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## Conversion degree of indirect resin composites and effect of thermocycling on their physical properties

Souza, R O

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# Conversion Degree of Indirect Resin Composites and Effect of Thermocycling on Their Physical Properties

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## Keywords

Degree of conversion; flexural strength; indirect resin composites; Vicker's hardness.

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## Abstract

**Purpose:** This study evaluated the degree of conversion (DC) of four indirect resin composites (IRCs) with various compositions processed in different polymerization units and investigated the effect of thermal aging on the flexural strength and Vicker's microhardness.

**Materials and Methods:** Specimens were prepared from four IRC materials, namely Gr 1: Resilab (Wilcos); Gr2: Sinfony (3M ESPE); Gr3: VITA VMLC (VITA Zahnfabrik); Gr4: VITA Zeta (VITA Zahnfabrik) using special molds for flexural strength test (N = 80, n = 10 per group) (25 × 2 × 2 mm<sup>3</sup>, ISO 4049), for Vicker's microhardness test (N = 80, n = 10 per group) (5 × 4 mm<sup>2</sup>) and for DC (N = 10) using FT-Raman Spectroscopy. For both flexural strength and microhardness tests, half of the specimens were randomly stored in distilled water at 37°C for 24 hours (Groups 1 to 4), and the other half (Groups 5 to 8) were subjected to thermocycling (5000 cycles, 5 to 55 ± 1°C, dwell time: 30 seconds). Flexural strength was measured in a universal testing machine (crosshead speed: 0.8 mm/min). Microhardness test was performed at 50 g. The data were analyzed using one-way and two-way ANOVA and Tukey's test ( $\alpha = 0.05$ ). The correlation between flexural strength and microhardness was evaluated with Pearson's correlation test ( $\alpha = 0.05$ ).

**Results:** A significant effect for the type of IRC and thermocycling was found ( $p = 0.001$ ,  $p = 0.001$ ) on the flexural strength results, but thermocycling did not significantly affect the microhardness results ( $p = 0.078$ ). The interaction factors were significant for both flexural strength and microhardness parameters ( $p = 0.001$  and 0.002, respectively). Thermocycling decreased the flexural strength of the three IRCs tested significantly ( $p < 0.05$ ), except for VITA Zeta (106.3 ± 9.1 to 97.2 ± 14 MPa) ( $p > 0.05$ ) when compared with nonthermocycled groups. Microhardness results of only Sinfony were significantly affected by thermocycling (25.1 ± 2.1 to 31 ± 3.3 Kg/mm<sup>2</sup>). DC values ranged between 63% and 81%, and were not significantly different between the IRCs ( $p > 0.05$ ). While a positive correlation was found between flexural strength and microhardness without ( $r = 0.309$ ) and with thermocycling ( $r = 0.100$ ) for VITA VMLC, negative correlations were found for Resilab under the same conditions ( $r = -0.190$  and  $-0.305$ , respectively) (Pearson's correlation coefficient).

**Conclusion:** Although all four IRCs presented nonsignificant DC values, flexural strength and microhardness values varied between materials with and without thermocycling.

Photo-activated resin composites are commonly used restorative materials in dentistry for both anterior and posterior restorations. Such tooth-colored restorations can adhere to the dental tissues, and they can be made directly or indirectly at chairside or at the dental laboratory. One drawback of direct application of resin composites is the polymerization shrinkage that influences the stress produced at the interface between the dental tissues, leading to marginal gaps or hypersensitivity when the stress exceeds the bond strength between the resin composite and the tooth.<sup>1</sup> Conversely, resin composite restorations built using indirect techniques result in lower polymerization shrinkage, reducing the stress between the tooth and the resin cement, avoiding postoperative sensitivity, with lower water sorption and, therefore, discoloration.<sup>1</sup> Also with indirect resin composites (IRCs), less finishing and polishing time is required at chairside. IRCs do not require high technical skills, occlusal anatomy and proximal contacts can be established by the laboratory technician, and IRCs can be repaired when needed.<sup>2-6</sup>

IRC materials are usually classified according to the size of their inorganic particles; hybrid IRCs contain particles greater than 1.0  $\mu\text{m}$ , microhybrid IRCs have particles smaller than 1.0  $\mu\text{m}$ , and nano-hybrid IRCs have particles smaller than 0.4  $\mu\text{m}$ . The increase in the size and the volume of inorganic particles improves their resistance to wear, decreases the polymerization contraction, and also increases the glaze and polishability, which favors the esthetics.<sup>2,5</sup>

The degree of conversion (DC) has a significant influence on the physical<sup>5</sup> and biological properties of resin composite restorations. DC is highly dependent on factors like composition of the material,<sup>7,8</sup> color and translucency,<sup>9,10</sup> distance of the light tip to the surface, and the irradiance of the polymerization lamp.<sup>11,12</sup> In this context, IRCs allow for higher DC, as polymerization is carried out in the laboratory or at chairside in special photo-polymerization units in which all surfaces of the restoration can be polymerized. Depending on the type of the polymerization unit, combination of light, heat, vacuum, and pressure result in an improvement from 10% to 20% in the mechanical properties of these materials as opposed to the direct polymerization techniques. Unfortunately, with the increasing number and improved properties of IRCs, dental technicians and some clinicians have to invest not only in the IRC material itself, but also on polymerization lamps. Polymerization modes also show variations between several devices.

Many studies have been accomplished with the purpose of evaluating the mechanical properties of IRCs,<sup>10-17</sup> especially with mechanical tests such as flexural strength and microhardness.<sup>10</sup> The DC of monomers to polymers in dental resins has been evaluated using microhardness tests.<sup>9,15</sup> While there is still no consensus in the dental literature as to which method should be used for the assessment of the DC, limited information is available on the mechanical properties of IRCs in aged conditions.<sup>18</sup> In fact, like all other restorative materials, IRCs are also subjected to temperature variations in the oral cavity induced by diet. Temperature elevations and water uptake could cause degradation of the monomer matrix and filler/matrix interface<sup>18</sup> and consequently influence the strength of the IRCs. Hardness of the resin composites is also influenced by the type of light polymerization device.<sup>19</sup>

Flexural strength and fracture toughness tests are the most frequently used methods to evaluate the mechanical behavior of resin-based materials.<sup>17,18,20-22</sup> Flexural strength tests can represent the dynamic nature of the existent stresses during mastication, creating different tensile, compression, and shear stresses upon fixed partial dentures (FPDs).<sup>17,18,22</sup>

This study evaluated the DC of four IRCs with various compositions polymerized in different devices, and also investigated the effect of thermal aging on the flexural strength and Vicker's microhardness of such composites. The null hypothesis tested was that there would be no difference between the mechanical properties and DC of IRCs before and after aging.

## Materials and methods

The types, brand names, main compositions, corresponding polymerization modes, batch numbers, manufacturers, and the shades of the four IRCs used for the experiments are presented in Table 1.

### Flexural strength test

IRC specimens ( $N = 80$ ,  $n = 10$  per group) were prepared in accordance with ISO 4049<sup>23</sup> using a polyethylene mold ( $25 \times 2 \times 2 \text{ mm}^3$ ). They were polymerized according to each manufacturer's recommendation and wet-ground finished using silicone carbide papers up to 1200 grit. The specimens were then randomly divided into two groups. While half of the specimens were stored in distilled water at 37°C for 24 hours (Groups 1 to 4), the other half (Groups 5 to 8) were submitted to thermocycling (5000 cycles, 5 to 55  $\pm$  1°C, dwell time: 30 seconds, transfer time from one bath to the other: 2 seconds) (Nova Etica, São Paulo, Brazil).

The flexural strength tests were then performed in a universal testing machine (Model DL-1000, EMIC Equipments and Systems Ltd, Sao Jose dos, Brazil) where the load was applied at a constant transverse speed of 0.8 mm/min until fracture. The formula in ISO 4049<sup>23</sup> was followed for the calculation of the data obtained from the flexural strength test. The load that led to initial fracture of the specimens was obtained with the load cell having 50 kgf capacity according to the following equation, where  $P$  is the maximum load at fracture (N),  $l$  the distance between two parallel supports (20 mm),  $b$  width, and  $d$  thickness of the specimen (mm):

$$\Sigma = \left( \frac{3PL}{2bd^2} \right)$$

### Vicker's microhardness test

Disc-shaped IRC specimens (diameter: 5 mm; thickness: 4 mm) ( $N = 80$ ) were prepared using a metal mold according to the polymerization process as described in Table 1. The specimens were embedded in acrylic resin blocks, and each block was finished with wet silicone carbide papers up to 1200 grit and polished in a polishing machine (Struers, Model DP 10, Panambra Ind. & Tec. S.A., São Paulo, Brazil) with diamond paste (3  $\mu\text{m}$ ). While half the specimens were randomly stored in distilled water at 37°C for 24 hours (Groups 1 to 4), the other half

**Table 1** Indirect composite resins tested

Type	Brand name	Volume % inorganic fillers	Main composition	Polymerization mode	Batch number	Manufacturer	Shade
Microhybrid	Resilab	53%	Bis-GMA, UDMA, TEGMA, Aluminium Borosilicate, acid silicon of high dispersal, photoinitiators, inhibitors and pigments	First cycle: Polymerization for 4 min in the EDG-Lux unit (400–500 mW/cm <sup>2</sup> ) Second cycle: Polymerization for 8 min in the EDG-Lux unit (400–500 mW/cm <sup>2</sup> )	156/05	Wilcos do Brasil Ltd., Petrópolis, Brazil	A2
Microhybrid	Sinfony	50%	HEMA and 10% to 30% (octahydro-4,7-methano-1H-indenediy) bis(methylene)diacrylate), strontium-aluminium borosilicate glass, silicon oxide, silane and photoinitiators	First cycle: Polymerization for 15 s in the Visio Alpha unit (400 mW/cm <sup>2</sup> ) Second cycle: Polymerization for 15 min in the Visio Beta unit (470 mW/cm <sup>2</sup> )	203216	3M ESPE, St Paul, MN	A2
Microfilled	VITA VMLC	46%	UDMA, TEGMA, Silica, primary particle (40–50 nm)	First cycle: Polymerization for 3 min in the Spectramat unit (350–500 mW/cm <sup>2</sup> ) Second cycle: Polymerization for 5 min in the Spectramat unit (350–500 mW/cm <sup>2</sup> )	7677	Vita Zahnfabrik, Bad Säckingen, Germany	A2
Microhybrid	VITA Zeta	44%	UDMA, TEGMA, Silica and feldspar	First cycle: Polymerization for 3 min in the Spectramat unit (350–500 mW/cm <sup>2</sup> ). Second cycle: Polymerization for 5 min in the Spectramat unit (350–500 mW/cm <sup>2</sup> )	7288	Vita Zahnfabrik, Bad Säckingen, Germany	A2

Bis-GMA = Bis-phenol-A-glycidylmethacrylate; UDMA = Urethane dimethacrylate; TEGMA = Triethyleneglycol methacrylate; HEMA = 2-hydroxyethyl methacrylate.

(Groups 5 to 8) were submitted to thermocycling [5000 times, 5 to 55 ± 1°C, dwell time: 30 seconds, transfer time from one bath to the other: 2 seconds (Nova Etica)].

The microhardness measurements were made employing a Vicker's microhardness testing device (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 on each specimen using the following equation, where P was the applied load in Kg, and *dv* was the average of the diagonal length in mm:<sup>20</sup>

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

### Degree of conversion

Disc-shaped IRC specimens (diameter: 5 mm; thickness: 4 mm) (N = 40) were prepared as described above. The specimens were stored in distilled water at 37°C for 24 hours and embed-

ded in acrylic resin blocks. Each block was finished with wet silicone carbide papers up to 1200 grit and polished (Struers, Model DP 10) with diamond paste (3 μm).

The surfaces were analyzed by FT-Raman Spectroscopy 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 to 4 cm<sup>-1</sup>. 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 specimen. The uncured resin was positioned on an aluminium rod in a specimen holder mounted on an optical rail for spectrum collection. For the 80 cured specimens, three spectra of the top surface and another three spectra of the bottom surface were collected, resulting in a total of 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<sup>-1</sup>. The Raman peaks corresponding to the vibrational stretching

modes at 1610 and 1640  $\text{cm}^{-1}$  were fitted in Gaussian shapes to obtain the height of the peaks by software (Microcal Software Inc, Northampton, MA). A comparison of the height ratio of the aliphatic carbon-carbon double bond (C=C) at 1640  $\text{cm}^{-1}$  with that of the aromatic component at 1610  $\text{cm}^{-1}$  for the cured and uncured conditions was performed to estimate the DC using the following equation:

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

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

The aromatic C=C peak at 1610  $\text{cm}^{-1}$  originated from the aromatic bonds of the benzene rings in the monomer molecules, and its intensity remained 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 determined by band height at 1640  $\text{cm}^{-1}$ /band height at 1610  $\text{cm}^{-1}$ .<sup>17</sup>

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

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

### 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). The means of each group from flexural strength and microhardness measurements were analyzed by two-way ANOVA, with the test type as the dependent variable and IRC types as the independent factors. *P* values less than 0.05 were considered to be statistically significant in all tests. Multiple comparisons were made by Tukey's adjustment test. DC values were analyzed using one-way ANOVA. The correlation between flexural strength and microhardness measurements was evaluated using Pearson's correlation test ( $p < 0.05$ ).

### Results

The type of IRC and thermocycling had a significant effect ( $p = 0.001$ ,  $p = 0.001$ , respectively) on the flexural strength results, but thermocycling did not significantly affect the microhardness results ( $p = 0.078$ ). The interaction factors for both flexural strength and microhardness parameters were significant ( $p = 0.001$ ,  $p = 0.002$ ) (Tables 2 and 3).

#### Flexural strength test

After thermocycling, the flexural strength of the three IRCs tested decreased [(Sinfony:  $177.1 \pm 45.4$  to  $127.2 \pm 15.1$ , Resilab:  $200 \pm 42$  to  $112 \pm 17.1$ , VITA VMLC:  $147.4 \pm 28$  to  $77 \pm 19$  MPa)] significantly ( $p < 0.05$ ) except for VITA Zeta ( $106.3 \pm 9.1$  to  $97.2 \pm 14$  MPa) ( $p > 0.05$ ) (Table 4, Fig 1).

**Table 2** Two-way ANOVA for the indirect resin composite types and the interaction terms after flexural strength test, (\* $p < 0.05$ )

Effect	DF	SS	MS	F	<i>p</i>
Resins	3	45,211	15,070	21.02	0.001*
Thermocycling	1	59,205	59,205	82.58	0.001*
Interaction	3	17,282	5761	8.04	0.001*
Residue total	72	51,618	717		
Total	79	173,315			

#### Vicker's microhardness test

The microhardness results of Sinfony were significantly affected by thermocycling ( $25.1 \pm 2.1$  to  $31 \pm 3.3$  Kg/mm<sup>2</sup>) (Table 5, Fig 2).

#### Degree of conversion

DC of all four IRCs did not show significant differences between the experimental groups ( $75 \pm 2$ ;  $75 \pm 11$ ;  $63 \pm 10$ ;  $81 \pm 12\%$  for Resilab, Sinfony, Vita VMLC, and Vita Zeta, respectively) ( $p > 0.05$ ) (one-way ANOVA). Micro-Raman spectra of the IRCs are presented in Figure 3.

The correlation between flexural strength and microhardness was evaluated. The Pearson's correlation coefficient (*r*) can range from  $-1$  to  $1$ , inclusive. Positive values indicate a positive correlation, negative values indicate negative correlation, and when *r* is 0, there is no correlation between the factors. Additionally, *r* values closer to  $+1$  or  $-1$  indicate strong positive or negative correlation, respectively, whereas *r* values further away from  $+1$  or  $-1$  indicate weak positive and negative correlation, respectively. While a positive correlation was found between flexural strength and microhardness values without ( $r = 0.309$ , weak correlation) and with thermocycling ( $r = 0.100$ , weak correlation) for VITA VMLC, weak negative correlations were found for Resilab in the same conditions ( $r = -0.190$  and  $-0.305$ , respectively) (Pearson's correlation coefficient)(Fig 4).

### Discussion

Evolution in the field of polymer technology has made the fabrication of metal-free restorations possible using IRCs, particularly due to the improvement in their physical properties.<sup>24</sup> Flexural strength values obtained from the IRCs tested in this study (106 to 200 MPa) without thermocycling were similar to

**Table 3** Two-way ANOVA for the indirect resin composite types and the interaction terms after Vicker's microhardness test, (\* $p < 0.05$ )

Effect	DF	SS	MS	F	<i>p</i>
Resins	3	8434.5	2811.5	202.38	0.001*
Thermocycling	1	44.4	44.4	3.20	0.078
Interaction	3	235.0	78.3	5.64	0.002*
Residue total	72	1000.2	13.9		
Total	79	9714.2			

**Table 4** The mean ( $\pm$  standard deviation) flexural strength values (MPa) for indirect resin composites before and after thermocycling. \*The same superscript letters indicate no significant differences (Tukey's test,  $p < 0.05$ )

Experimental groups	Thermocycling	
	Without	With
Resilab (Groups 1 and 5)	200 $\pm$ 42 <sup>a</sup>	112 $\pm$ 17.1 <sup>c,d,e</sup>
Sinfony (Groups 2 and 6)	177.1 $\pm$ 45.4 <sup>a,b</sup>	127.2 $\pm$ 15.1 <sup>c,d</sup>
VITA VMLC (Groups 3 and 7)	147.4 $\pm$ 28 <sup>b,c</sup>	77 $\pm$ 19 <sup>f</sup>
VITA Zeta (Groups 4 and 8)	106.3 $\pm$ 9.1 <sup>d,e</sup>	97.2 $\pm$ 14 <sup>d,e</sup>
Mean	158 $\pm$ 48.3	103.2 $\pm$ 24.4

those observed in other studies on IRCs (120 to 160 MPa for ArtGlass, Heraeus-Kulzer and Targis, Ivoclar Vivadent).<sup>18,25-27</sup> Such resin composites are examples of second-generation IRCs, and the flexural strength values make their indication possible for inlays, onlays, FPDs, and FPDs reinforced with fibers.<sup>28</sup> According to the International Organization for Standardization (ISO 4049),<sup>23</sup> IRCs should present a minimum flexural strength of 100 MPa in order to be indicated for such restorations. The results of our study exceeded the value required by ISO 4049, regardless of the IRCs and polymerization units used; however, three of the tested IRCs, (Resilab, Sinfony, VITA VMLC) showed a significant decrease in flexural strength values after 5000 thermocycles. Therefore, the hypothesis that hydrothermal aging would decrease the flexural strength of the IRC could only be partially accepted. High or elevated temperatures are known to weaken the resin-based materials.<sup>16,29</sup>

Thermocycling is a combination of hydrolytic and thermal degradation and a method to simulate temperature-related breakdown by repeated sudden temperature changes. Particularly in the case of Sinfony, a decrease in the flexural strength after thermocycling was noticed in almost 50% of specimens. Although the curing time for Sinfony was longer (15 minutes) than those of the other IRCs (8 to 12 minutes), lower results were achieved, which could be due to the differences in monomer types of the IRCs. While Sinfony contains HEMA at 10% to 30% (octahydro-4,7-methano-1H-indenediyl)bis(methylene)diacrylate, the other three IRCs con-

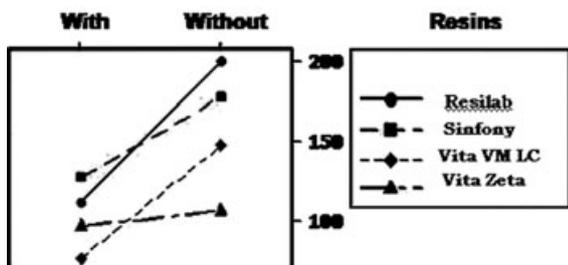
**Table 5** The mean ( $\pm$  standard deviation) Vicker's microhardness values (Kg/mm<sup>2</sup>) for indirect resin composites before and after thermocycling. \*The same superscript letters indicate no significant differences (Tukey's test,  $p < 0.05$ )

Experimental groups	Thermocycling	
	Without	With
Resilab (Groups 1 and 5)	53 $\pm$ 6 <sup>a</sup>	55 $\pm$ 6.4 <sup>a</sup>
Sinfony (Groups 2 and 6)	25.1 $\pm$ 2.1 <sup>c</sup>	31 $\pm$ 3.3 <sup>b</sup>
VITA VMLC (Groups 3 and 7)	34.4 $\pm$ 2.3 <sup>b</sup>	30.5 $\pm$ 3.1 <sup>b</sup>
VITA Zeta (Groups 4 and 8)	30 $\pm$ 2 <sup>b,c</sup>	32 $\pm$ 2 <sup>b</sup>
Mean	35.4 $\pm$ 11.1	36.92 $\pm$ 11.2

tain some UDMA in their composition. The results obtained with IRCs having UDMA are comparable to those reported in Yamaga et al's study.<sup>10</sup> In that study, even higher hardness and fracture toughness values were found with IRCs containing four-functional urethane methacrylate (UTMA) than those of two-functional UDMA. In fact, it has already been shown that increased contents of UDMA increase the flexural strength of experimental resins.<sup>30</sup> In Kakaboura et al's study,<sup>24</sup> BelleGlass HP exhibited significantly higher DC and mechanical properties compared to Sinfony. Also, Gohring et al<sup>31</sup> studied several IRCs and found the lowest flexural strength with Sinfony. Based on these previous observations and ours, it seems that Sinfony composition plays a major role in its flexural strength. In this case, increasing the light-exposure time could be a solution to obtaining a higher DC.<sup>32</sup> Furthermore, because it is flowable, Sinfony is more likely to contain voids that might influence the flexural strength results.<sup>33</sup> In this context, besides requiring the IRC compositions and their corresponding polymerization device, ISO requirements should also take aging factors into consideration. It can be anticipated that, depending on the number of thermal cycles, more degradation on the resins is likely to occur.

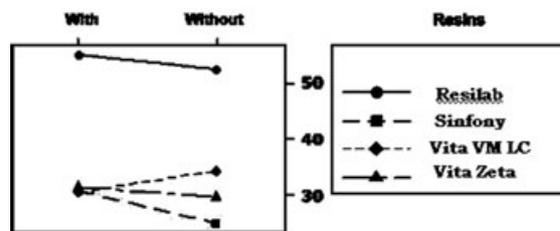
Surface hardness measurement is a simple technique that facilitates the evaluation of a large number of specimens. Although it was found to be a good predictor for resin conversion, it was also reported to be especially sensitive to small changes of the polymer cross-linking in areas of high conversion.<sup>15</sup> Even though hardness values may not be used for a direct comparison among materials, they are valuable tools for relative measurements within the same material, and their simplicity makes

**Flexural Strength (MPa)**



**Figure 1** Flexural strength test results with and without thermocycling for the indirect resin composites tested.

**Vicker's microhardness (kg/mm<sup>2</sup>)**



**Figure 2** Vicker's microhardness results with and without thermocycling for the indirect resin composites tested.

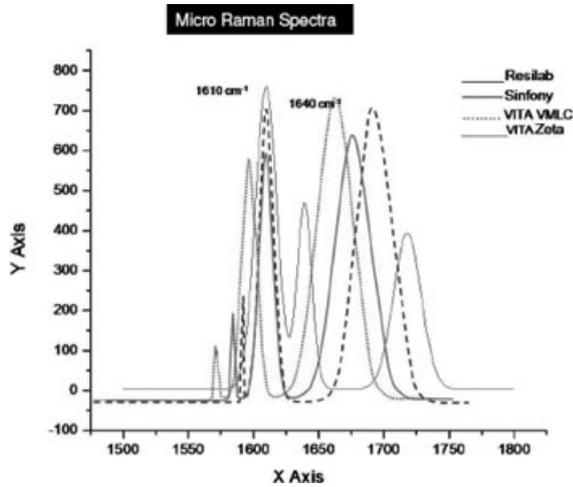


Figure 3 Micro-Raman spectra of the indirect resin composites tested.

them suitable for comparison between different polymerization techniques.<sup>34</sup>

Vicker’s microhardness is dependent on the extension of the polymeric matrix polymerization and the quantity of inorganic fillers of the resin.<sup>35</sup> Although the DCs were not significantly

different, there were significant differences between the microhardness values, again leading to partial acceptance of the first hypothesis. Despite the fact that the polymerization devices employed had similar light output, only VITA Zeta and VITA VMLC (similar monomer matrices, polymerized under the same conditions) did not show significant differences in microhardness. A previous study showed that the filler content in the IRC tended to be linearly proportional to both hardness and fracture toughness.<sup>10</sup> The filler content in volume percentage was similar in all materials tested. In this study, the differences in microhardness cannot be attributed to differences in DC, as it was not significantly different between the groups. This strengthens the fact that mechanical properties do not always work as a function of DC, which requires further investigation.

The differences in microhardness obtained with three IRCs polymerized with three different light sources are best explained by the polymerization mode of the equipment used or the monomer matrix type. After thermocycling, the mean microhardness values of other IRCs were not significantly changed, but interestingly, only Sinfony showed significantly higher results. It may be that dwelling at 55°C water bath during thermocycling contributed to further polymerization of the surface, initially leading to increased hardness. Prolonged thermocycling may lead to water absorption by a diffusion-controlled process, and it causes leaching of unreacted monomers and

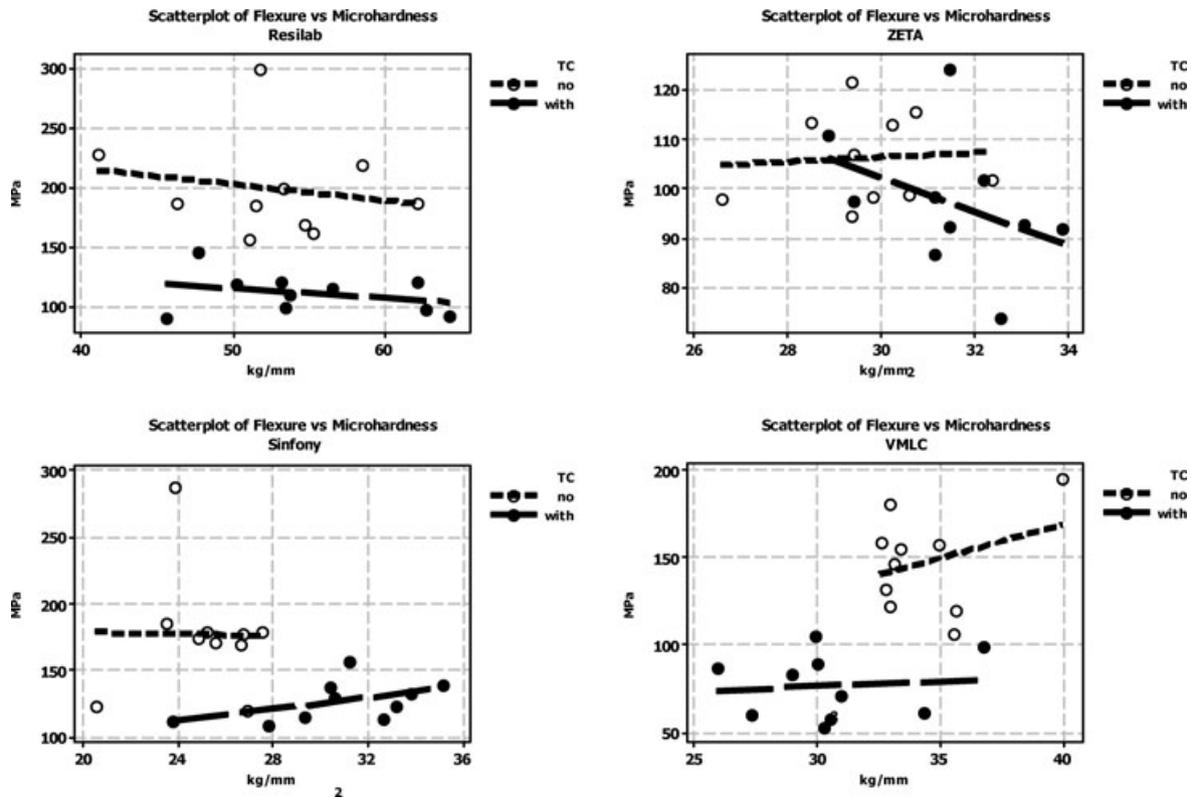


Figure 4 Scatter plot of correlation values between flexural strength (MPa) and Vicker’s microhardness (Kg/mm<sup>2</sup>) for Resilab ( $r = -0.190, p = 0.600$ ;  $r = -0.305, p = 0.391$ ); VITA Zeta ( $r = 0.077, p = 0.832$ ;  $r = -0.394, p = 0.260$ ); Sinfony ( $r = -0.016, p = 0.965$ ;  $r = 0.477, p = 0.163$ ); VITA VMLC ( $r = 0.309, p = 0.385$ ;  $r = 0.100, p = 0.783$ ) without and with thermocycling (TC), respectively.

swelling of the matrix. Water acts like a plasticizer and thereby weakens the polymer structure. It also degrades the matrix/filler interface directly by hydrolytic breakdown of the silane/filler interface and the surface of the filler particles.<sup>36</sup> Unfortunately, the minimal number of thermocycles necessary for plasticization is not known.

While a positive but weak correlation was found between flexural strength and microhardness for VITA VMLC with and without thermocycling, negative correlations were found for Resilab under the same conditions. The findings of this study need to be confirmed on a larger number of specimens. Measurements of behavior (hardness, Young modulus) are more sensitive than spectroscopy for following slow changes in the DC when the network is fully cross-linked in aging experiments. Leung *et al*<sup>33</sup> concluded that the best technique to evaluate the DC was Fourier transformation infrared spectroscopy (FTIR), even though the hardness measurement provided good information. On the other hand, the study conducted by Rueggeberg and Craig<sup>35</sup> revealed that the hardness measurement is more sensitive than FTIR for detecting the small changes in the DC and following the change occurring in the first stages of polymerization after the network is cross-linked.

In FTIR evaluations, it was found that UEDMA/TEGDMA phase had a DC of 70% and exhibited superior wear resistance, while Bis-GMA/TEGDMA had a DC of 55% when conventional resin composites were used.<sup>37</sup> 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 affected.<sup>38</sup> The DC results obtained in this study with the tested IRCs were slightly higher than those reported previously with the conventional resin composites for direct applications. There were variations between the DC values of the IRCs, but the results were not statistically significant. Currently available IRCs are mostly polymerized by xenoscopic light devices in which a higher DC is expected as a result of heat and light combination.<sup>25</sup> Therefore, polymerization processes and their effect on the mechanical and chemical properties of IRCs show variations, and this should be taken into consideration when choosing IRCs for clinical applications.<sup>39</sup>

## Conclusions

From this study, the following could be concluded:

1. The degree of polymer crosslink assessed by FT-Raman Spectroscopy did not correspond to the resulting mechanical behavior of the materials tested.
2. Although the microhardness of Sinfony was affected by thermocycling, 5000 thermocycles did not decrease the microhardness of the majority of the IRCs. On the contrary, thermocycling decreased the flexural strength of most IRCs studied, except for VITA Zeta.
3. IRCs designed for the same applications may present different mechanical behavior.

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