

Comparison of the Flexural Strength of Two Dual Cure Adhesive Resin Cements Under Oral Simulated Conditions: An *In-Vitro* Study

P.Awasthi*, A. Nair†, K.M. Regish†, M.Viswambaran and M. Kumar

Abstract - The purpose of this *in vitro* study was to evaluate the flexural strength of the newly developed self-adhesive dual cure resin cement and compare it with conventional resin cement under oral simulated conditions. A conventional resin cement (Calibra®) and self adhesive resin cement (RelyX™ U100) were selected and 40 specimens of each cement were fabricated for the study. Half of these specimens were polymerized directly whereas the other half were polymerized through 2mm of porcelain disc. Specimens were tested after 24hrs and after 30 days immersion in artificial saliva. A three point bending test was performed using universal testing machine at a crosshead speed of 1mm/min. Overall RelyX™ U100 showed higher mean flexural strength compared to Calibra® (141.55 MPa, 119.46 MPa, respectively). When the specimens of both the cements were light cured through 2 mm porcelain disc, their flexural strength decreased significantly. The mean flexural strength of both self adhesive and conventional dual cure adhesive resin cements was increased significantly after storage in artificial saliva for 30 days at 37°C. Among the two dual cure resin cements, the self adhesive dual cure cement (RelyX™ U100) showed increased overall mean flexural strength as compared to conventional resin cement (Calibra®) under all the curing and storage protocols.

KEY WORDS: Flexural strength, adhesive resin cement and dual cure cement

INTRODUCTION

The practice of fixed prosthodontics has changed dramatically with the introduction of innovative dental materials and techniques. The development of aesthetic dentistry has led to numerous applications for adhesive resin cements. They are based on resin composite technology and have been used for retaining crowns on short tapered preparations lacking optimal retention and resistance form and luting resin bonded fixed partial dentures. Wide variations in physical and mechanical properties are found among the commercially available adhesive resin cement products¹. Adequate polymerization is a crucial factor for obtaining optimal mechanical properties and satisfactory clinical performance of dental resin material. When compared to chemically polymerizing and light polymerizing materials, dual-cured resin luting agents were expected to provide further polymerization by chemical catalysts in deeper areas or under thicker restorations. However, many studies have suggested that, even under dual-curing mode, degree of conversion and final hardness of most dual-cured resin luting agents were reduced when the thickness of restorative materials were increased². Recently, a new group of dual cure resin cement, the self-etching, self-adhesive resin cements, have been introduced (e.g. RelyX™ Unicem and RelyX™ U100 3M ESPE; St. Paul, MN, USA). Literature concerning mechanical and physical properties of this group such as the flexural strength, curing efficiency, ultimate hardness, fracture toughness, wear resistance, solubility and hydrolytic degradation is limited.

The purpose of this *in vitro* study was to evaluate the flexural strength of the newly developed self-adhesive dual cure resin cement and compare it with conventional resin cement under oral simulated conditions which were mimicked by curing half of the specimens through 2mm porcelain disc and by storing the specimens in artificial saliva.

MATERIALS AND METHOD

Materials selected for the study represented two different dual cure adhesive resin cements; one of them being a conventional dual cure adhesive resin cement (Calibra®, Dentsply Caulk, Milford DE) and the other being the newly introduced self etching, self adhesive, dual cure adhesive resin cement (RelyX™ U100, 3M ESPE; St. Paul, MN, USA). 40 specimens for each cement were fabricated and designated as Group I and Group II respectively. Each group was subdivided into 4 subgroups of 10 specimens each and were named from 'a' to 'd' according to different curing and storage protocols as shown in (Table 1). In total, 25 x 2 x 2 mm rectangular bar specimens were fabricated using a custom made glass plate mould (Fig. 1). To prevent scattering of light beyond the edges of the porcelain disc, its shape was modified to that of a crown which fitted snugly on the light tip as half of the specimens were polymerized through 2 mm porcelain disc for simulating clinical conditions.

Prior to specimen fabrication the glass mould was sprayed with a separating agent for ease of specimen removal. The equal lengths of base and catalyst paste of both the adhesive resin cements were dispensed, weighed to en-

* BDS, MDS

† MDS

Table 1. SPECIMEN GROUPS

| <i>Group I - Conventional resin cement (Calibra®)</i> | | |
|---|------------------------|---------------------------------------|
| Groups | Curing Protocol | Storage Protocol |
| Group I (a) | Without Porcelain Disc | Artificial saliva for 24 hrs at 37°C |
| Group I (b) | With Porcelain Disc | Artificial saliva for 24 hrs at 37°C |
| Group I (c) | Without Porcelain Disc | Artificial saliva for 30 days at 37°C |
| Group I (d) | With Porcelain Disc | Artificial saliva for 30 days at 37°C |
| <i>Group II - Self adhesive cement (RehyXTM U100)</i> | | |
| Groups | Curing Protocol | Storage Protocol |
| Group II (a) | Without Porcelain Disc | Artificial saliva for 24 hrs at 37°C |
| Group II (b) | With Porcelain Disc | Artificial saliva for 24 hrs at 37°C |
| Group II (c) | Without Porcelain Disc | Artificial saliva for 30 days at 37°C |
| Group II (d) | With Porcelain Disc | Artificial saliva for 30 days at 37°C |

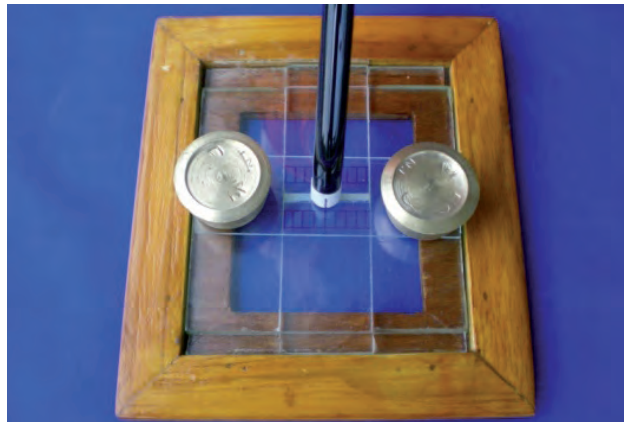


Figure 1. Custom made glass plate mould with overlapping curing procedure



Figure 2. Dimensions of the Specimen (25 x 2 x 2 mm)

sure equal volumes and mixed following manufacturer's recommendations. Using plastic filling instrument, the mix was loaded in the rectangular portion of mould (25 x 2 x 2 mm). Care was taken to avoid incorporation of any air bubbles while loading the material. The mix was then covered with a 2 mm glass slide. The cement specimen was then photo activated using light cure unit with an intensity of 550 mW/cm² in nine overlapping section for 20 seconds per section on both the sides either directly or through porcelain disc according to the designated group (Fig. 1). The specimen was retrieved from the mould and any excess material was carefully removed with composite finishing discs (Soflex™Extra Thin, 3M). The dimensions of the specimen (25 x 2 x 2 mm) were verified using digital

vernier caliper (Fig. 2). A mark was made at the end of the specimen using a permanent marker to indicate the top for subsequent reorientation during testing of specimens. Following the same protocol, 40 specimens were made for each cement and then divided into subgroups as previously described.

Custom made three-point bending platform was fabricated which acted as the base of the three point bending device. It consisted of a metal plate with two metal round bars of 2 mm diameter placed 20 mm apart. The stored specimens were removed from artificial saliva and tested wet after blotting dry. A reference dot which was marked previously on the specimen at the time of fabrication was used to orient

the specimen on the bending device. A compressive load was applied to the specimen at a cross head speed of 1 mm/min using 50 kg load cell (Fig. 3). The bending data was recorded as load to fracture.

The flexural strength was calculated from the fracture load and specimen dimensions according to the following formula:

$$\text{Stress} = \frac{3 \times \text{fracture load} \times \text{length}}{2 \times \text{width} \times \text{thickness}^2}$$

The results were obtained in Kgf/cm² and then converted to megapascals (MPa) for statistical analysis.

MINITAB-16 software was used for statistical analysis with Windows 7 operating system. Three-way analysis of variance (ANOVA) followed by two-way analysis of variance (ANOVA) were carried out to determine whether there existed any significant differences in the mean flexural strength between the two dual cure adhesive resin cements,

under different curing and storage protocol. Finally one-way ANOVA was carried out to compare the two cements when polymerized with disc and stored for 24 hours and 30 days in artificial saliva.

RESULTS

The highest mean flexural strength was obtained for RelyXTMU100 when polymerized without discs and stored for 30 days in artificial saliva (159 MPa). The least mean flexural strength was obtained for Calibra[®] with disc and stored for 24 hours in artificial saliva (89 MPa) (Figure-4). three-way ANOVA results (Table – 2) showed that there were significant differences (p < 0.0001) in the mean flexural strength between the two levels of each protocol. Hence, two-way ANOVA was carried out for curing protocol and storage protocol for each cement separately. The two-way ANOVA results for Calibra[®] (Table – 3) showed that the difference between the mean flexural strength between the two levels of curing protocol as well as between the two levels of storage protocol factor were highly significant corroborating the result of three-way ANOVA. A two-way ANOVA was also carried out for RelyXTMU100 (Table – 3). The results were identical to the results showed by Calibra[®]. The results of two levels of curing protocols differed between groups with highly significant values (F = 86.58 and p < 0.0001). The differences in the mean flexural strength of two levels of storage protocol factor were also significant (F = 1470.64, p < 0.0001). Incidentally, the interaction effect of curing protocol factor and storage protocol factor was statistically significant in terms of RelyXTMU100 (F = 18.69 and p < 0.0001). Hence, one-way ANOVA was conducted to find out, if there existed any significant difference between the mean flexural strength of two dual cure adhesive resin cements when polymerized with disc for two levels of storage protocol and results presented in (Table – 4). The mean flexural strength of RelyXTMU100 (with disc) was statistically higher when compared to the same quantity in respect of Calibra[®] (F = 92.13, p < 0.0001). The test of

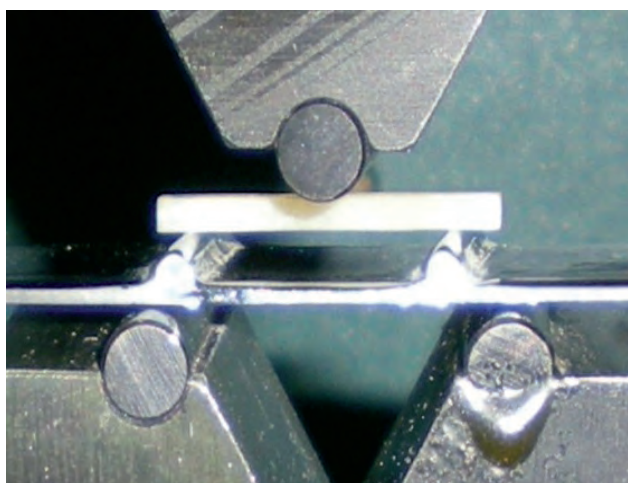


Figure 3. Testing the Flexural Strength Using Universal Testing Machine

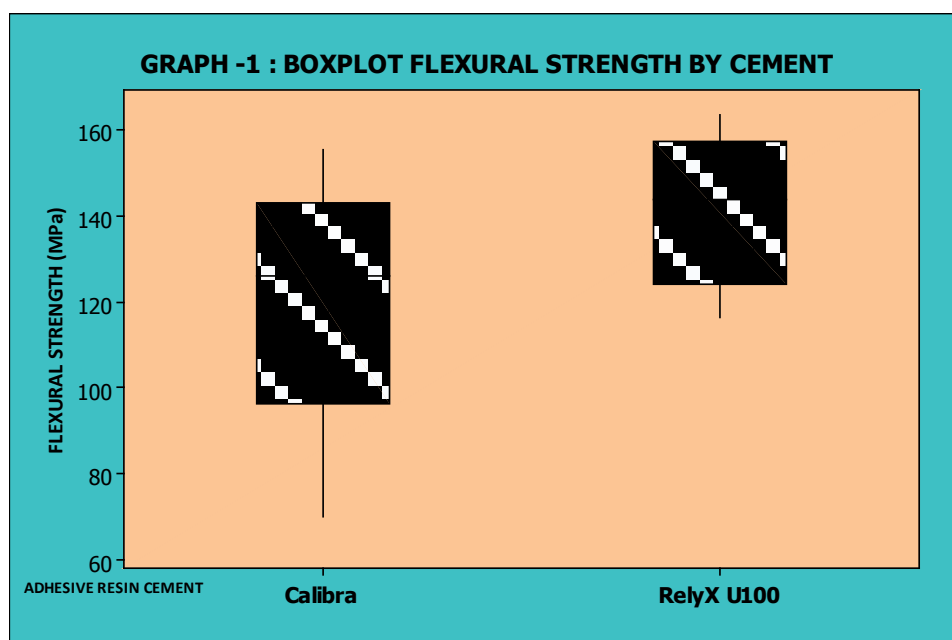


Figure 4. Boxplot Flexural Strength By Cement

Table 2. THREE - FACTOR ANOVA FOR FLEXURAL STRENGTH (MPa)

| Source | DF | SS | MS | F | P |
|--|----|---------|---------|--------|--------|
| Cement | 1 | 9763.2 | 9763.2 | 249.09 | 0.0001 |
| Curing protocol | 2 | 1801.3 | 1801.3 | 45.96 | 0.0001 |
| Storage protocol | 1 | 30093.4 | 30093.4 | 767.78 | 0.0001 |
| Cement* Curing protocol | 1 | 64.5 | 64.5 | 1.65 | 0.2040 |
| Cement * Storage protocol | 1 | 1002.6 | 1002.6 | 25.58 | 0.0001 |
| Curing protocol * Storage protocol | 1 | 206.6 | 206.6 | 5.27 | 0.0250 |
| Cement* Curing protocol * Storage protocol | 1 | 2.6 | 2.6 | 0.07 | 0.7980 |
| Error | 72 | 2822.1 | 39.2 | | |
| Total | 79 | 45756.3 | | | |

Table 3. TWO-WAY ANOVA FOR FLEXURAL STRENGTH (MPa)

TWO-WAY ANOVA FOR FLEXURAL STRENGTH (MPa): CALIBRA®

| Source | DF | SS | MS | F | P |
|------------------------------------|----|---------|---------|--------|--------|
| Curing protocol | 1 | 1273.9 | 1273.9 | 17.80 | 0.0001 |
| Storage protocol | 1 | 21040.9 | 21040.9 | 294.06 | 0.0001 |
| Curing protocol * Storage protocol | 1 | 81.4 | 81.4 | 1.14 | 0.2930 |
| Error | 36 | 2575.9 | 71.6 | | |
| Total | 39 | 24972.1 | | | |

TWO-WAY ANOVA FOR FLEXURAL STRENGTH (MPa): RelyX™U100

| | | | | | |
|------------------------------------|----|---------|---------|---------|--------|
| Curing protocol | 1 | 592.0 | 592.0 | 86.58 | 0.0001 |
| Storage protocol | 1 | 10055.1 | 10055.1 | 1470.64 | 0.0001 |
| Curing protocol * Storage protocol | 1 | 127.8 | 127.8 | 18.69 | 0.0001 |
| Error | 36 | 246.1 | 6.8 | | |
| Total | 39 | 11021.0 | | | |

Table 4. ONE - WAY ANOVA

CALIBRA® - WITH DISC - 24 HOURS, RELYX™U100 - WITH DISC - 24 HOURS

| Source | DF | SS | MS | F | P |
|--------|----|--------|--------|-------|--------|
| Factor | 1 | 4685.0 | 4685.0 | 92.13 | 0.0001 |
| Error | 18 | 915.3 | 50.9 | | |
| Total | 19 | 5600.3 | | | |

CALIBRA® - WITH DISC - 30 DAYS, RELYX™U100- WITH DISC - 30 DAYS

| | | | | | |
|--------|----|---------|---------|--------|--------|
| Factor | 1 | 1474.18 | 1474.18 | 170.76 | 0.0001 |
| Error | 18 | 155.39 | 8.63 | | |
| Total | 19 | 1629.57 | | | |

significance through one-way ANOVA (Table – 4) confirms the superiority of RelyX™U100 in comparison with Calibra® in respect of mean flexural strength in all situations (F = 170.76, p < 0.0001).

DISCUSSION

The development of aesthetic dentistry has led to numerous applications for adhesive resin cements. Resin cement was introduced in 1980s, in order to overcome drawbacks of low early strength, moisture sensitivity and non adhesion of restorative materials to tooth structure. Resin cements are increasingly used for luting all-ceramic, metal or composite

indirect restorations. Resin cements have been divided into two subgroups according to the adhesive system used to prepare the tooth prior to cementation. One group utilizes conventional etch and rinse adhesive systems (e.g. Variolink and Variolink II, Calibra and Nexus). In the other group, enamel and dentin are prepared using self-etching primers (e.g. Panavia 21, Panavia F and Panavia F 2.0 [Kuraray Medical; Tokyo, Japan]; Multilink [Ivoclar Vivadent]).

Recently in an attempt to simplify procedures, a new subgroup of resin cements, the self-etching, self-adhesive resin cements, have been introduced (e.g. RelyX™Unicem and RelyX™U100 3M ESPE; St. Paul, MN, USA). These materials were designed with the intent to overcome some of

the shortcomings of both conventional (zinc phosphate, polycarboxylate, and glass-ionomer cements) and resin cements, as well as to bring the favourable characteristics of different cement classes into a single product. According to their manufacturers, these products are self-adhesive, including acidic and hydrophilic monomers in their composition, which simultaneously demineralise and infiltrate enamel and dentin, resulting in strong bonding³. Literature concerning other mechanical and physical properties of self-etching, self-adhesive resin cements, such as the flexural strength, curing efficiency, ultimate hardness, fracture toughness, wear resistance, solubility and hydrolytic degradation is limited.

Among the two cements evaluated in the present study, mean flexural strength of newly introduced self adhesive dual cure resin cement (Rely X™U100) was 141MPa which was found to be superior to the mean flexural strength of conventional dual cure resin cement (Calibra®) which was 119 MPa under all protocols tested. The results obtained for Calibra® are in accordance with the study carried out by Pace et al and Lu et al^{4,5}. The results obtained in the present study for flexural strength of self etch resin cements are in agreement to the result obtained by Saskalaukaite et al⁶. In the present study when the specimens of both the cements were light polymerized through the 2 mm porcelain disc, their flexural strength decreased significantly when compared to polymerizing without the disc. Mean flexural strength of self adhesive cement decreased from 131 MPa to 120 MPa and conventional resin cement decreased from 103 MPa to 89 MPa when polymerized through the 2 mm porcelain disc and tested after 24 hours. Similarly a reduction in the mean flexural strength was evident among the groups polymerized through the porcelain disc when tested after 30 days which corroborated the findings of Meng et al which showed that dual-cured resin luting agents with direct irradiation produced the best mechanical properties and polymer structure⁷. This may be because of the light intensity reaching the cement drastically reduces when light is transmitted through a ceramic or composite restoration. The mean flexural strength of both self adhesive and conventional dual cure adhesive resin cements was increased significantly after storage in artificial saliva for 30 days at 37°C.

Within the limitations of the present study, it may be concluded that the overall mean flexural strength of both conventional and self adhesive dual cure resin cements were within the clinically acceptable range under all the test conditions. Among the two dual cure resin cements, the self adhesive dual cure cement showed better overall mean flexural strength as compared to conventional resin cement. Both the cements when cured through porcelain disc showed reduction in mean flexural strength. When comparing the cements after 24 hours storage in artificial saliva, self adhesive dual cure resin cements showed better mean flexural strength as compared to conventional resin cement. However, after ageing the specimens in artificial saliva for 30 days, both cements showed an increase in mean flexural strength, but self adhesive dual cure resin cements showed better mean flexural strength as compared to conventional resin cement.

It was also seen that there was decrease in flexural strength when the samples were polymerized through the 2 mm porcelain disc both at 24 hours and 30 days when the irradiation time recommended by manufacturer was fol-

lowed for curing the specimens. It may be suggested that clinically to compensate for this decrease in the flexural strength of dual cure resin cements when irradiated through porcelain restoration, the duration of the light exposure should be increased from that which is recommended by the manufacturers; as has been suggested in various studies^{8,9,10,11}. The results of this study need to be verified by comparing more number of cements with a larger sample size and by in-vivo studies.

CONCLUSION

Within the limitations of the present study, it was concluded that:

Both the cements when polymerized through porcelain disc showed reduction in mean flexural strength.

When comparing the cements after 24 hours storage in artificial saliva, the self adhesive dual cure resin cement showed increased mean flexural strength compared with the conventional resin cement

However, after ageing the specimens in artificial saliva for 30 days, both cements showed an increase in mean flexural strength, but, self adhesive dual cure resin cements showed better mean flexural strength as compared to conventional resin cement.

ADDRESS FOR CORRESPONDENCE

Dr. Anoop Nair, Senior Lecturer, Dept. Of Prosthodontics, Govt. Dental College and Research Institute, Bangalore, Victoria Hospital Campus, Fort, Bangalore, India. 560002. Email: Dranoopnair@Yahoo.Com

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