



Degree of Conversion and Hardness of Two Different Systems of the Vitrebond™ Glass Ionomer Cement Light Cured with Blue LED

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ABSTRACT

This study investigated the physicochemical properties of the new formulation of the glass ionomer cements through hardness test and degree of conversion by infrared spectroscopy (FT-IR). Forty specimens ($n = 40$) were made in a metallic mold (4 mm diameter x 2 mm thickness) with two resin-modified glass ionomer cements, Vitrebond™ and Vitrebond™ Plus (3M/ESPE). Each specimen was light cured with blue LED with power density of 500 mW/cm² during 30 s. Immediately after light curing, 24h, 48h and 7 days the hardness and degree of conversion was determined. The Vickers hardness was performed by the MMT-3 microhardness tester using load of 50 gm force for 30 seconds. For degree of conversion, the specimens were pulverized, pressed with KBr and analyzed with FT-IR (Nexus 470). The statistical analysis of the data by ANOVA showed that the Vitrebond™ and Vitrebond™ Plus were no difference significant between the same storage times ($p > 0.05$). For degree of conversion, the Vitrebond™ and Vitrebond™ Plus were statistically different in all storage times after light curing. The Vitrebond™ showed higher values than Vitrebond™ Plus ($p < 0.05$). The performance of Vitrebond™ had greater results for degree of conversion than Vitrebond™ Plus. The correlation between hardness and degree of conversion was no evidence in this study.

Keywords: Degree of conversion, Glass ionomer cement, Hardness.

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INTRODUCTION

Wilson and Kent introduced the glass ionomer cement (GIC) with the objective to unite biological and adhesive properties

of the silicate cement and the zinc polycarboxylate since 1972. These materials have been commonly used due to the properties of fluoride release with a potential reduction in secondary caries, thermal compatibility with tooth enamel and dentin, minimized microleakage at the modulus of elasticity similar to dentin, tooth-enamel interface due to low shrinkage and low cytotoxicity.¹⁻¹⁰

The GIC sets via an acid-base reaction between calcium and/or aluminum cations released from a reactive glass and carboxyl anions pendent on polyacid. The polymer backbones of GIC has been by poly (acrylic acid) homopolymer, poly (acrylic acid-co-itaconic acid) or/and poly (acrylic acid-co-maleic acid) copolymers.^{11,12} The conventional GICs are of insufficient strength for use in high load bearing regions. For minimize these problems, changes occurred in the composition of GIC over the years had been obtained improvements physical properties through of the inclusion of resinous components in 1988 for Antonucci¹³ and with the addition of photoinitiators that it made possible the increase of the working time in 1989 for Mitra.¹⁴

The resin-modified GIC (RMGIC) has more advantages than conventional glass ionomer, as activation by light cure, therefore, more convenient and easy to use. Furthermore, they present a greater initial resistance and working time, allowing the use as pulp protection before the introduction of composite resin.¹⁵

The RMGIC for pulp protection generally is presented in a bottle (contents) powder and another one (contents) liquid, which are mixed and after inserted in the cavity, and then the mixture is light cured. Nevertheless, errors of proportion powder/liquid can bring damage to the properties of the material. The increase in the amount of powder occurs

to decrease the resistance, and the decrease of powder occurs to increase of the HEMA (2-hydroxyethyl-methacrylate), which is strongly hydrophilic resulting greater uptake of water by the material and the negative effects.¹⁶⁻¹⁸

Recently, a new formulation of RMGIC for pulp protection is commercially available. This new material is a two-part liquid/paste system. The liquid/paste materials are contained in the dispensing system. The dispensing system provides simultaneous dispensing of each component for a consistent mix.

Thus, the objective of this study was to evaluate the physicochemical properties of this new formulation of RMGIC through hardness test and degree of conversion by fourier transform infrared spectroscopy (FT-IR).

MATERIALS AND METHODS

Materials

The materials used in this study are described in Table 1. The Vitrebond™ is a commercial modified GIC and it is characterized by two reactive mechanisms. One of them, an acid-base crosslinking reaction between an ion-leachable glass and an aqueous polyacid and a polymerization activated by visible light. The product is made of a solid powder and a liquid solution mixed in the ratio 1.4:1 by weight and this way is more susceptible to occur mixed error. On the other hand, the Vitrebond Plus™ is a two-part liquid/paste system. The liquid/paste materials are contained in the 3M™ESPE™Clicker™Dispensing System. This dispensing system provides simultaneous dispensing of each component for a consistent mix. The main compositions as indicated by the manufacturers are listed in Table 1.

Specimens Preparation

The specimens (diameter 4.0 ± 0.1 mm, thickness 1 ± 0.1 mm) were made using a stainless steel mold. The materials were prepared and mixed according to the manufacturer's instructions. The mold was filled with the RMGIC used and a polyethylene film covered each side of the mold. After this, a glass slide was placed on the top surface of the mold.

To standardize the top surface of the specimens, a circular weight (1 kg) with an orifice to pass the light tip of the light-curing unit (LCU) was placed on the top surface (Fig. 1).

The LCU based on blue LED (LEC 1000, MM Optics, São Carlos, Brazil) was used to irradiate ($\lambda = \pm 470$ nm, 500 mW/cm², $\phi = 8$ mm) the specimens from one side along the whole extension in constant polymerization mode for 30 s. The power was checked using Fieldmaster (Sensor LM-3 HTD, Coherent Commercial Products Division, model number FM, set n° WX65, part number 33-0506, USA) and, then, the power density was calculated by the equation:

$$I = P/A$$

The glass slide thickness standardized the distance from the light tip to the RMGIC and provided a smooth surface. Five specimens were made for each group (n = 40). The groups were divided in different storage times: Immediately after curing, 24 and 48 hours and 7 days.

Hardness Test

The top surface of the specimens was divided in four exactly quadrants. The hardness testing machine, MMT-3 Microhardness Tester (Buehler, Lake Bluff, Illinois, USA, see Fig. 2) equipped with the Vickers diamond (VHN) was used with load of 50 gf (gram force) during 30 seconds. An

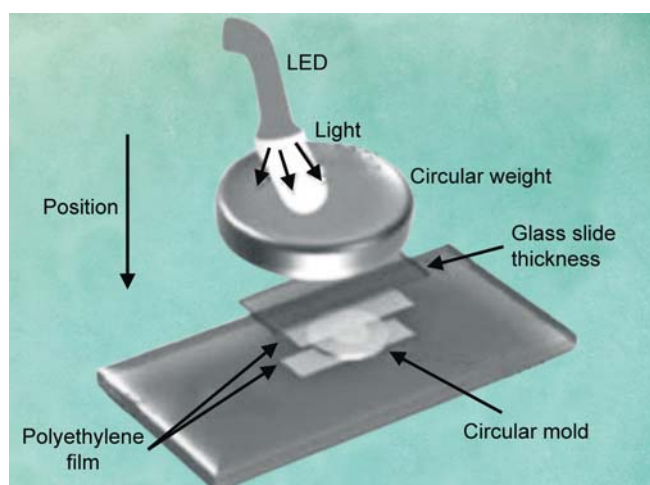


Fig. 1: Schematic drawing of the specimen's preparation for hardness and FT-IR tests

Table 1: GIC used in this study

Glass-ionomer cement	Composition	Manufacturer	Batch number
Vitrebond™ Plus paste	Fluoroaluminosilicate glass		
	Camphorquinone, co-initiator, acrylic-itaconic acid copolymer with pendant methacryloxy groups		
Vitrebond™ Plus liquid	2-hydroxy-ethyl-methacrylate (HEMA) and water	3M/ESPE Seefeld, Germany	AX7AG
Vitrebond™ powder	Fluoroaluminosilicate glass		
VitreBond™ liquid	Camphorquinone, coinitiator, acrylic-itaconic acid copolymer with pendant methacryloxy groups, 2-hydroxy-ethyl-methacrylate (HEMA) and water	3M/ESPE Seefeld, Germany	7KY 7HK

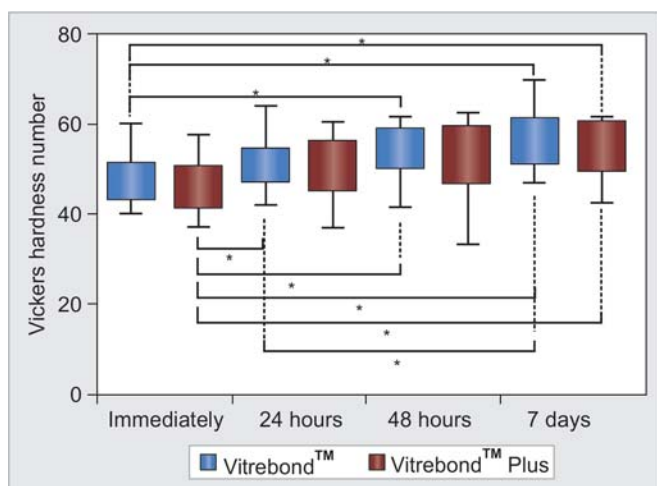


Fig. 2: Results of hardness immediately after light irradiation, 24 and 48 hours and 7 days of the Vitrebond™ and Vitrebond™ Plus. Bars indicated statistically significant differences at 5% level (Tukey's test; * $p < 0.05$)

impression for quadrant took place on the top surfaces of the specimens. The specimens were measured immediately after light-curing process, 24 and 48 hours and 7 days. The hardness mean values were calculated and submitted to statistical analysis.

Degree of Conversion Measurements (DC%)

Immediately after light-curing process, 24 and 48 hours and 7 days, the RMGIC were pulverized into a fine powder. The pulverized RMGIC were maintained in a dark room until the moment of the FT-IR analysis. Ten milligrams of the ground powder was thoroughly mixed with 100 mg of KBr powder salt. This mixture was placed into a pelleting device and then pressed in a press with a load of 10 tons during 1 minute to obtain a pellet.

The number of double carbon bonds, which are converted in single bonds, provides the degree of conversion (%) of RMGIC. To measure the degree of conversion, the pellet was then placed into a holder attachment into the spectrophotometer (Nexus™ 470 FT-IR ESP, Thermo Nicolet, UK, serial number: AEPO301044). For this technique, the specimens were made and analyzed immediately after light-curing process, 24 and 48 hours and 7 days. FT-IR spectra of both uncured and cured specimens were analyzed using an accessory of reflectance diffuse. The measurements were recorded in absorbance operating under the following conditions: 32 scans, 4 cm^{-1} resolution, 300 to 4000 cm^{-1} wavelength.

The net peak absorbance height of methacrylate C = C bonds (C = C, peak at 1638 cm^{-1}) was used as the analytical frequency, while the peak absorbance height of ester bonds (C = O, peak at $1,712 \text{ cm}^{-1}$) was used as a reference frequency. The C = C/C = O peak absorbance height ratios

were used to calculate the percentage amount of residual C = C bonds. After calculating the degree of conversion (DC%), the values were submitted to statistical analysis.

Statistical Analysis

DC and VHN data were submitted to two-way ANOVA/Tukey's test at significance level of 5%. Also, linear regression analysis was conducted with both variables.

RESULTS

The results of hardness expressed as Vickers hardness number (VHN) at different storage times are shown in Figure 2.

The statistical analysis of the data by ANOVA indicated that the Vitrebond™ and Vitrebond™ Plus showed not significant differences between the same storage times ($p > 0.05$). The mean values increased significantly in the first 48 hours ($p < 0.05$) and no significant differences were found between 48 hours and 7 days for Vitrebond™ and Vitrebond™ Plus.

The representative FT-IR spectra of the RMGIC before and after light-curing process are shown in Figures 3A and B.

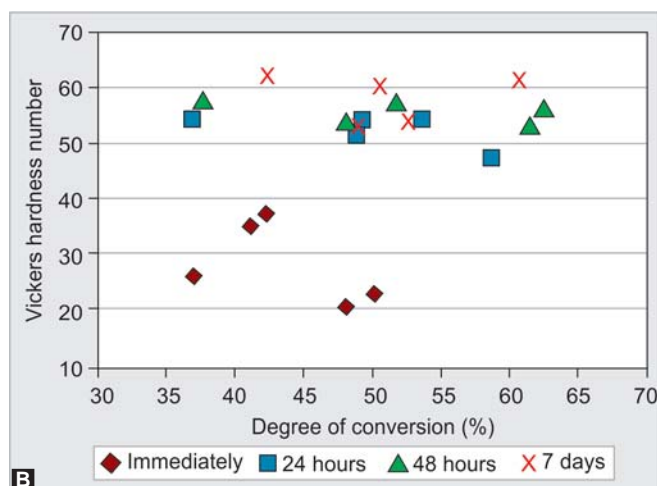
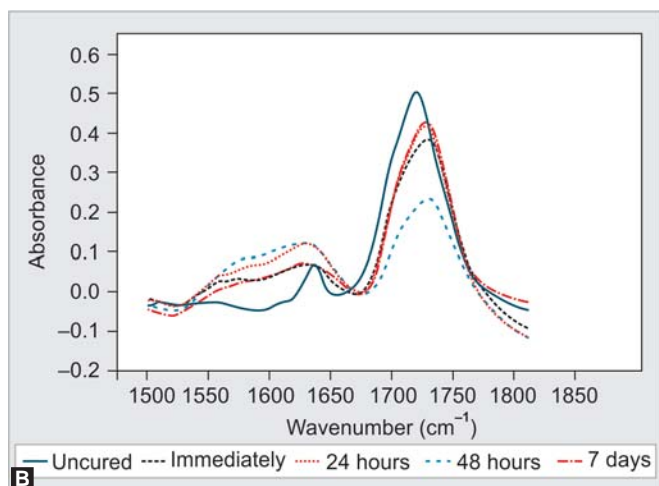
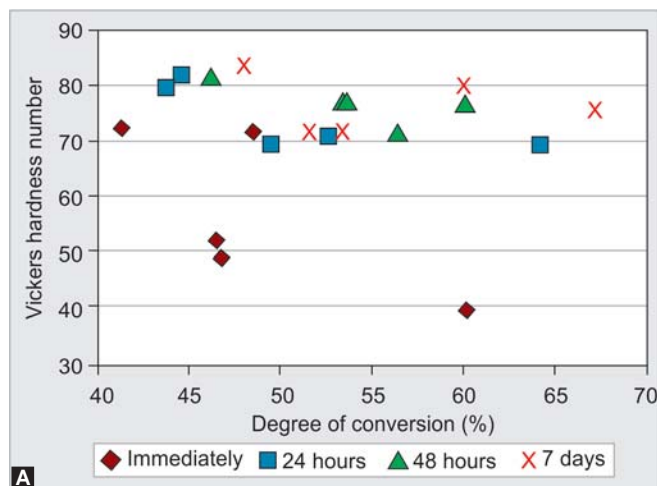
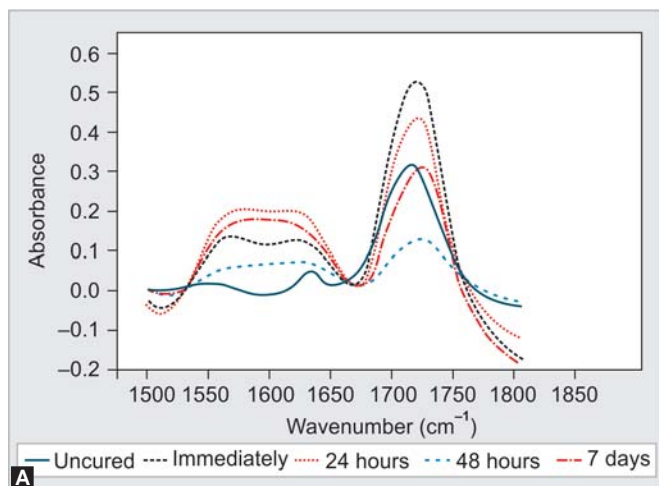
The degree of conversion of the Vitrebond™ and Vitrebond™ Plus are shown in Figure 4. There was significant difference only between immediately and 24 hours after light-curing process ($p < 0.05$). For the other storage times, any significant difference was found ($p > 0.05$). The Vitrebond™ and Vitrebond™ Plus were statistically different in all storage times, which the Vitrebond™ showed higher mean values than Vitrebond™ Plus ($p < 0.05$).

The Pearson's correlation tests demonstrated no significant relationships between DC and hardness from the Vitrebond™ immediately after light-curing process ($r = 0.5069$, $p = 0.1773$), 24 hours ($r = 0.5735$, $p = 0.1382$), 48 hours ($r = 0.4736$, $p = 0.1989$) and 7 days ($r = 0.0129$, $p = 0.8556$), and from the Vitrebond™ Plus immediately after light-curing process ($r = 0.2796$, $p = 0.3595$), 24 hours ($r = 0.3770$, $p = 0.2706$), 48 hours ($r = 0.2297$, $p = 0.4140$) and 7 days ($r = 0.0000$, $p = 0.9904$). The correlation between degree of conversion and hardness of the Vitrebond™ and Vitrebond™ Plus are shown in Figures 5A and B.

DISCUSSION

The RMGICs are commonly used in dentistry due to the properties, such as fluoride released, biocompatibility, low shrinkage, thermal compatibility and less caries formation¹⁻¹⁰ with tooth becoming an important material for studying.

The hardness is one of most important physical properties to dental materials that can evaluate the resistance



Figs 3A and B: FT-IR spectra of uncured and cured RMGI dental cements (A: Vitrebond™ and B: Vitrebond™ Plus at different storage times)

Figs 5A and B: Correlation between the Vickers hardness number (VHN) and the degree of conversion for (A) Vitrebond™ and (B) Vitrebond™ Plus immediately after curing, 24 and 48 hours and 7 days

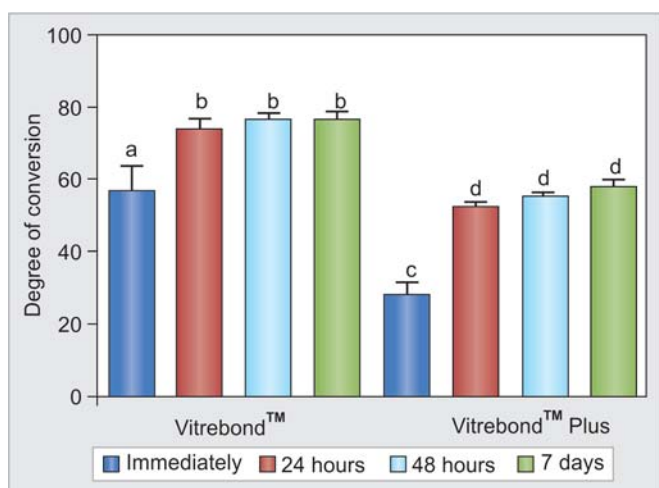


Fig. 4: Results for degree of conversion of the RMGIC used in this study. Different letters (a-d) indicate statistically significant difference at 5% level (Tukey's test; $p < 0.05$)

by indentation.¹⁹ The RMGICs specimens of Vitrebond™ and Vitrebond™ Plus appear to be still increasing in hardness when the last measurement was collected at 7 days

after light-curing process. The both RMGICs showed similar increasing of the hardness mean values. The GICs are known to continually harden and strengthen²⁰ due the cations released from the glass are increasingly bound to the polyacid (anionic) chains and the hydration process continues²¹ and has been reported that the crosslinked polysalt matrix structure of GICs, a secondary hydrated silicate structure form with time.^{22,23} Both the groups showed significant differences between immediately and 48 hours after light-curing process. The initial resistance observed can be explained due to the chemical composition, glass structure, concentration and molecular weight of the polycarboxylic acid and the proportion powder/liquid^{19,24} and by storage condition, which becomes important to avoid degradation.^{25,26}

The handling properties of GICs are dependant on the correct proportion of the powder/liquid that can be modified by errors on mixture. The Vitrebond™ Plus showed as dispensing system that provides simultaneous dispensing of each component. The use of dispensing system eliminates the errors of proportion powder/liquid.

The FT-IR method to measure the DC% in dental materials is widely used^{27,28} and the present study showed differences significant between immediately and 24 hours after light curing for both RMGICs. The GICs are hydrophilic and absorb water that continues to take a mediating role in the acid-base reaction on the polymerization process²⁹ and this initial reaction is important factor for overall clinical success because an adequate degree of conversion obtains slow shrinkage and biocompatibility of the restoration. Nevertheless, the higher degree of conversion in composites promotes higher polymerization shrinkage.^{30,31} The Figure 3 shows the middle infrared spectral regions taken into consideration for DC% evaluation and reports the spectrum in the 1,500 to 1,850 cm^{-1} region showing the bands of ester bonds (1,712 cm^{-1}) and methacrylate C = C bonds (1,610 cm^{-1}) stretching vibrations of the cement from which DC, as a function of the storage time, can be calculated. The performance of VitrebondTM was greater than VitrebondTM Plus, which can be notable in the initial reaction (immediately) residual C = C bonds of the VitrebondTM that had similar values after 7 days of storage time after light-curing process to VitrebondTM Plus.

The correlation between hardness and degree of conversion was not found in our results (see Figs 5A and B). Some studies correlated degree of conversion, filler concentration and mechanical properties of dental materials and concluded that no correlation was found between the degree of conversion and any of the mechanical properties of the composite resins³²⁻³⁴ and GIC.³⁵

The new formulation of RMGIC do not shown higher degree of conversion as the old formulation and the hardness of them were similar, however, the new formulation is more easy to use, consequently this way is less susceptible to occur mixed error.

CONCLUSION

Within the limitations of the current study, it can be concluded that RMGICs may show increased 7 days after curing, which the first 48 hours promotes initial resistance. Nevertheless, the performance of VitrebondTM had greater results of degree of conversion than VitrebondTM Plus. The correlation between hardness and degree of conversion was not evident in this study.

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