

EFFECT OF DIFFERENT POLYMERIZATION DEVICES ON THE DEGREE OF CONVERSION AND THE PHYSICAL PROPERTIES OF AN INDIRECT RESIN COMPOSITE

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ABSTRACT

Polymerization of indirect resin composites (IRC) is carried out in the laboratories using special photo-polymerization devices to achieve a higher degree of conversion (DC). Such devices present variation in chambers and light output which may have consequences on the chemical and physical properties of IRCs. This study evaluated the effect of different polymerization devices on the flexural strength, Vickers microhardness and DC of an IRC.

Specimens were prepared from an IRC material, Sinfony (3M ESPE), using special molds for flexural strength test (N=30) (25x2x2 mm, ISO 4049), Vickers microhardness test (N=30) (5x4 mm) and for DC (N=30) utilizing Micro-Raman Spectroscopy. All specimens were submitted to initial polymerization with a Visio Alpha unit (3M ESPE) and then randomly divided into three groups (n=10/group). Specimens in Group 1 (control) received additional polymerizations using a Visio Beta

Vario device (3M ESPE), and those in Group 2 and Group 3 using Powerlux (EDG) and Strobolux (EDG) devices, respectively. DC and mechanical tests were then conducted. For the mechanical tests, the data were analyzed using ANOVA and Tukey's tests ($p < 0.05$) and for DC, one-way ANOVA was used. Polymerization in Strobolux (Group 3) resulted in significantly lower flexural strength (MPa) values (134±27) compared to Visio Beta Vario (165±20) (Group 1) ($p < 0.05$). The lowest microhardness values (Kg/mm²) were obtained in Group 3 (30±1) ($p < 0.05$). DC was similar in all groups (75±1, 91±5, 85±7 % for Visio Beta Vario, Powerlux and Strobolux, respectively) ($p = 0.1205$).

The type of polymerization device may affect the flexural strength and Vickers hardness of the IRC tested. DC also seems to be affected by the type of polymerization device but the results were not significant.

Key words: tensile strength, composite resins, hardness test.

EFEITO DE DIFERENTES UNIDADES POLIMERIZADORAS NO GRAU DE CONVERSÃO E NAS PROPRIEDADES FÍSICAS DE UMA RESINA COMPOSTA INDIRETA

RESUMO

As polimerizações de resinas compostas indiretas (RCI) são realizadas em Laboratório em dispositivos fotopolimerizadores especiais para que seja alcançado um maior grau de conversão (GC). Estes dispositivos apresentam variações nas câmeras e nas lâmpadas polimerizadoras as quais podem gerar conseqüências nas propriedades físicas e químicas das RCIs. Este estudo avaliou o efeito de diferentes unidades polimerizadoras na resistência à flexão, dureza Vickers e GC de uma RCI.

Amostras da RCI Sinfony (3M ESPE) foram preparadas, utilizando matrizes especiais para o teste de resistência à flexão (N=30) (25x2x2 mm, ISO 4049), teste de microdureza Vickers (N=30) (5x4 mm) e para o GC (N=30), utilizando a espectroscopia Micro-Raman. Todas as amostras foram submetidas à polimerização inicial na unidade Visio Alpha (3M ESPE) e em seguida elas foram divididas aleatoriamente em três grupos (n=10/por grupo). As amostras do Gr1 (controle) tiveram sua polimerização final realizada na unidade Visio Beta Vario (3M

ESPE), e as do Gr2 e Gr3 nas unidades Powerlux (EDG) e Strobolux (EDG), respectivamente e então os testes mecânicos e do GC foram conduzidos. Para os testes mecânicos, os dados foram analisados utilizando a análise de Variância (ANOVA) e o teste de Tukey ($p < 0.05$) e ANOVA 1-fator para o GC.

A polimerização na unidade Strobolux (Gr3) gerou valores de resistência à flexão (MPa) significativamente inferiores (134±27) comparado à unidade Visio Beta Vario (165±20) (Gr1) ($p < 0.05$). Os menores valores de microdureza (Kg/mm²) foram obtidos para o Gr3 (30±1) ($p < 0.05$). O GC em todas as unidades polimerizadoras (75±1, 91±5, 85±7 % para Visio Beta Vario, Powerlux e Strobolux, respectivamente) foi semelhante entre os grupos ($p = 0.1205$).

O tipo de unidade polimerizadora afetou a resistência à flexão e a dureza Vickers da RCI testada. O GC também foi afetado pelo tipo de unidade polimerizadora, mas a diferença não foi significativa.

Palavras chaves: resistência à flexão, resina composta, teste de dureza.

INTRODUCTION

Photo-activated resin composites are commonly used restorative materials in dentistry for both anterior and posterior restorations. Such tooth-colored restorations can be adhered to the dental tissues and they can be made directly or indirectly at chairside or at dental laboratories. One drawback of direct application of resin composites is the polymerization shrinkage that generates stress at the interface between the resin and dental tissues, leading to marginal gaps or hypersensitivity when it exceeds the bond strength between the resin composite and the tooth¹. On the other hand, resin composite restorations built using indirect techniques result in lower water sorption and less discoloration¹. In addition, indirect resin composites (IRC) require less finishing and polishing time at chairside. They do not require high technical skills and occlusal anatomy, and proximal contacts are easier to obtain in the laboratory than using direct methods²⁻⁶.

Degree of conversion (DC) has a significant influence on the physical^{1,7} and biological properties⁸ of resin composite restorations and is highly dependent on factors such as composition of the material,⁹ color and translucency,¹⁰⁻¹² distance of the light tip to the surface¹³ and the irradiance of the polymerization lamp^{11,14}. In this context, IRCs allow for higher DC since polymerization is carried out in laboratories in special photo-polymerization devices in which all surfaces of the restoration can be polymerized in the chamber of the unit. Depending on the

type of polymerization device, a combination of light, heat, vacuum and pressure results in a 10 to 20% improvement in the mechanical properties of these materials when compared to values obtained using direct polymerization techniques¹⁵. Unfortunately, with the increasing number and improved properties of the IRCs, dental technicians and some clinicians have to invest not only in the IRC material itself but also in the polymerization devices. Polymerization modes also vary among devices.

The conversion degree of monomers to polymers in dental resins can be evaluated using hardness tests¹⁶⁻¹⁸ or FT-Raman Spectroscopy¹⁷. However, there is still no consensus in the dental literature on which method should be used for the assessment of the DC. Furthermore, polymerization devices intended for IRCs have varying numbers of lamps and different light outputs and they also vary in design; specifically, they contain different polymerization chambers, all of which may have consequences on the chemical and physical properties (i.e. microhardness, flexural strength) of IRCs. This study therefore evaluated the effect of three different polymerization units on the flexural strength, Vickers microhardness and DC of an IRC.

MATERIALS AND METHODS

Table 1 shows the brand names, main characteristics, manufacturers and serial numbers of the photo-polymerization devices and the resin composite used for the experiments.

Table 1: Main characteristics, brand names, manufacturers and serial numbers of the photo-polymerization devices and the resin composite used for the experiments.

Brand name	Power	Manufacturer	Serial Number
Photo-polymerization devices			
Visio Alfa	400 mW/cm ²	3M ESPE, St Paul, MN, USA	900021000229
Visio Beta Vario	470 mW/cm ²	3M ESPE, St Paul, MN, USA	910012000169
Powerlux	1200 mw/cm ²	EDG, São Carlos, Brazil	12/0392
Strobulux	1200 mw/cm ²	EDG, São Carlos, Brazil	02/10435
Resin composite			
Sinfony	Composition HEMA and 10-30% Octahydro-4,7-methano-1H-indenediyl) bis(methylene) diacrylate, strontium-aluminium borosilicate, glass, silicone oxide, silane and photoinitiators	3M ESPE, ST Paul, MN, USA	03216

Flexural strength test

IRC specimens (N = 30) (Shade A2) were prepared in accordance with ISO 4049¹⁹ using a transparent polyethylene mold (25 x 2 x 2 mm) and increments (1 mm thick each) and they were polymerized initially from the top surface in a Visio Alpha unit for 5 seconds, according the manufacturer's recommendation. They were then randomly divided into three groups and polymerization was conducted in one of the three photo-polymerization devices (Figs. 1a-c) according to the procedures described in Table 2. The specimens were subsequently submitted to a three-point flexural test.

The flexural strength tests were performed in a universal testing machine (Model DL-1000, EMIC Equipments and Systems Ltd, Sao Jose dos Campos, Brazil) where the load was applied at a constant transverse speed of 0.8 mm per minute until fracture occurred. Flexural strength was calculated according to ISO 4049¹⁹ guidelines, using the following equation, where "P" is the maximum load upon fracture (N), "L" the distance between two parallel supports (20 mm), "b" width and "d" thickness of the specimen:

$$\Sigma = \left[\frac{3PL}{2bd^2} \right]$$

Degree of conversion

isc-shaped IRC specimens (diameter: 5 mm; thickness: 4 mm) (N = 30) were prepared as described above. The specimens were stored in distilled water at 37°C for 24 hours and embedded in acrylic resin blocks. In order to remove the oxygen-inhibited

layer, each block was finished with wet silicone carbide papers up to 1200-grit and polished (Strues, Model DP 10, Panambra Ind. & Tec. S.A., São Paulo, Brazil) with diamond paste (3 µm).

The surfaces were analyzed by FT-Raman spectroscopy in order to evaluate the DC. The spectra of the uncured and cured resins were obtained by an FT-Raman Spectrometer (RFS 100/S, Bruker Inc, Karlsruhe, Germany) using 100 scans. The spectrum resolution was set at 4 cm⁻¹. The specimens were excited by the defocused line of an Nd:YAG laser source at λ=1064.1 nm with maximum laser power of approximately 90 mW at the

Table 2: Photo-polymerization devices tested and their polymerization modes

<i>Photopolymerization device</i>	<i>Polymerization mode</i>
<i>Visio Alfa</i>	<i>Pre- and intermediate polymerization designed for Sinfony; Photo-polymerization; 400 mw/cm2; 5 seconds each layer</i>
<i>Visio Beta Vario</i>	<i>Four fluorescent lights of blue spectra; final polymerization of Sinfony; mirrored polymerization chamber; photo-polymerization: 15 minutes under vacuum</i>
<i>Powerlux</i>	<i>Two xenon bulbs; Photo-polymerization: 1200 mw/cm2, 4 minutes under vacuum</i>
<i>Strobolux</i>	<i>One xenon bulb; mirrored circular chamber; Photo-polymerization: 1200 mw/cm2, 4 minutes under vacuum</i>

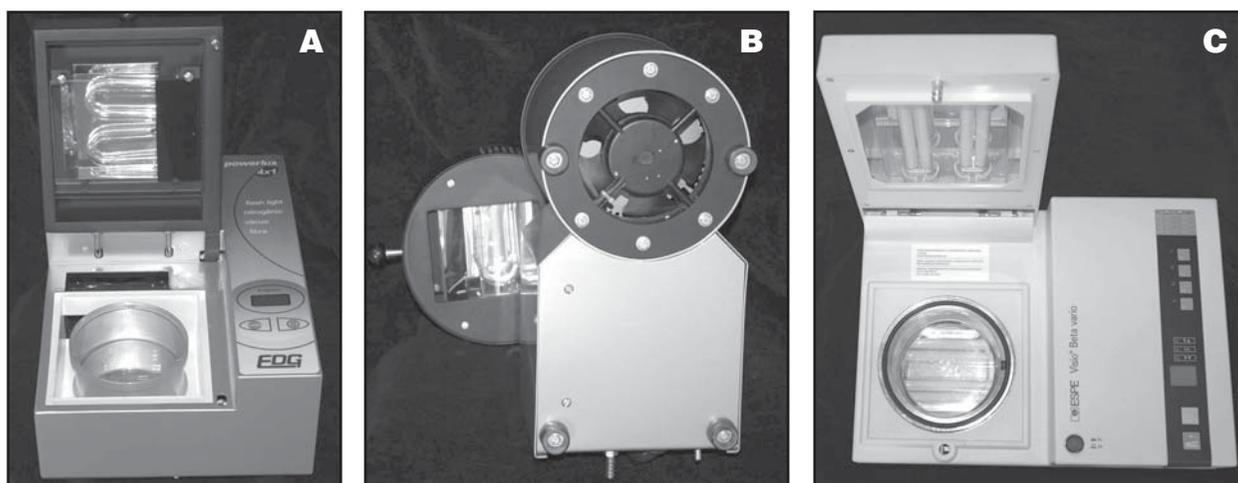


Fig.1: Light polymerization devices tested a) Powerlux, b) Strobolux, c) Visio Beta.

specimen. The uncured resin was positioned on an aluminum rod in a sample holder mounted on an optical rail for spectrum collection. For the 90 cured specimens, three spectra of the top surface and another three spectra of the bottom surface were collected, resulting in a total 480 spectra. Based on the measurements, one average spectrum for each surface was obtained, resulting in 160 spectra. The average FT-Raman spectra were analyzed by selecting a range between 1590 and 1660 cm^{-1} . The Raman peaks corresponding to the vibrational stretching modes at 1610 and 1640 cm^{-1} were fitted in Gaussian shapes to obtain the height of the peaks using specific software (Microcal Software Inc, Northampton, MA, USA). A comparison of the height ratio of the aliphatic carbon-carbon double bond (C=C) at 1640 cm^{-1} with that of the aromatic component at 1610 cm^{-1} for the cured and uncured conditions was performed in order to estimate the DC using equation (1). The aromatic C=C peak at 1610 cm^{-1} originated from the aromatic bonds of the benzene rings in the monomer molecules, and its intensity remains unchanged during the polymerization reaction. The mean value and standard deviation of the DC were calculated for each series where R = the percentage of uncured resin that is determined by band height at 1640 cm^{-1} /band height at 1610 cm^{-1} :

$$R_{\text{unpolymerized}} = \frac{\text{band height at } 1640 \text{ cm}^{-1}}{\text{band height at } 1610 \text{ cm}^{-1}} \quad (1)$$

$$R_{\text{polymerized}} = \frac{\text{band height at } 1614 \text{ cm}^{-1}}{\text{band height at } 1640 \text{ cm}^{-1}}$$

The percentage of DC was then calculated using the following equation (2):

$$DC (\%) = 100 * [1 - (R_{\text{polymerized}} / R_{\text{unpolymerized}})] \quad (2)$$

Table 3: Mean (\pm standard deviations) flexural strength values (MPa) for the resin composite processed in three different photo-polymerization devices

Groups	Mean \pm SD (MPa)
Visio Beta (Gr1)	165 \pm 20 ^a
Powerlux (Gr2)	151 \pm 27 ^{a,b}
Strobulux (Gr3)	134 \pm 27 ^b

*The same superscripted letters indicate no significant difference (Tukey's test, $\alpha = 0.05$)

Vickers microhardness test

Disc shaped resin composite specimens (diameter: 5 mm; thickness: 4 mm) (N = 30) were prepared as described above. The resin-rich layer was finished with wet silicone carbide papers up to 1200-grit and polished (Struers, Model DP 10) with diamond paste (3 μm).

The microhardness measurements were made employing Vickers microhardness test (FM 700, Future-Tech, Equilam, Tokyo, Japan). The specimens were submitted to 50 g load for 30 seconds. Three readings were made at different regions of each specimen at the top surface using the following equation where "P" was the applied load in Kg and "dv" was the average of the diagonal length in mm²:

$$\text{Vicker's microhardness (Kg/mm}^2\text{)} = 1.8544 \cdot \left[\frac{P}{dv^2} \right]$$

Statistical analysis

Statistical analysis was performed using Statistical Software for Windows (StatSoft Inc., version 5.5, 2000, Tulsa, OK) and Statistix for Windows (Analytical Software Inc., version 8.0, 2003, Tallahassee, FL, USA). The data were analyzed using one-way analysis of variance and Tukey's multiple comparisons test when appropriate. Significance level was set at $P < 0.05$.

RESULTS

Polymerization in Strobulux (Group 3) resulted in significantly lower mean flexural strength values of the IRC (134 \pm 27 MPa) compared to Visio Beta (165 \pm 20 MPa) (Group 1) ($p < 0.05$) (Tukey's test) (Table 3).

Microhardness values did not show significant differences between groups ($p > 0.05$) (Table 4).

DC values in all three polymerization devices (75 \pm 1, 91 \pm 5, 85 \pm 7 % for Visio Beta Vario, Powerlux and Strobulux, respectively) were not significantly different between groups ($p = 0.1205$).

Table 4: Mean (\pm standard deviations) Vicker's micro hardness values (Kg/mm²) for the resin composite processed in three different photo-polymerization devices

Groups	Mean \pm SD (Kg/mm ²)
Visio Beta (Gr1)	31 \pm 1 ^a
Powerlux (Gr2)	32 \pm 1 ^a
Strobulux (Gr3)	30 \pm 1 ^a

*The same superscripted letters indicate no significant difference (Tukey's test, $\alpha = 0.05$)

DISCUSSION

Development in the field of polymer technology made metal-free restorations possible by using IRCs, particularly due to the improvement in their physical properties¹⁵.

One of the most frequently used methods for quantifying the fracture strength of the restorative materials is the flexural strength test^{1,14}. This type of test could represent the dynamic nature of the stresses produced during mastication, which create different tensile, compression and shear stresses upon fixed-partial-dentures (FPD)²⁰. Flexural strength values obtained from the IRCs tested in this study (134 - 165 MPa) were similar to the values observed in other studies for other IRCs (120 to 160 MPa for ArtGlass, Heraeus-Kulzer and Targis, Ivoclar Vivadent) used for the same purposes.²⁰⁻²² Such resin composites are examples of second generation IRCs and the flexural strength values obtained show that they are suitable for inlays, onlays, FPDs and FPDs reinforced with fibers²³. The International Organization Standardization (ISO 4049)¹⁹ stated that IRCs should have minimum 100 MPa values in order to be suitable for such restorations. The results obtained exceeded the required value proposed by ISO 4049, regardless of the polymerization devices used. However, there were significant differences between the groups polymerized with either Powerlux or Strobolux, with the latter resulting in lower mean flexural strength values. Both these polymerization devices were used for the first time and had light intensity of 1200 mw/cm² according to the manufacturer's instructions, and worked with the same vacuum and photo-polymerization duration (4 minutes), according to the manufacturer's instructions. The only difference was that Powerlux had two stroboscopic xenon lamps, while Strobolux had only one, in a mirrored circular chamber. The higher number of lamps may have affected the results. The differences between lower flexural strength and microhardness, as well as the lower, though not significantly different DC results, may therefore be attributed to the variations in design and the number of stroboscopic xenon lamps in these two polymerization devices. It has been reported that increasing the exposure time of the resin to the light source could improve DC²². However, although polymerization duration was 15 minutes, being relatively longer than with the other two polymerization devices (4 minutes),

neither flexural strength and microhardness nor DC differed significantly from those of Powerlux.

The DC is one of the critical parameters that may influence the physical properties of resin composite materials and thus the clinical behaviour of restorations made of such materials. Surface hardness measurement is a simple technique that facilitates the evaluation of a large number of specimens. Although it was found to be a good predictor for resin conversion²⁴, it was also reported to be especially sensitive to small changes of the polymer cross-linking in areas of high conversion. In addition, it allows for measurements at specific locations within the specimen. According to Rueggeberg and Craig²⁵ (1988), even though hardness may not be useful for making direct comparisons among materials, it is a valuable tool for relative measurements within the same material and its simplicity makes it suitable for comparing different polymerization techniques. The Vickers microhardness is also dependent on the extension of the polymeric matrix polymerization and the quantity of inorganic fillers of the resin²⁵. Although the DC values were not significant, the group processed in Strobolux resulted in slightly lower, though not significantly different values for microhardness. According to Pianelli et al.²⁴ (1999), microhardness measurements should not be associated with the DC. Microhardness values may be valid for making comparisons within a given material but not among different materials. Thus, the results of this test should be considered complementary to the other tests employed in this study.

Mechanical property measurements (hardness, Young modulus) appear to be more sensitive than vibrational techniques for following slow changes in the DC, when the network is cross-linked.²⁴ This is why FT-Raman Spectroscopy offers a direct approach for determining the DC²⁶. Indeed, for methacrylate-based resins, this method allows for the evaluation of the DC conversion (i.e., the percentage of vinyl functions converted to aliphatic functions) by comparing the vibration bands of the residual unpolymerized methacrylate. The vibration band of the residual unpolymerized methacrylate C=C bond at 1640 cm⁻¹ with the aromatic C=C stretching band at 1610 cm⁻¹ are used as internal standards. Leung et al.²⁷ (1984) concluded that the best technique for evaluating the DC was FTIR (Fourier transformation infrared spectroscopy), even though the hardness measurement provides good

information. On the other hand, the study conducted by Rueggeberg and Craig²⁵ (1988) revealed that the hardness measurement is more problematic than FTIR for detecting the small changes in the DC, to follow the change occurring in the first stages of polymerization and after the network is cross-linked. Microhardness measurements are not more sensitive than FTIR to changes in DC in the early stages of polymerization because the material has no structural integrity at this point. One cannot test the hardness of a soft, initially polymerizing material until after gelation point is reached. In fact, FTIR is much more sensitive in the early time period.

In FTIR evaluations, it was found that the UEDMA/TEGDMA phase had a DC of 70% and superior wear resistance, while the Bis-GMA/TEGDMA had a DC of 55%¹. Monomer mixtures of Bis-GMA and TEGDMA give rise to polymers in which the quantity of remaining double bonds increases with the content of Bis-GMA, without the mechanical properties being significantly affected¹². The DC results obtained in this study were similar to those reported. The material studied was Sinfony, which is a flowable resin. In the study by Kakaboura, Rahiotis and Zinelis, et al.¹⁵ (2003), BelleGlass HP, a kind of IRC, exhibited significantly higher DC and mechanical properties than Sinfony. The results of DC values for Sinfony were higher than those reported by Kakaboura, Rahiotis and Zinelis et al.¹⁵ (2003), who found 66% DC. Also, Gohring, Galho and Luthy²⁸ (2005) studied several IRCs and found the lowest flexural strength with Sinfony. This was attributed to the lower

filler content (50%) of this IRC compared to other resins. Flowable resins are more likely to contain voids that might have impaired the flexural strength results²⁷. Other types of IRCs with more filler particles and different monomer matrices, either flowable or in paste forms, should be tested in future studies. Some of the currently available IRCs are polymerized in xenoscopic light polymerization devices with which higher DC is expected as a result of the heat, pressure and light used for processing. This causes improvement in mechanical strength and hardness⁸. Polymerization processes and their effect on the mechanical and chemical properties of IRCs show variations²⁹. Nevertheless, clinicians and dental technicians should not expect the same mechanical and chemical properties for the IRCs when they are polymerized in different polymerization devices. Although without significant differences, the IRC materials tested showed higher DC in other polymerization devices than the one (Visio Beta) originally designed for this material.

CONCLUSION

Flexural strength values were lower when they were polymerized in the Strobolux device than in the Powerlux and Visio Beta Vario devices. Considering all DC, flexural strength and Vicker's hardness results, the Powerlux device could be suggested for better polymerization and clinical outcome of the IRC tested. The type of polymerization device may affect the flexural strength and Vickers hardness of the IRC tested in varying degrees.

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