05	0.48±0.05
Ketac Cem	0.26±0.03	0.17±0.03

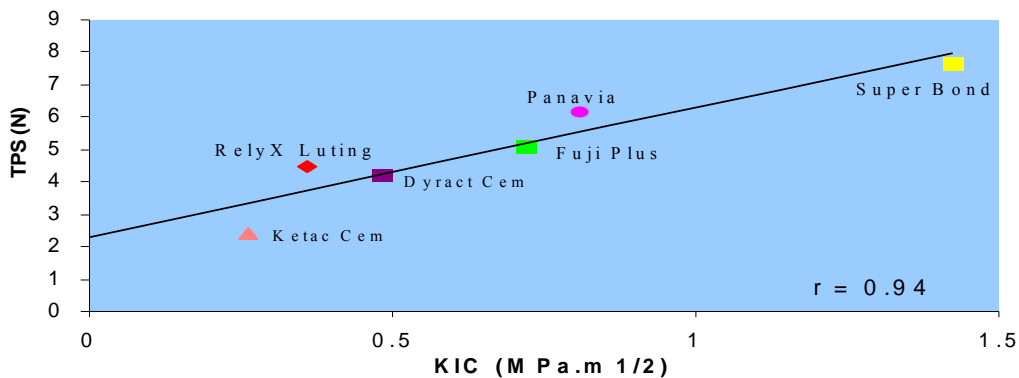


Fig. 1: The regression analysis between the mean fracture toughness (KIC) values after 24 hours and the tensile peel strength (TPS) showed a positive relationship between fracture toughness and tensile peel strength. Correlation coefficient for the linear regression $r = 0.94$

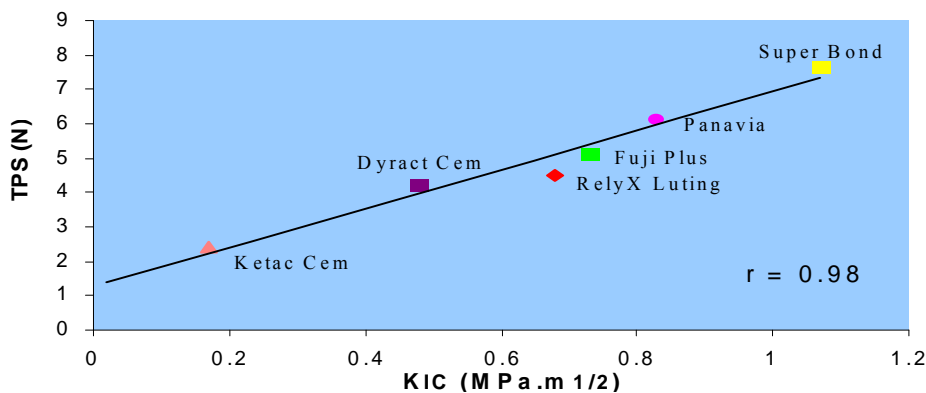


Fig. 2: The regression analysis between the mean fracture toughness (KIC) values after 7 days and the tensile peel strength (TPS) showed a positive relationship between fracture toughness and tensile peel strength. Correlation coefficient for the linear regression $r = 0.98$

($\text{MPa}\cdot\text{m}^{1/2}$) for those cements are summarised in Table II. The relationship between KIC and TPS shows a highly positive correlation. The linear regression analysis showed that the correlation coefficient (r) is equal to 0.94 after 24 hours and 0.98 after 7 days (Fig. 1 and 2). The correlation coefficient after 7 days is better than that after 24 hours.

Discussion

The TPS of different luting cements were measured in this study. The experimental apparatus used was similar to that used by Northeast *et al.*⁽⁶⁾ except that the thickness was the same for all the beams (0.5 mm) and the only variable was the luting cement. The design of TPS experimental apparatus is somewhat similar

to the design of RBB if we assume that the Ni-Cr beam acts as the retainer of RBB, the block as the tooth structure, and the pull out load as the load responsible for failure of RBB. Not all the tested cements are used for bonding RBB although all of them can bond well to the metal. This is to show that its not only the ability of the cement to bond to metal is the only requirement for bonding RBB, it must also be able to resist stresses generated in the cement layer and to resist propagation of cracks. All the KIC values were obtained from Knobloch *et al*⁽¹⁰⁾ except for Dyract Cem, which was obtained from a study by Ryan *et al*.⁽¹¹⁾ Using a wide variety of luting cements with different KIC values is better for studying the relationship between KIC and TPS. Although Knobloch *et al* and Ryan *et al* used different methods to measure the KIC (The mini-compact tension method and chevron notch short rod method respectively) the measured values for the same type of cement were close in both studies. The loads required to cause debonding in the TPS experimental apparatus are lower than those obtained from tensile bond strength tests.^(12,13) This observation is consistent with Northeast *et al*.⁽⁶⁾ The tensile bond strength of Super-Bond and Panavia bonded to Ni-Cr alloy was reported to be 28.5 MPa and 70 MPa respectively.⁽¹²⁾ With retainers having a surface area of 10mm², the tensile force required to cause debonding would have to be about 280 N - 700 N, and such high loads are unlikely to occur clinically, nevertheless, debonding is a common mode of failure of RBB.⁽³⁻⁵⁾ Another surprising observation is that the RBB most frequently fails at the resin-metal interface leaving a layer of resin on the enamel.^(4,5) This contrasts with the observation that the tensile bond strength of resin-metal is generally higher than that of resin-enamel.⁽¹⁴⁾ Therefore, the load required for bond failure to occur due to tensile peel stresses within the adhesive interface is potentially more clinically relevant than measurement of tensile or shear bond strengths.

The main disadvantage of the TPS test is that it is structural dependent; it depends on the thickness of the beam.⁽⁶⁾ That means the values of tensile peel strength for the luting cements used in this study will change if we change the thickness of the beams but the 0.5 mm beam

thickness is similar to the recommended retainer thickness used clinically.⁽¹⁵⁾

The mode of failure of all the studied cements was virtually identical, this was cohesive in nature. The crack initiation took place close to the cement-substrate where there are maximum tensile peel stresses as was shown by the finite element analysis done by Northeast *et al*.⁽⁶⁾ The fracture subsequently travelled close to the beam-cement interface leaving most of the cement on the substrate. This is similar to the clinical failure where most of the cement is left on the tooth surface after debonding.^(4,5) Since the fracture started from within the cement itself and not at the beam-cement interface, whether or not one material bonds better than another becomes irrelevant. This proves that it is the KIC of the cement itself that may play an important role in the adhesive bond quality of the RBB. The regression analysis of the relationship between KIC and TPS showed a positive linear relationship. That means the material with higher KIC will result in higher tensile peel forces to cause debonding compared with more brittle material assuming both of them bond well to the metal surface. The statistical regression analysis showed a better relationship between the TPS and the measured KIC values after 7 days (correlation coefficient = 0.98) than that after 24 hours (correlation coefficient = 0.94). This may be due to the effect of water as the KIC test was done after water storage in order to simulate the oral conditions.⁽¹⁰⁾ Water storage will plasticize the luting cement and delay the onset of fracture.⁽¹⁰⁾ Super-Bond and Panavia are adhesive resin cements that form chemical bonds with clean sand blasted base metal surfaces.^(16,17) The TPS and KIC of Super-Bond are significantly greater than that of Panavia. This may be due to differences in the chemical composition of those materials; Panavia 21 is Bis-GMA-based resin cement, which contains high volume fraction of inorganic fillers. While Super-Bond is an unfilled poly methyl methacrylate (PMMA) based resin cement that contains long flexible chains of high molecular weight, which tend to lead to higher KIC.⁽¹⁰⁾ Plastic deformation delays the onset of brittle fracture, resulting in higher KIC^(7,18) and so higher TPS. There was no significant difference between the TPS of Dyract cem, Fuji

Plus and RelyX Luting, which are compomer and resin-modified glass ionomer cements. This may be due to the relatively close chemical composition of those materials. Their KIC and TPS were significantly greater than that of conventional glass ionomer cements, which may be due to resinous components of those materials.⁽¹¹⁾ The KIC and TPS of Ketac Cem, which is conventional glass ionomer cement are significantly lower than all the other luting cements. Conventional glass ionomer cements are susceptible to dehydration and crazing during the initial setting reaction.^(10,19) The resultant microcracks would act to initiate and facilitate crack propagation within the cement matrix.^(10, 19) The TPS test is somewhat similar to the KIC test principle in that both of them measure the ability of the material to resist crack propagation. That's why it is not surprising to find relationship between KIC and TPS. The high KIC and TPS of the adhesive resin cements may explain their good clinical performance in RBB. On the other hand, glass ionomer cement showed unacceptable rate of debonding although it adheres well to tooth structure and metals.^(20,21) This could be due to their low KIC and TPS which may contraindicate their use in RBB. It is unclear how valuable compressive strength data are in selecting luting cement for RBB. The compressive strength of glass ionomer cement was shown to increase over several weeks to about 200 MPa.⁽²²⁾ Panavia has a very high compressive strength compared to Super-Bond, which exhibits too much plastic deformation to be tested in this way.⁽²³⁾ In contrast, Super-Bond has higher fracture toughness than Panavia.⁽¹⁰⁾ So there is a need to know which material property is responsible for the better clinical performance in order to help in material selection. The absolute value of KIC is a material property, which should be independent of the size and geometry of specimen and may be a more reliable parameter to predict clinical performance than the compressive or diametral tensile strength measurements.^(11,19) The compressive or tensile strength measurement might not provide accurate information on the likely performance of the material in clinical use, due to influence of the specimen geometry and flaws introduced during manufacture on the values obtained.⁽²⁴⁾ This does not mean that the KIC of the luting cement is the

only property that is needed to enhance the outcome of the RBB. Other mechanical properties of the luting cement are still important. We still need a material with a high diametral tensile and compressive strength in order to resist stresses within the adhesive layer and tolerate the masticatory forces. Elastic modulus is also important to prevent microleakage.⁽²⁵⁾ It has been suggested that luting cement with an elastic modulus in the intermediate range between that of tooth structure and the indirect restorative material is desirable because this can reduce interfacial stress concentrations without causing excessive strains.⁽²⁵⁾

Recommendation

To enhance the clinical outcome of the RBB it is suggested to use structural adhesive cement; the cement must not only be able to bond well to the metal surface, it must also be able to resist stresses generated in the cement layer and to resist propagation of cracks.

Conclusion

The results of this study showed a direct relationship between fracture toughness and tensile peel strength. The fracture toughness of the cement is the mechanical property that could help in material selection i.e. the material with a higher KIC may predict a higher clinical performance of RBB compared to more brittle material assuming both are able to produce an adequate and durable bond to the metal.

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