



DEGREE OF CONVERSION OF INDIRECT COMPOSITE RESIN UNDER FOURIER TRANSFORM INFRARED SPECTROSCOPY - AN IN VITRO STUDY

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Article Info	ABSTRACT
<p>Received 30/10/2015 Revised 05/11/2015 Accepted 07/11/2015</p>	<p>To evaluate the degree of conversion (DC) of the indirect composite resin (IRC) materials, seeking how the combination of time and power of curing applied during polymerization, as well as the temperature of the light-curing composite, influenced the degree of conversion (DC) under Fourier transform infrared spectroscopy (F.T.I.R spectroscopy). SR Adoro (Ivoclar Vivadent), second generation microfilled indirect composite resin. Indirect composite resin specimens (diameter: 5 mm; thickness: 4 mm) (N = 40) (cured and uncured) will be prepared. The surfaces will be analyzed by Fourier transform infrared spectroscopy. The DC results obtained in this study with the tested IRCs were slightly higher (67.91%) than those reported previously with the conventional resin composites for direct applications. Within the limitations of this study it can be concluded that the degree of conversion of indirect composites is slightly higher (67.91%) than those reported previously with the conventional resin composites for direct applications. The degree of conversion (DC) of direct composite resin ranges from 55% to 65% (11) and further studies are required to derive more conclusive results.</p>
<p>Key words: Indirect composite resin, Degree of conversion,</p>	

INTRODUCTION

Dental composite formulations have been continuously evolving ever since Bis-GMA was introduced to dentistry by Bowen in 1962. Recent developments in material science technology have considerably improved the physical properties of resin-based composites and expanded their clinical applications. Dental restorative composite materials can be divided into direct and indirect resin composites (IRC). IRCs are also referred to as prosthetic composites or laboratory composites. These materials offer an esthetic alternative for large posterior restorations. There are a plethora of materials available nowadays [1].

Morphological and functional restoration of compromised teeth can be achieved by means of a direct or indirect restoration. The need for amalgam alternatives and the demand for aesthetic restorations have lead to increased

popularity of resin composite restorations for posterior teeth. 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 [2].

Light activated direct composite resin restorative materials have revolutionized clinical adhesive dentistry. Composite restorations are popular as compared to other restorations due to the following advantages –i) they are esthetic. ii) Conservative of tooth structure removal. iii) Less complex when preparing the tooth. iv) Insulative, having low thermal conductivity and v) Bonded to tooth structure, resulting in good retention, low microleakage, minimal interfacial staining, and increased strength of remaining tooth structure [3].



Direct composites resins are among the most widely used materials in restorative dentistry. These materials are indicated for use as follows: i) Class I, II, III, IV, V, and VI restorations. ii) Foundations or core buildups. iii) Sealants and prevented resin restorations. iv) Esthetic enhancement procedures- partial veneers, full veneers, tooth contour modifications & diastema closures. V) Temporary restorations and vi) periodontal splinting [3].

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. This promotes micro-leakage, which can lead to secondary caries, pulp irritation, postoperative sensitivity and marginal discoloration [2]. Another important clinical consideration regarding the effects of polymerization shrinkage is the configuration factor (C-factor). The C-factor is the ratio of bonded surfaces to the unbonded or free surfaces in a tooth preparation. The higher the C-factor, the greater is the potential for bond disruption from polymerization effects [3].

The process of adhesion, conditioning the tooth surface, application of primers and adhesives, followed by the layering of composite resins to complete the restorative process, involves many independent steps that must be properly executed to ensure good results. This discussion covers some of the latest advances in composite materials and techniques.

- Self – adhering flowable composites: Vertise Flow (combines the adhesive and flowable steps). Brushing this first layer of material vigorously into the tooth surface enhances this penetration into the smear layer and creates a bond that is comparable to that achieved by seventh generation dentin bonding agents.
- Highly filled flowable resins: several more highly filled flowable composites are available to restore conservative class I to V cavities. Material such as G-aenial Universal Flo (GC America), Grandio SO (VOCO America) are examples of products that are designed with the physical properties and polish retention capabilities of most nano- microhybrid materials designed to withstand the occlusal forces generated in the posterior areas of the mouth.
- Bulk –fill flowables as “dentin replacements”: A class of newer flowable composites (eg. SureFil SDR, Bulk Fill Flowable Base) is indicated for use as a bulk-fill base (dentin replacement) beneath posterior composite restorations and can be bulk filled in layers up to 4 mm in depth. It increased depth of cure, a viscosity that will readily adapt to the internal walls of the cavity without the need for manipulation of the material and low polymerization shrinkage stress.
- Sonic delivery of composite resin: The sonic energy generated by the handpiece causes a change in the

viscosity of the composite resin, so that during placement, it behaves like a flowable liner in its ability to adapt to the internal surface of the cavity preparation. Unique property of SonicFill is that it has a polymerization shrinkage of about 1.6% and can be bulk filled to a depth of 5 mm while still having a greater than 97% full cure at its deepest point.

- Giomer- A class of “Smart” Composite: Giomer products are characterized by their surface pre-reacted glass (S-PRG) filler. S-PRG fillers have a glass core that is pre-reacted with a polyacrylic acid solution. The glass-ionomer phase in Giomer fillers are protected from water sorption and material degradation by a surface-modified layer.

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, discoloration. Also with indirect resin composites (IRCs), less finishing and polishing time is required at chairside. Occlusal anatomy and proximal contacts can be established by the laboratory technician, and IRCs can be repaired when needed [2].

IRC materials are usually classified according to the size of their inorganic particles; hybrid IRCs contain particles greater than 1.0 μm , microhybrid IRCs have particles smaller than 1.0 μm , and nano-hybrid IRCs have particles smaller than 0.4 μm . 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 [2].

Adequate polymerization transforms the monomers into a complex polymer structure. Monomer conversion into polymers does not attain 100%, but results in monomers that remain unreacted. Resin composites start the polymerization process by absorbing light in a specific range of wavelength around 400-500 nm; once activated, react with the aliphatic amine to produce free radicals. The number of double carbon links (C=C) present in the monomers, which are converted into single links (C-C) to form the polymer chains during the polymerization process, is called degree of conversion [4].

The percentage of carbon-carbon bonds converted to single bonds to form a polymeric resin, also the percentage of polymerized methacrylate groups. The higher the degree of conversion (DC), the better the strength, wears resistance and many other properties essential to resin performance [5].

A conversion of 50% to 60%, typical of highly cross-linked bis-GMA- based composites, implies that 50% to 60% of the methacrylate groups have polymerized. However, this does not imply that 40% to 50% of the monomer molecules are left in the resin because one of the two methacrylate groups per dimethacrylate molecule could still have reacted and could be covalently bonded to the polymer structure, forming a pendant group [5].



The degree of conversion (DC) has a significant influence on the physical and biological properties of resin composite restorations. There are several contributing factors that can influence the degree of conversion (DC) such as composition of the material, color and translucency, distribution and quantity of inorganic fillers, the type and quantity of the photo-initiator, distance of the light tip to the surface and the irradiance of the polymerization lamp, light source used, power density, wavelength, light tip size, photo-activation method [4].

The Physical and mechanical properties of dental composites are directly influenced by the degree of conversion achieved during polymerization. Lower degree of conversion provides composites with an inferior mechanical properties and greater discoloration and degradation and as a result, restorations with poor wear resistance and poor color stability [4].

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 [2].

Many studies have been accomplished with the purpose of evaluating the mechanical properties of IRCs, especially with mechanical tests such as flexural strength and microhardness. The degree of conversion (DC) of monomers to polymers in dental resins has been evaluated using microhardness tests [5]. 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 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 and consequently influence the strength of the IRCs [6]. Hardness of the resin composites is also influenced by the type of light polymerization device.

Flexural strength, fracture toughness, degree of conversion tests are the most frequently used methods to evaluate the mechanical behavior of resin-based materials. 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) [7, 8].

The effectiveness of cured composite can be assessed by direct and indirect methods.

Direct methods that assess the degree of conversion are:

- Fourier transformation infrared spectroscopy
- Laser Raman spectroscopy
- Differential thermal analysis
- Magnetic resonance microimaging

Indirect methods to find the degree of conversion are:

- Calorimetric investigation
- Scraping method
- Dye method
- Surface microhardness

Micro hardness can be used as one of the indicators for the completeness of polymerization since the hardness of a polymer is directly related to its degree of cure. Vicker hardness is a suitable method for measuring the surface hardness of a restorative dental material [9].

Mechanical property measurements (hardness, Young modulus) appear to be more sensitive than vibrational techniques for following slow changes in the degree of conversion (DC), when the network is cross-linked. This is why FTIR Spectroscopy offers a direct approach for determining the degree of conversion (DC).

Among the several methods to determine the degree of composite is Fourier transform infrared spectroscopy (F.T.I.R spectroscopy). F.T.I.R spectroscopy has been proved to be a widely used reliable method. This method detects the C=C stretching vibration directly before and after polymerization of composite resin [10].

The aim of this study is to evaluate the degree of conversion (DC) of indirect composite resin under Fourier transform infrared spectroscopy (F.T.I.R spectroscopy).

This in vitro study evaluated the degree of conversion (DC) of the indirect composite resin materials employed in this procedure, seeking how the combination of time and power of curing applied during polymerization, as well as the temperature of the light-curing composite, influenced the degree of conversion (DC) under Fourier transform infrared spectroscopy (F.T.I.R spectroscopy).

MATERIALS AND METHODS

Materials

SR Adoro (Ivoclar Vivadent), second generation microfilled indirect composite resin.

Model Separator- Polyglycol, polyethylene glycol in a water/alcohol solution.

Link- Dimethacrylate, phosphate ester, solvent and benzoyl peroxide

Opaquer- Dimethacrylate (> 55 wt. %); inorganic fillers (43 wt. %). Additional contents are catalysts, stabilizers and pigments (< 2.5 wt %).

Layering materials (Dentin body, Transpa)- Dimethacrylate (17–19 wt.%); copolymer and silicon dioxide (82–83 wt.%). Additional contents are catalysts, stabilizers and pigments (<1 wt %). The total content of inorganic fillers is 64–65 wt. %/46–47 vol. %. Particle size 10-100 nm.





SR Adoro (Ivoclar Vivadent), second generation microfilled indirect composite resin

SAMPLE DESIGN

A white transparent hollow tube will be taken (5 mm in diameter), cut by Bard-Parker blade at length of 4 mm.

Model separator will be added with a brush Curing for 2-4 minutes (400-500mW/cm²)

Sr link (margin separator) added with a brush

Dentin body mixed with spatula and added Curing for 2-4 minutes (400-500mW/cm²)

Transpa mixed with spatula and added Curing for 2-4 minutes (400-500mW/cm²)

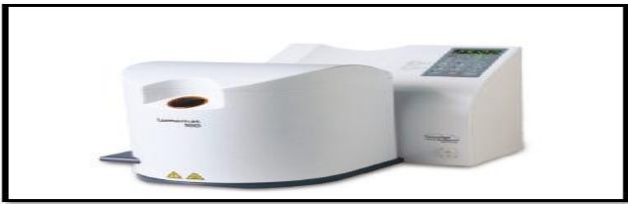
Final curing (lumamat 100) {Firing 26 minutes {light and heat}}

Copping done

Finishing and polishing done with polishing wheels and points

Glazing done with SR Gel Firing for 16 minutes {light and heat}

Sample will be prepared Other samples will be prepared in the same manner.



LUMAMAT 100

METHODS

For cured sample

Indirect composite resin specimens (diameter: 5 mm; thickness: 4 mm) (N = 20) will be prepared as described above.

The surfaces will be analyzed by Fourier transform infrared spectroscopy to evaluate the degree of conversion of indirect composite resins.



SAMPLES

For uncured sample:

Indirect composite resin specimens (diameter: 5 mm; thickness: 4 mm) (N = 20) will be prepared.

FT-IR spectra of uncured specimen are recorded under (F.T.I.R spectroscopy).



F.T.I.R SPECTROSCOPY

PARAMETERS TO BE STUDIED

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 to estimate the degree of conversion (DC) using the following equation:

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

$$R_{\text{polymerized}} = \frac{\text{Peak at } 1640 \text{ cm}^{-1}}{\text{Peak at } 1610 \text{ cm}^{-1}}$$

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 remained unchanged during the polymerization reaction. The mean value and standard deviation of the degree of conversion (DC) will be calculated for each series where R = the percentage of uncured resin determined by bond height at 1640 cm⁻¹.

The percentage of degree of conversion (DC) will be calculated using the following equation:

$$DC(\%) = 100 \times \left[1 - \left(\frac{R_{\text{polymerized}}}{R_{\text{unpolymerized}}} \right) \right]$$

The rate of conversion (RC) is determined by subtracting the % C=C from 100% according to the formula:

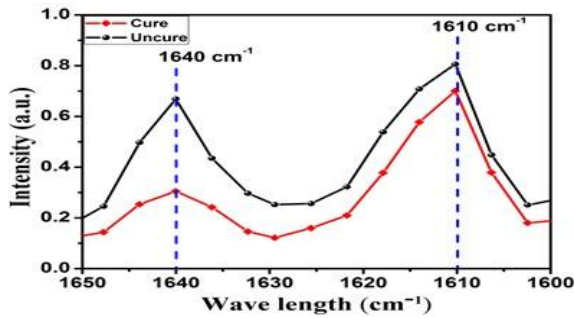
$$RC = \frac{1 - \left(\frac{C=C \text{ peak}}{C-C \text{ peak}} \right)_{\text{Cured}}}{\left(\frac{C=C \text{ peak}}{C-C \text{ peak}} \right)_{\text{Uncured}}} \times 100\%$$



The DC calculations from all the articles reviewed were obtained from the spectrum of the monomer absorbance of the aliphatic (C = C) band at approximately 1637 cm^{-1} and the absorbance of the aromatic (C - C) band at approximately 1608 cm^{-1} [20]. The band peak variations up to ~ 5 cm^{-1} , as obtained by different authors, is not significant because they can be associated to variations in composition and/or geometry of the structures. The ratio between C = C/C - C of polymers and C = C/C - C of monomers are taken as the fraction of unreacted double bonds in the polymer and it is important to directly compare the spectra to follow the conversion process [20].

RESULTS

Fig 1. FTIR spectra of uncured and cured dental resin cements



$$RC = \frac{1 - \left(\frac{C = C \text{ peak}}{C - C \text{ peak}} \right)_{\text{Cured}}}{\left(\frac{C = C \text{ peak}}{C - C \text{ peak}} \right)_{\text{Uncured}}} \times 100\%$$

$$= \frac{1 - (0.30575 / 0.70077)_{\text{Cured}}}{(0.66898 / 0.80601)_{\text{Uncured}}} \times 100\%$$

$$= 0.6791 \times 100\%$$

$$= 67.91\%$$

DISCUSSION

Dental restorative composite materials can be divided into direct and indirect resin composites (IRC). An indirect restoration refers to restorations fabricated outside the mouth and restored using indirect techniques. Most indirect restorations are made on a replica of a prepared tooth in a dental laboratory and are laboratory processed. However, chair-side CAD-CAM systems (computer-aided designing-computer-aided machining) are available for fabricating indirect ceramic restorations [11].

Though direct composite restorations have seen improvements in materials and techniques that overcome many of their limitations, they still exhibit certain drawbacks, some of which are as follows:

Polymerization shrinkage: Methacrylate based resins are responsible for polymerization shrinkage related problems such as gap formation and microleakage.

Degree of polymerization: One of the disadvantages of direct composite restoration is that however well cured, the resin does not undergo complete polymerization. The

degree of conversion (DC) ranges from 55% to 65% only. With the additional curing using heat, light, pressure, etc., employed with indirect composites, DC is increased to about 75%–81%. Hence, more amount of composite resin is polymerized.

Depth of cure: Effective cure of composites in deeper layers of a cavity is questionable since the light source cannot adequately cure composite resins at a depth of more than 4 mm.

Contacts and contours: Establishing a proper proximal contour and ensuring a tight contact with the adjacent tooth in direct restorations is a challenge.

Wear: Direct composite restorations exhibit excessive wear in areas of high occlusal stress.

Some of the aforementioned problems can be overcome by curing the composite resin extraorally with the help of secondary/additional curing using light, pressure, vacuum, heat, inert gas, or a combination of these methods resulting in a dense well-cured restoration. Such restorations can be finished and polished better and can then be cemented to the prepared cavity resulting in a dense well-cured restoration. The physical properties of such restorations are greatly improved.

ADVANTAGES OF IRC RESTORATION ARE:

Control of polymerization shrinkage: Since polymerization of the restoration occurs outside the oral cavity, the resulting restoration is slightly smaller than the preparation due to polymerization shrinkage. This space is occupied by the luting cement, and the only polymerization shrinkage that occurs is that of the thin layer of the resin cement at the time of cementation.

Secondary polymerization: In addition to light curing, the indirect composites undergo further polymerization by heat, intense light, and /or pressure. Hence, laboratory processing of composites produces a greater degree of polymerization that improves the strength and hardness of composites.

Resistance to occlusal wear: Since the composite is well polymerized and the physical properties are enhanced, it is more resistant to occlusal wear than direct composites with values less than 1.5 mm/year.

Improved control over contacts and contours: Proximal contour and contact areas can be well established with the indirect technique. The contact area can be better controlled and well finished to reduce loss of contact during function. Excellent occlusal morphology can be achieved with the indirect technique.

Improved physical properties: Due to higher filler loading, some physical properties such as wear, strength, and marginal integrity are better than direct composites. Hence, longevity is also better with indirect composites.

Esthetics: Since the restoration can be well polished in the laboratory, there is better retention and excellent esthetics over a longer period of time compared to direct composite restoration.



Reinforcement: Cross-splinting of the compromised tooth can be done with cuspal coverage. Unlike direct composite restorations, there is no pulling of buccal and lingual cusps toward the long axis of the tooth due to polymerization shrinkage.

Repair: Indirect composite restorations can be adjusted and re-polished easily and can also be repaired with light-cured direct composite resin material.

DISADVANTAGES OF IRC RESTORATION ARE:

Increased cost to the patient: There is additional laboratory cost involving impression and temporization leading to increased cost to the patient.

Tooth structure removal: Indirect restorations may require more tooth reduction as compared to direct composites to create a path of insertion and removal.

Modifications: It is difficult to modify or add extrinsic color at the chair side.

Lutting: The thin layer of luting resin cement is liable for shrinkage at the tooth–restoration interface.

INDICATIONS FOR INDIRECT COMPOSITE RESTORATIONS:

1. Inlays and onlays
2. Laminate veneers
3. Jacket crowns
4. Implant-supported restorations
5. Patients with bone loss and poor periodontal support requiring occlusal coverage
6. Full coverage crowns
7. Fiber-reinforced bridges/retainers

Touati *et al* and Mormann *et al* were the first to develop the technique for using early generation composite resin. The composition was similar to that of direct composite resin material.

Examples: SR Isosit Inlay system, Coltene Brilliant, Visio-gem (ESPE), Dentocolo (Kulzer), Concept (Ivoclar).

Disadvantages of first generation IRC materials are

1. Poor clinical performance
2. Deficient bonding between the organic matrix and inorganic fillers
3. Unsatisfactory wear resistance
4. High incidence of bulk fracture

The second-generation indirect composites were introduced in the mid-1990s with improvements in their compositions and different curing mechanisms. They contain microhybrid ceramic fillers with a diameter of 0.04–1 mm, with the filler content twice that of the organic matrix, leading to better physical properties and wear resistance. The inorganic filler content is 70%–80% by weight and 50%–60% by volume, the resin content being lower, around 33%.

In this present study SR Adoro (Ivoclar Vivadent), the second-generation indirect composite resins are used.

It contain- Matrix: UDMA and TEGDMA, Copolymer fillers: Microfilled composite ground into particles of 10–30 mm and incorporated with inorganic microfillers (49 wt% of barium glass particles), SR link: Monomer that contains a highly hydrophobic aliphatic hydrocarbon chain and a phosphoric ester with a methacrylate function.

Phosphoric acid group of the SR link reacts with the metal/metal oxide to form phosphates. These form a very inert passivating layer on the metal surface. The methacrylate group of the composite then reacts with the monomer in the SR link forming a copolymer and thus provides a bond with the veneering resin.

Properties- The flexural strength increases to 120–160 MPa and the modulus of elasticity ranges between 8500 MPa and 12,000 MPa.

The higher filler loading reduced the polymerization shrinkage, increased modulus of elasticity, and improved the mechanical properties and wear resistance of this second-generation indirect composite materials. Degree of conversion (DC) has a significant influence on the physical [12] and biological properties of resin composite restorations and is highly dependent on factors such as composition of the material, color and translucency, distance of the light tip to the surface and the irradiance of the polymerization lamp [13].

The cure of the resin cement is established by conversion of the carbon double bonds to carbon single bonds that can be measured through degree of conversion. The extent to which monomers react to form polymers during the polymerization reaction is expected to affect the physical properties of dental resins.

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 techniques [14].

Mechanical property measurements (hardness, Young modulus) appear to be more sensitive than vibrational techniques for following slow changes in the degree of conversion (DC), when the network is cross-linked. This is why FTIR Spectroscopy offers a direct approach for determining the degree of conversion (DC). Indeed, for methacrylate-based resins, this method allows for the evaluation of the degree of conversion (DC) (i.e., the percentage of vinyl functions converted to aliphatic functions) by comparing the vibration bands of the residual un-polymerized methacrylate. The vibration band of the residual un-polymerized methacrylate C=C bond at 1640 cm^{-1} with the aromatic C=C stretching band at 1610 cm^{-1} are used as internal standards [15].

Leung *et al.* (1984) concluded that the best technique for evaluating the degree of conversion (DC)



was FTIR (Fourier transformation infrared spectroscopy), even though the hardness measurement provides good information [16].

On the other hand, the study conducted by Rueggeberg and Craig (1988) revealed that the hardness measurement is more problematic than FTIR (Fourier transformation infrared spectroscopy) for detecting the small changes in the degree of conversion (DC), to follow the change occurring in the first stages of polymerization and after the network is cross-linked [17].

Microhardness measurements are not more sensitive than FTIR to changes in degree of conversion (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% [18]. 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 [19].

The DC results obtained in this study with the tested IRCs were slightly higher (67.91%) than those reported previously with the conventional resin composites for direct applications.

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.

CONCLUSION

Within the limitations of this study it can be concluded that the degree of conversion of indirect composites is higher (67.91%) than those reported with the conventional resin composites for direct applications and further studies are required to derive more conclusive results.

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