Degree of Monomer conversion for different resinous materials used to lute ceramic laminate veneers (in vitro study)

Yasmeen A. Fahmy[1], Ghada A. Abdelsattar[2], Maged M. Zohdy[3]

Abstract:

Objective: Mechanical properties enhancement of luting materials Used for Ceramic Laminate Veneers Cementation may affect the performance and the durability of esthetic restorations. The present study was performed to evaluate the Degree of Monomer Conversion of three different light polymerized resinous materials. **Materials and Methods:** Forty five ceramic slices of shade A2 were machined from low translucenct (LT) IPS Emax CAD blocks (n=45)using (Isomet 4000) sawing machine cutting at a speed of 2500 rpm with water cooling**,** the block were sliced to a uniform standard thickness of 0.5 mm. IPS Emax CAD slices were covering three resinous materials, RelyX Veneer, filtek Z350xt flowable composite and preheated filtek Z350xt composite at 68°C, cured for 30 seconds and then separated from the slices, the 45 specimens were then divided according to the used luting material into three groups (n=15), the samples then prepared for analysis by Fourier Transform Infrared Spectroscope, uncured cement samples were also subjected to analysis. One-way ANOVA followed by Tukey's post hoc test was used to study the effect of resinous material on degree of conversion.The significance level was set at $P<0.05$. **Results:** The highest mean value of DC was recorded for Rely X Veneer resin cement, followed by flowable composite, while the least DC was recorded for the preheated composite. **Conclusion:** Both light cured resin cement and flowable composite were superior to preheated composite regarding their degree of monomer conversion.

Introduction

Using artificial materials to mimic natural appearance is one of the major challenges in modern dentistry[1]. Ceramic restoration optical behavior is generally determined by the combination of the color of underneath tooth structure, the thickness of the ceramic material and the color of the resinous material used for cementation[2]. The material of choice to lute ceramic veneers is Light cured resin cement.

A great advantage of light cured cements over dual cured and chemical cured is its controllable working time, so the ability of the clinician to remove excess cement before curing and the finishing time required is decreased[3] also they have superior color stability compared to the other cured types[4].

In order to benefit from the physical properties of light activated composite resins, as well as an improved cost benefit compared to resin cements, some practitioners have been using preheated composite and flowable resin composites for the cementation of ceramic veneers.[5,6] Flowable composites have a decreased filler concentration compared with restorative composites, which allows for an increase in the surrounding resin matrix making it a suitable alternative luting agent when bonding laminate veneers.[7]

Blalock et al.,[8] found that preheating composite resin did yield higher flow rates than those composite resins at room temperature. However, Daronch et al. stated that decreasing shelf life and requiring quick work are the drawbacks of composite preheating.[9]

[1]*Postgraduate MSc student, Department of Fixed Prosthodontics, Faculty of Dentistry, Ain Shams University, Cairo, Egypt.* Yasmeenalsaeed2014@gmail.com
[2]*Assistant Professor of Fixed Prosthodontics Department, Faculty of Dentistry, Ain Shams University, Cairo, Egypt.*
[3]*Assistant Professor of Fixed Prosthodontics Department, Faculty of Dentistry, Ain Shams University, Cairo, Egypt.*

Also, pulp compatibility is of concern when composite resins are preheated to 54-68°C. Nevertheless, studies have found that pulp temperature is raised only by 0.8°C after placement of a 60°C preheated composite resin while 20s of light curing increases pulp temperature by 5°C.[10]

The term "Degree of Conversion" (DC%), can be defined as the monomeric carbon-carbon double bonds conversion into polymeric carbon-carbon single bonds, the importance of increasing the conversion and thus increased cross-linkage results in improvement in properties as higher surface hardness, flexural strength, fracture toughness, and tensile strength.[11]

Low degree of conversion can affect mechanical properties of the cement and results in increase in solubility, water sorption and subsequently affect its color stability[12]. The thickness and shade of ceramics affects the degree of polymerization of light–polymerized resin luting materials, and achieving optimal polymerization is important for long-term colour stability.[13,14]

The purpose of this study was to evaluate Degree of monomer conversion of three different Resinous materials used for cementation of ceramic laminate veneers. The Null hypothesis tested in this study was, there would be no difference in degree of monomer conversion of the three tested resinous materials.

Materials and Methods:

2.1-Materials used:

In this in vitro study 45 ceramic slices were made out of lithium disilicate (IPS Emax CAD) blocks covering three resinous materials were investigated in this study: preheated *filtek*[tm]Z350XT *universal* restorative material, Rely*X*[TM] Veneer and *filtek*[tm]Z350XT Flowable Composite, all from the same manufacturer(3M ESPE, St. Paul, MN, USA).

Samples preparation:

Total number of 45 sample (n=45) were divided into three groups according to the type of resinous material used:

Group (R) light cured resin cement (n=15)

Group (P) preheated composit (n=15)

Group (F) flowable composite (n=15).

2.2-Methods:

1-Construction of ceramic slices:

Fourty five ceramic slices were machined from low translucenct (LT) IPS Emax CAD blocks shade A2 (n=45). Using sawing machine (Isomet 4000) cutting at a speed of 2500 rpm with water cooling**,** the slices were sliced to a uniform standard thickness of 0.5 mm.

2-Crystallization:

Slices were subjected to crystallization firing per manufacturer's instructions using a Programat CS ceramic furnace (Ivoclar Vivadent). The manufacturer-set Program 1 was used for crystallization firing, included a stand-by temperature of 403°C followed by a 6-minute closing time. The first firing temperature of 820°C (T1) was reached with a heating rate of 90°C/min and held for 10 minutes. The second firing temperature of 840°C (T2) was reached with a heating rate of 30°C/min and held for 7 minutes. Vacuum cycles were held between 550°C and 820°C, and again between 820°C and 840°C. The long-term cooling was at 700°C with a 20°C/min cooling rate.

3-Fabrication of Teflon mould:

A Teflon mould were fabricated to ensure a standard thickness of the resinous material samples with external diameter of 20 mm and inner dimension 14*14 assure square shape with thickness 0.6 mm ensure a 0.1 mm uniform thickness of resinous material and room of 0.5 mm for ceramic disc positioning.

4-Application and curing of the resinous material:

The ceramic slices were seated on the inner stopper of the mould. Resinous material was then dispensed from the syringe on the ceramic slice, a celluloid strip placed and then the glass slab was applied with standardized weight to ensure complete seating and uniform 0.1 mm thickness of sample created by the mould.

A-Rely X Veneer: light-cured resin cement (RelyX Veneer; 3M ESPE, St. Paul, MN, USA), shade A1 was dispensed from the syringe into the ceramic slices to fill the 0.1 mm space between the surface of the mould and the surface of the ceramic slice seated in the square cut.

B-Flowable Composite: Also, flowable composite (FiltekTM, Z350XT; 3M Deutschland GmbH), shade A1 was dispensed directly from the Syringe.

C-Preheated composite: a thin layer from composite which had been preheated first for 15 minutes at 68$^{+}_{-}$2°C using a Composite Warmer (ceramic one) ,was dispensed by carver that was preheated at the same device, which then applied from the syringe over the ceramic slice.

To reduce heat dissipation, the maximum time between the removing of the composite from the heater and placing it in the mould was 10 seconds. However, in this study, maximum studied temperature (68°C) was selected to compensate heat dissipation; The temperature of the glass slabs was standardized at 37 °C which placed before each sample fabrication in an incubator to simulate the clinical conditions.

5-Evening out the luting agents:

After applying any of the tested materials two glass slabs were placed with the mould in between while the ceramic slice side facing upward and the luting material side facing downwards, celluloid strips were placed facing the samples in order to prevent the adhesion to the glass slab. A standardized weight of (0.5 kg) was applied at the top of the upper glass slab to get a uniform cement thickness of 0.1mm underneath the ceramic[15].

6-Curing of the luting materials:

Initial curing was done from the peripheries of the glass slabs using the IvoclarVivadent (Bluephase) providing a light intensity of 1,200 mW/cm2, for 2 seconds and then after removing the weight and the upper glass slab full curing through ceramic was completed for the 45 specimen for 30 seconds according to manufacturer's instructions, on a dark background to avoid light reflection[11]. Speciemens were then separated from the ceramic slices using (blade #15).

3-Measurement:

Measuring degree of conversion:

Fourty five specimen where degree of conversion of each will be tested using FTIR, groups divided according to the type of resinous material (n=5).

3.1. Samples preparation for FTIR test:

A-Cured samples Preparation:

The cured samples previously prepared, were ground into fine powder then mixed with potassium bromide powder. The mixture is then compacted between two stainless steel disks under hydraulic pressure forming a disc shaped specimen for the FTIR spectroscopy.

B- Uncured samples Preparation:

Uncured cement specimens from each material were dispensed from the syringe mixed with potassium bromide powder and compacted to form a disc shaped specimen for FTIR spectroscopy.

3.2. Measuring the Degree of Conversion values:

Cured and uncured cement samples were collected after preparation and scanned at a resolution of 4cm^{-1} and given a blot of wave number from 4000-400cm^{-1}against absorbance peak intensities, using OMNIC 5.1c software connected to the FTIR unit. Degree of conversion for each specimen was evaluated using Fourier Transform Infra-Red spectroscopy. In the MIR region, DC is determined by measuring the intensity (or area) decrease of the methacrylate (C=C) stretch absorption band at the absorption peaks of the aromatic double bonds were recorded at 1608 cm^{-1} (Abs 1608) and the peak of the aliphatic double bonds (C=C) were registered at 1637 cm^{-1} (Abs 1637 as the methacrylate monomer is converted to polymer.

The percentage of unreacted aliphatic C=C bonds remaining throughout the polymerization reaction is obtained by the following equation:

$$(\%C{=}C) = \frac{[Abs(aliphatic)/Abs(aromatic)]_{polymer}}{[Abs(aliphatic)/Abs(aromatic)]_{monomer}} \times 100$$

DC is determined by subtracting the residual percentage of aliphatic C=C from 100%,

$$DC\% = 100-(\%C{=}C)$$

Numerical data were explored for normality by checking the data distribution, calculating the mean and median values and using Kolmogorov-Smirnov and Shapiro-Wilk tests. Data showed parametric distribution so; it was represented by mean and standard deviation (SD) values. One-way ANOVA followed by Tukey's post hoc test was used to study the effect of luting agent on degree of conversion. Statistical analysis was performed with IBM® SPSS® Statistics Version 26 for Windows.

® IBM Corporation, NY, USA.

®SPSS, Inc., an IBM Company.

Results:

Mean Standard deviation (SD) for degree of conversion for different resinous materials were presented in Table (1) and Figure (1). There was a significant difference between different resinous materials ($p<0.001$). Light cure resin cement (62.13±1.71) showed the highest mean value followed by flowable composite (59.87±1.74) while pre-heated composite (52.88±2.76) showed the lowest mean value. Post hoc pairwise comparison showed pre-heated composite to have significantly lower mean value than other resinous materials ($p<0.001$).

Table (1): Mean ± standard deviation (SD) of degree of conversions for different resinous materials.

Resinous materials (mean±SD)			P-value
Light cured resin cement (R)	**Flowable composite (F)**	**Pre-heated composite (P)**	
62.13±1.71^{A}	59.87±1.74^{A}	52.88±2.76^{B}	**<0.001***

Different superscript letter within the same row indicates a statistically significant difference*; significant (p ≤ 0.05) ns; non-significant (p>0.05)

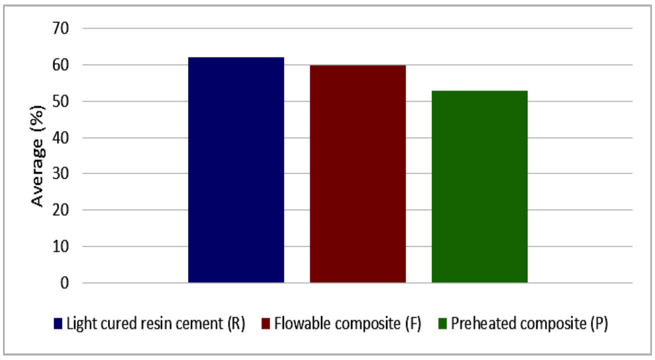

Figure (1): Bar chart showing average degree of conversions for different resinous materials.

Discussion:

In this study, cement thickness was controlled at 0.1 mm to clinically imitate the maximum accepted cement thickness under ceramic laminate veneers, as different cement thicknesses could influence the final veneer restoration colour, as recommended by other studies.[16,17] Cement shade A1 was used for all cements for standardization.

In clinical conditions, the temperature at which the material is inserted in the tooth and the time delayed to insert, model, and photoactivate the material on the tooth should be considered, the heating temperature used to preheat composite in this study was 68°C to compensate for heat dissipation.[18,19] A study by Daronch et al. found that after removing composites from heating device, 50% of the temperature attained will be lost after 2 minutes and almost 90% will be lost after 5 minutes.[20]

In This study Ceramic slices were constructed from IPS e.max CAD blocks of shade A2 for all samples for standardization. The thickness of the ceramic slices was 0.5 mm as it is the ideal thickness for minimal invasive preparations and also to allow the increase light transmission through the thin translucent ceramic to obtain a higher degree of conversion of the underlying resinous materials.[21]

To eliminate variables that might affect the degree of conversion such as curing time, mode and intensity, a single light curing device was used in this study.[22,23] To avoid the oxygen inhibiting layer during the polymerization of cement, a mylar strip was placed under each resin cement specimen during the curing of the cement and to avoid sticking to the glass slab, the light cure tip was positioned in direct contact to ceramic slices to confirm maximum light penetration through it and to ensure a standardized distance between the ceramic and the light curing tip throughout all specimens.[24]

Potassium Bromide (KBr) pellet method was used to prepare the specimens for FTIR spectrometer as all the solid samples were grinded with a mortar and pestle and then mixed with KBr ground particles. The mixture is then compacted between two stainless steel disks under hydraulic pressure. This was shown in other studies.[25,26]

Regarding the tested null hypothesis that there would be no difference in the degree of monomer conversion of the three tested resinous materials was rejected.

There was a statistical significant difference in the degree of monomer conversion values between the three tested materials (Rely X Veneer resin cement, Filtek™ Z350 XT Flowable composite and Preheated Filtek™ Z350 XT Universal composite). Rely X Veneer resin cement showed the highest conversion values.

The differences found in the conversion values could be attributed to variation in resins composition, in terms of monomeric systems, type and quantity of load particles, concentration of diluents and initiators[27].

Rely x veneer was founded to has the highest value for degree of conversion which can be explained by both its smaller filler particle size, lesser filler loading (66% by weight) and the viscosity of the resins.

The lower viscosity of resins allows better monomeric mobility and distribution of free radicals inside the material, which can enhance the polymerization process leading to a greater monomer conversion. Because of this principle, low viscosity composites may allow enhanced diffusion of reactive groups and promote the curing reaction, resulting in a higher DC. So that the filler loading and the viscosity of composites may interfere in the monomer conversion, since they could restrict the mobility of monomers and the propagation of polymerization reaction.[28,29,30]

In addition, the higher molecular mobility in the early stages of the polymerization reaction in the flowable composite may have also contributed to the higher conversion of the polymer network into formation in comparison to the preheated composite.

On the other side, the higher filler particles loading in the preheated composites (72.5% by weight) may have reflected more of the incident light, reducing light-transmittance through the resin material and, consequently, reducing the degree of conversion.[31]

However, the pre-heating did not influence on statistical difference in the degree of conversion of composite. B. Procopiak et al. supported our results in their findings that pre-heating of composite resin did not increase their DC.[32]

Conclusion:

Degree of conversion of light cured resin cement showed better values than flowable composite and preheated composite.

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