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## SHEAR BOND STRENGTH AND FAILURE PATTERN OF TWO SELF-ADHESIVE RESIN CEMENTS TO HYBRID CERAMIC UNDER DIFFERENT MODES OF POLYMERIZATION

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#### ABSTRACT

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**Abstract:** The purpose of this study was to evaluate the shear bond strength of two selfadhesive cements with different polymerization modes. cemented to a Hybrid-Ceramic CAD/CAM blocks (VITA ENAMIC® for CEREC®/ inLab®, VITA Zahnfabrik H. Rauter GmbH & Co.KG., Germany),

**Methods**: Two self-adhesive resin cements: Rely X U200 (RX, 3M ESPE,St. Paul, MN, USA) and G-CEM Link ACE (GC, GC corporation, Tokyo, Japan). RX and GC groups with different polymerization modes (photo-curing (P) and self-curing (S)), were used in this study on hyprid ceramic Vita Enamic CAD/CAM blocks. Shear bond strength (SBS) tests were performed on all the specimens.

**Results:** The highest SBS was recorded with RX S group whilst the lowest SBS values were recorded with the GC *S* groups. Based on Tukey's post hoc significance analysis test, significance differences were observed between the RX S group compared to the GC *S* group (p < 0.05). No significance differences were found between data obtained from different curing modes of neither, RX *S* and RX *P* nor GC *S* and GC P. Adhesive pattern of failure was predominantly for most of the groups except RX *S* that was presented as predominant cohesive form of failure with the CAD/CAM hybrid ceramic block.

**Conclusion:** Within the limitations of this in vitro study, polymerization modes had no significance, but more mixed failure patterns were observed when using the photo-curing mode than when using the self-curing mode.

Keywords: self-adhesive resin cement; polymerization mode; failure mode, hybrid ceramic.

#### **INTRODUCTION**

Aesthetic indirect restorative materials bonded with resin cement are used to simulate the natural tooth translucency and color, mask tooth discoloration, provide a the desirable aesthetic outcome , and durable long-term retention of the prosthesis <sup>(1)</sup>.

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Resin cements are classified according to their setting reaction into chemical, photo, or dualcured polymerization reaction. Resin cements with chemical polymerization reaction exhibit a shorter working time, but their completeness of polymerization is not affected by the thickness of the adherent restorative material. Photo-initiated polymerization reaction resin cements present a more favorable working characteristics with an adequate amount of light <sup>(2)</sup>. The photo-initiated polymerization of a cement may be affected by light intensity emitted by the light source, wavelengths emitted beyond the spectrum range of the photo-initiator within the resin material, and the thickness of the adherent indirect restoration<sup>(3)</sup>. Combining the desirable properties of chemical polymerization with those of light curing to assure enough working time and adequate polymerization, or degree of conversion, even in the deepest area of the preparation was presented with the developed dual cured resin cement  $^{(4, 5)}$ .

Optimizing the mechanical properties and adhesive capacity of the resin cement is dependent the completeness of monomers conversion into polymers during the polymerization reaction. Most of the available polymers exhibit a varying amount of residual monomer with degrees of conversion ranging from 55% to 75% with conventional irradiation conditions. Residual monomers present adverse consequences and may result in unfavorable biological tissues effects, mainly in the pulp and increase the incidence of marginal leakage and development of secondary caries because of insufficient resin cement polymerization <sup>(5-6)</sup> Inadequate polymerization of the resin cement as a result of Inadequate required light quality may compromises its physical properties by interfering with its resistance, hardness, water absorption, adhesion ability, and color stability.

Color, thickness and type of the adherent restorative material and tooth structure through which the emitted light passes, the time length of light exposure, may influence the amount of light that is perceived by the resin cement, compromising the degree of conversion and their mechanical properties also the Dual-cured resin cement degree of polymerization, is affected by the amount of emitted light, since polymerization is not purely initiated by the chemical component of the reaction <sup>(7-10)</sup>.

Hybrid ceramics are based on the concept of combining the positive characteristics of ceramics and composites <sup>(11)</sup>.

VITA ENAMIC is a hybrid dental ceramic material with a dual-network structure. The dominant ceramic network is strengthened by a polymer network, with both networks fully integrated with one another, therefore combining the positive characteristics of a ceramic and a composite. This new material is highly heat cured through a controlled, proprietary manufacturing process, which eliminates the need for a firing step after milling. The material is easily machined chairside or in a dental lab, polishes quickly to an esthetic finish and if necessary, can be further adapted using light-cure restoratives.<sup>(12-14)</sup>

It is well known that the composition of resin cements is the same as resin composite but with lesser filler content, the manufacturer of VITA ENAMIC stated that it can be corrected intraorally with resin composite so examining the bond strength of resin cement to hybrid ceramics is of great importance not only for achieving a better retention of restorations but also for understanding the intra oral repair of these materials.

Therefore, the aim of this in vitro study was to evaluate the impact of polymerization mode on the shear bond strength of two self-adhesive resin cements to hybrid ceramic.

#### MATERIALS AND METHODS

#### **Samples preparation**

A Total number of 24 blocks of VITA ENAMIC were included in the study, 3 blocks with equal thickness of 3mm were cut from each Vita ENAMIC blocks using a low-speed diamond saw under copious water cooling. Blocks were mounted in ring holders made of self-cured acrylic resin. The uppermost surface of all samples was etched using hydrofluoric acid (VITA CERAMICS ETCH) for 60 seconds washed and dried clean then salinized subsequently according the manufacturer recommendations.

The samples were then divided into two main groups (A and B groups) representing the selfadhesive cement type, each counting 36 samples. Each group were further subdivided into two subgroups (As, Ap, Bs and Bp) representing the polymerization reaction mode, each counting 18 sample (self or photopolymerization).

TABLE	(1)	Samples	distribution	(grouping	and
	sul	bgrouping	)		

Samples distribution (grouping and subgrouping)								
Total number of sample. (72 sample)								
Group A (H	Rely x resin	Group B (G-CEM resin						
cem	ent)	cement)						
36 sample		36 sample						
Group As	Group Ap	Group Bs	Group Bp					
18 sample	18 sample	18 sample	18 sample					
Self-cured	Photo-cured	Self-cured	Photo-cured					
(RX S)	(RX P)	(GC S)	(GC P)					

#### **Cement application**

Transparent polyurethane tubes with an internal diameter of 2.4mm (so the surface area of adhesion is calculated from the equation  $\pi r^2 = 3.14 \times 1.4 = 4.4 \text{mm}^2$ , r is half the diameter) were fitted on the pre-cutted CAD/CAM Blocks) using wax,

cement was mixed according to the manufacturer's recommendation and applied on the pretreated VITA ENAMIC surface thought the pre-fitted polyurethane tubes placed into the tube.<sup>(15)</sup>

Samples of group (Ap and Bp) were light-cured for 20 s with 1200 mW LED light-curing unit, while samples of groups (As and Bs) were left for 1 h at room temperature in darkness for self-cure. Before testing, all the specimens were kept in a 100% humid environment at room temperature for 24 h after cementation.<sup>(16)</sup>

Samples were mounted and jigged to a universal testing machine (Fig.1). Shear force was applied to the adhesive interface until fracture at a crosshead speed of 0.5 mm/min. The force (N) at which the bond failed was recorded and divided by the surface area of adhesion between resin cement and the block.



Fig. (1)

The shear bond strength (SBS) was calculated in MPa. Data were analyzed using one-way analysis of variance (ANOVA) with Tukey's post hoc test.

Hybrid ceramic sample deboned surface were examined for mode of failure under digital stereo microscope at magnification (80X), the type of failure was considered as "cohesive failure in resin cement or the CAD/CAM block" and "adhesive failure at the interface cement - hybrid ceramic" and "mixed failure".

#### RESULTS

The mean shear bond strength and standard deviations (SD) are mentioned in Table 2. The Relay X S group has recorded the highest shear bond strength, while the G-Cem S group has recorded the lowest Shear bond strength. Based on Tukey's post hoc significance analysis test, significance differences was observed between the Relay X S group compared to the G-Cem S group (p < 0.05). No significance differences were found between data obtained from different curing modes of neither, RX S and RX P nor GC S and GC.

TABLE (2) Shear bond strength mean values and standard deviations.

Group	RX S	RX P	GC S	GC P
SBS ± SD Mpa	10.9± 4.9	9.5±4.3	6.5±2.8	9.5±4.5

Fig. (2) Cohesive failure



Fig. (3) Mixed failure

# Microscopic analysis of the bonding joint failure pattern

Adhesive failure was the predominant pattern along the adhesive joint between the resin cement-hybrid ceramic interface. A predominant cohesive mode of failure in the CAD/CAM block was noted with Relay X S group (Figure. 2). While a mixed form of failure was observed for the Relay X P and G-Cem P groups, at the block-cement interface, resin remnants were seen on the failed bonding interface. (Figure.3). The GC S group has shown an adhesive mode of failure with complete separation of the resin cement at the bonding interface with the hybrid /ceramic blocks (Figure.4).



Fig. (4) Adhesive failure

#### DISCUSSION

The VITA-ENAMIC (Hyprid- Ceram) presents a three-dimensionally interconnected pre-sintered ceramic network of 86 vol% that is infiltrated with a monomer mixture; thus, intertwined networks of polymers and ceramics are created. Clinical reliability of the material adhesion of the restoration is very important. However, information about bonding protocols to this new composite and polymerinfiltrated ceramic materials is still scarce<sup>(17)</sup>.

The results of this study showed that the RX exhibited significantly higher shear bond strength than GC-cem when self-cured, without any significant difference between the shear bond strength recorded for both self-adhesive resin cements when cured by photo-curing mode. This could be attributed to the lower viscosity of RX as a result of lower filler content <sup>(18)</sup>.

Viscosity changes during the autopolymerization reaction possibility has reduced radicals migration and continuing the conversion reaction process ability<sup>(19)</sup>, also the self-curing mode slow polymerization could have been impaired by water produced during the neutralization reaction of the phosphate monomers with basic filler within the self-adhesive resin cement<sup>(20)</sup>.

The low shear bond strength recorded for the self cured group of G-CEM than that of the light-cured group with no significant differences (p > 0.05) are in agreement with previous studies evaluated the flexural strength, hardness and the degree of conversion of dual-cured resin cements and stated that self-curing components of this type of resin cements were not enough for compensating for light intensity attenuation or its total absence and the self-curing mode of resin cements is less effective than the dual-cured or photo-cured cements <sup>(21-24)</sup>.

The results of this study also agrees with Suk Shim et al. (25) who concluded that self-chemical polymerization is not sufficient to cause the resin cement to achieve the highest polymerization in both early and late stage of polymerization.

However, this result is not agreed with the manufacturer data who stated that they used a new innovative chemical initiator system offering the highest polymerization in the self-curing mode. They claimed that it polymerizes within four minutes in self-curing mode and provides the maximum bond strength after 20 min.

In contrary to GC-CEM, the recorded shear bond strength of the self-cured group of Rely X U200 was slightly higher than that of the light-cured group but also with no significant differences (p > 0.05), this could be attributed to the deleterious consequences of polymer stiffens resulting from immediate light application, limiting the processes of chemical activation during polymerization preventing the cement from achieving its maximum degree of conversion and compromising its mechanical properties as stated by Manso AP etal <sup>(26)</sup>.

The light-activation delay to allow more time for the chemical activation to occur before lightactivation seems to affect the dual-cured resin cement properties depending on the material's composition and the time interval between the mixture until light-activation. Light-activation delay of 5 min, was suggested as being advantageous to reduce shrinkage stress during polymerization, without impairing the degree of conversion, the extent of polymerization and hardness of some dual-cured cements<sup>(27-29)</sup>.

It was also demonstrated that a 6-minute delay after the manipulation of resin cements may result in increased color stability<sup>(30)</sup>, but a 10-minute delay may be harmful <sup>(31)</sup>

Most of the groups except RX S revealed adhesive failure patterns along the resin cementhyprid ceram block interface. The RX S group showed a predominant cohesive failure pattern in its hyprid- ceram (50%). These results are indicative of more efficient interfacial polymerization. while comparing RX S with RX P and GC S with GC P, a higher percentage of mixed failure was observed in the RX P and GC P groups. In microscopic observation, the RX S group showed a predominant cohesive failure pattern in the hybrid ceramic, that goes with the statement of Toledano et al. who reported that cohesive and mixed failure patterns are desirable in a clinical situation when analyzing the fracture patterns of specimens<sup>(32)</sup>.

The RX P and G-Cem P group of samples has presented an adhesive pattern of failure along the block-cement interface with remnants of resin cements. While samples of the GC S group, complete separation of the luting cement from the CAD/ CAM blocks was observed. This variation in failure pattern observed in this study could be attributed to the analysis of Fischer et al. who reported that bonding strength can be influenced by the internal strength of the cements and the strength between the adhered materials and cements<sup>(33)</sup>.

Also in Accordance with Asmussen et al., who stated that the physical property of the resin cements can be influenced by the type of monomer, filler content, structure, and degree of conversion<sup>(34)</sup>.

#### **CONCLUSION:**

Within the limitations of this in vitro study, polymerization modes had no significance, but more mixed failure patterns were observed when using the photo-curing mode than when using the self-curing mode.

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