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## Effect of delay in layering on the incremental adhesion of indirect dental composite resins

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### ABSTRACT

Adhesion of the new layer of indirect composite resin (ICR) to the already polymerized one may be affected when the time between the applications of subsequent layers is prolonged. The aim of this study was to compare the shear bond strength and degree of conversion (DC) of two ICR systems with different compositions and adhesive promoters, relayered after four time points. Disk shaped ICR materials (ICR1 = Sinfony and ICR2 = Targis) ( $N=96$ ,  $n=48$  per material) were fabricated and processed according to each manufacturer's instructions. They were then randomly assigned to 4 groups. While immediate layering acted as the control group, after 5 min, 24 h and 1-week delay, a new layer of the ICR of the same kind with the substrate was adhered to the substrate in polyethylene molds. The bonded specimens were loaded under shear (1 mm/min) and bond strength was calculated. DC of ICR1 and ICR2 were similar ( $75 \pm 1$ ,  $75 \pm 2$ , respectively). Delay in relayering at different timepoints did not significantly affect the adhesion between the incremental layers of ICR1 (32–34 MPa) compared to the control group (34 MPa), but the adhesion between the layers of ICR2 showed decreased bond strength after 24 h (30.9) and especially more after 1 week (25 MPa) compared to immediate layering (38.9 MPa). The effects of ageing before bonding additional layers of ICRs is variable, and depends on the chemical formulation of the ICR as reflected in the different brands.

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### 1. Introduction

Dental composite resins are types of synthetic resins that are used in dentistry as restorative materials or adhesives [1]. Similar to other composite materials, a dental composite resin typically consists of a resin-based oligomer matrix usually based on bisphenol A-glycidyl methacrylate (bis-GMA) or urethane dimethacrylate (UDMA) consisting silanized inorganic fillers such as silicon dioxide (silica), barium, quartz or zirconia [2,3]. In today's dentistry, composite resins, applied directly by the clinician or fabricated indirectly in a dental laboratory and cemented in vivo, occupy a paramount position and present acceptable clinical performance with much lower costs than their ceramic counterparts [4,5]. Indirect composite resins (ICRs) were introduced in the dental market in an effort to address the disadvantages of the direct adhesive restorative materials such as technique sensitivity, difficult establishment of superior anatomical form, polymerization shrinkage or wear [6,7]. ICRs are

polymerized outside the mouth, in polymerization units that are capable of delivering higher intensities and levels of energy for polymerization compared to those of hand-held polymerization units. Indirect fabrication of composite resin restorations in a laboratory or chairside on a plaster model, subsequently adhesively cemented, provide improved quality of interproximal tooth contact, that is the contact between restorative material and the adjacent tooth, compared to direct application of such restorations [4]. ICR materials provide alternative ways for clinicians to overcome some inherent deficiencies of direct composite resin restorations, including polymerization shrinkage, their inadequate polymerization in deep interproximal areas between two neighboring teeth. Limited light transmission in such areas may yield to insufficient polymerization of the material [8–10].

Fixed dental prosthesis (FDP) is any dental prosthesis that is luted, screwed or mechanically attached or otherwise securely retained to natural teeth, tooth roots, and/or dental implant abutments that furnish the primary support for the dental prosthesis [11]. They can be metallic, ceramic or combination of both. ICRs are advantageous from optical point of view over metal-ceramic FDPs due the lack of metal in the reconstruction. Moreover, ICR FDPs show decreased wear of opposing dentition when compared to ceramic [6,7]. Also, finishing and polishing procedures are easy to perform and restorations can be repaired, if needed, using proper adhesive repair protocols [8,9,12,13].

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In order to overcome some of the deficiencies of the directly placed composite resins, attention has been directed to laboratory-processed composite resins. During the last two decades ICRs have gone through substantial changes in their process and compositions [7]. Ultra-small filler particles and polyfunctional methacrylate monomers are frequently used in ICRs [14,15]. Compared with direct composites, they are processed utilizing different laboratory techniques based on combinations of heat, pressure, vacuum and photo-polymerization. Indications of ICRs are inlays, onlays, veneers, single-unit and short-span anterior or posterior FDPs [7,9,10,12,13].

A variety of materials with remarkable differences in composition, polymerization modes and conditions comprise the second generation of the laboratory-processed ICRs [16–23]. Second generation ICRs became available in 1995 and their inherent characteristics and clinical performance have not been widely investigated [6,8,24,25]. Although the recently introduced systems claim improved clinical performance and optical properties, some studies noted delamination of the veneering ICR from the substructure that is attributed to layering techniques used during the fabrication process [5,26,27]. The reason for this could be associated with the inadequate adhesion during incremental build-up between the highly converted ICR layers [28]. In general, adhesion between two composite resin layers is achieved in the presence of an oxygen-inhibited layer of unpolymerized resin [29–31]. Yet, controversial opinions exist on the effect of oxygen-inhibited layer on the adhesion between two composite resin layers [32–35]. Considering the time needed for the completion of an ICR restoration that often ranges from some minutes to hours, changes in the oxygen-inhibited layer could affect the adhesion between the layers during build-up [36,37]. In some cases, especially after clinical trial, color adjustments may necessitate addition of new layers even after some days. Adhesion between two composite resin layers is achieved in the presence of an oxygen-inhibited layer of unpolymerized resin but radical half-life decreases by time [29–31]. Thus, it can be hypothesized that adhesion between ICR layers can be impaired when relayering is performed at delayed time points compared to immediate incremental layering. While manufacturers of some ICR systems recommend the use of silane coupling agent between the increments to increase the wettability of the subsequent increment,

others suggest the use of methacrylate based adhesive resin to achieve interpenetrating polymer network to activate the substrate surface for co-polymerization [28].

The objectives of this study therefore were to evaluate (a) the degree of conversion and (b) the incremental bond strength of ICR substrate and adherend in two systems with different compositions and adhesive promoters, relayered after four time points.

## 2. Experimental

### 2.1. Materials and methods

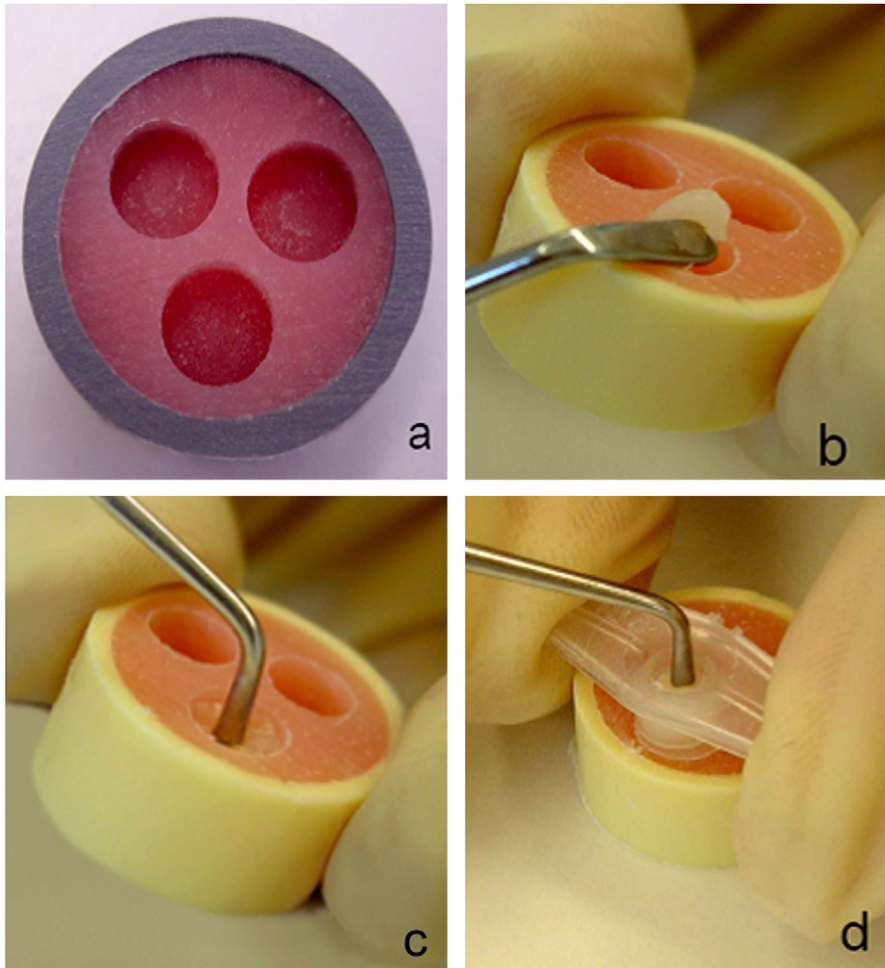
The brands, main chemical compositions, corresponding polymerization modes, batch numbers, manufacturers, the shades of the ICRs and the conditioning materials used for the experiments are listed in Table 1.

Cylindrical cavities (diameter: 5 mm, height: 3 mm) prepared in auto-polymerized polymethylmethacrylate (Autoplast, Candulor AG, Wangen, Switzerland) surrounded by a PVC cylinder (3 specimens per cylinder) were filled with either flowable ( $n=48$ , 12 per group) (ICR1-Sinfony) or packable ICRs ( $n=48$ , 12 per group) (ICR2-Targis). The unpolymerized composite resins were packed into the cavities with a hand instrument and photo-polymerized incrementally in layers of not more than 2 mm (Fig. 1a–d). Each increment was photo-polymerized initially with a halogen polymerization unit (Demetron LC, SDS Kerr, Orange, CA, USA) for 40 s from a constant distance of 2 mm from the surface. Light intensity ( $800 \text{ mW/cm}^2$ ) was verified by a radiometer (Demetron LC, SDS Kerr) after fabrication of every 12 specimen. The surface layer was flattened by translucent Mylar strip (KerrHawe SA, Bioggio, Switzerland) in order to create a smooth surface. ICR1 and ICR2 specimens were further subjected to oven polymerization. For ICR1 specimens, preliminary polymerization was achieved with Visio Alpha Unit (3M ESPE AG, Seefeld, Germany) ( $400 \text{ mW/cm}^2$ , Programme 3, no vacuum) for 5 min. A second cycle of polymerization was performed for 15 min in the Visio Beta Vario Unit (3 M ESPE AG) ( $40^\circ \text{C}$  with vacuum,  $470 \text{ mW/cm}^2$ ). For ICR2 specimens, preliminary polymerization was performed with Targis Quick Unit (Ivoclar Vivadent AG, Schaan, Liechtenstein) for 5 min ( $300 \text{ mW/cm}^2$ ). A second cycle of polymerization was made for

**Table 1**  
Brands, main chemical compositions, corresponding polymerization modes, batch numbers, manufacturers, the shades of the ICRs and the conditioning materials used for the experiments.

Brand	Chemical composition	Polymerization mode	Batch number	Manufacturer	Shade
Sinfony	HEMA and 10–30 wt% octahydro-4,7-methano-1H-indenediyl bis(methylene)diacrylate, strontium–aluminum borosilicate glass, silicon oxide, silane and photoinitiators weight (%) organic fillers: 50%	First cycle: Polymerization for 5 min in the Visio Alpha unit ( $400 \text{ mW/cm}^2$ , Programme 3, no vacuum) Second cycle: Polymerization for 15 min in the Visio Beta Vario unit ( $40^\circ \text{C}$ with vacuum, $470 \text{ mW/cm}^2$ )	203216	3 M ESPE AG, Seefeld, Germany	A2
Targis	bis-GMA 20%, DDDMA,UDMA, TEGDMA Silanized barium glass fillers, photoinitiators, inhibitors and pigments weight (%) organic fillers: 46.2%	First cycle: Polymerization for 5 min in the Targis Quick unit. ( $300 \text{ mW/cm}^2$ ) Second cycle: Polymerization for 25 min in the Targis Power unit ( $68^\circ \text{C}$ with vacuum, $400 \text{ mW/cm}^2$ )	15605	Ivoclar Vivadent AG, Schaan, Liechtenstein	A2
Targis wetting agent	3-methacryloxypropyltrimethoxysilane		815137	Ivoclar Vivadent	
Sinfony	activator	> 90 wt% (octahydro-4,7-methano-1H-indenediyl)bis(methylene)diacrylate		034	3 M ESPE AG

bis-GMA=Bis-phenol-A-glycidylmethacrylate.  
UDMA=Urethane dimethacrylate.  
TEGMA=Triethyleneglycol methacrylate.  
HEMA=2-hydroxyethyl methacrylate.  
DDDMA=1,10-Decanediol dimethacrylate.



**Fig. 1.** (a–d). Specimen preparation procedures: (a) cylindrical cavities (diameter: 5 mm, height: 3 mm) were prepared in auto-polymerized poly(methylmethacrylate) surrounded by a PVC cylinder (3 specimens per cylinder), (b and c) unpolymerized ICRs were packed into the cavities with hand instruments not exceeding 2 mm and photo-polymerized, (d) a new layer of ICR of the same substrate material was adhered onto the conditioned substrates using translucent polyethylene molds (inner diameter: 3.6 mm, height: 5 mm) and photo-polymerized.

25 min in the Targis Power Unit (Ivoclar Vivadent AG) (68 °C with vacuum, 400 mW/cm<sup>2</sup>) (Table 1).

The specimens of the control groups for both ICR1 and ICR2 were conditioned either with their corresponding adhesive (Sinfony Activator Resin, 3 M ESPE AG) or wetting agent (Targis Wetting Agent, Ivoclar Vivadent AG), respectively using a micro-brush and air-thinned. Sinfony Activator Resin was photo-polymerized for 10 s. Immediately, a new layer of ICR of the same substrate material was adhered onto the conditioned substrates using translucent polyethylene molds (inner diameter: 3.6 mm, height: 5 mm) and photo-polymerized for 40 s. This group acted as the control group.

5 min, 24 h and 1 week after conditioning each ICR resins with their corresponding surface activator, a new layer of ICR of the same substrate material was adhered onto the conditioned substrates as described above. One operator (M.Ö.) carried out the bonding procedures throughout the experiments. The ICR was packed against the substrate incrementally with a hand instrument and photo-polymerized in two layers of not more than 2 mm. Each layer was polymerized for 40 s. After polymerization, the polyethylene molds were gently removed from the test specimens. The specimens were then stored in a dark box in dry conditions for 24 h at 37 °C until the testing procedures.

Specimens were mounted in the jig of the Universal Testing Machine (Zwick ROELL Z2.5 MA 18-1-3/7, Ulm, Germany) and

shear force was applied to the adhesive interface until failure occurred. The load was applied to the adhesive interface, as close as possible to the surface of the substrate at a crosshead speed of 1 mm/min and the results were analyzed with the software program (TestXpert<sup>®</sup>, Zwick ROELL, Ulm, Germany).

## 2.2. Degree of conversion (DC)

Additional disk shaped specimens (diameter: 5 mm; thickness: 4 mm) ( $N=10$ ,  $n=5$  per ICR) were prepared for Sinfony and Targis. Each block was finished with wet silicone carbide papers up to 1200-grit and polished (Struers, 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. FT-Raman Spectrometer (RFS 100/S, Bruker Inc, Karlsruhe, Germany) provided the spectra of the unpolymerized and polymerized resins after 100 scans. The spectrum resolution was 4 cm<sup>-1</sup>. The specimens were excited by the defocused line of an Nd:YAG laser source at  $\lambda=1064.1$  nm with maximum laser power of approximately 90 mW at the specimen. The unpolymerized resin was positioned on an aluminum rod in a sample holder mounted on an optical rail for spectrum collection. Three spectra of the top surface and another three spectra of the bottom surface were collected. Based on the measurements, one average spectrum for each surface was

obtained. The average FT-Raman spectra were analyzed by selecting a range between 1590 and 1660  $\text{cm}^{-1}$ . 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, USA). 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 polymerized and unpolymerized conditions was performed in order to estimate the DC using the equation (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 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  $\text{cm}^{-1}$ /band height at 1610  $\text{cm}^{-1}$

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

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

DC of each ICR was then calculated using the following Eq. (2)

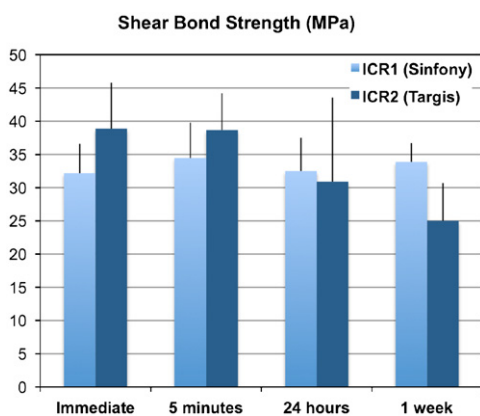
$$DC = 100 * [1 - (R_{\text{polymerized}} / R_{\text{unpolymerized}})] \quad (2)$$

### 2.3. Statistical analysis

Statistical analysis was performed using SPSS 11.0 software for Windows (SPSS Inc., Chicago, IL, USA). Mean bond strength data (MPa) were submitted to analysis of variance (2-way ANOVA and Tukey's test) with the shear bond strength as the dependent, ICR material type [2 levels] and timepoints for layering [4 levels] as the independent factors.  $P$  values less than 0.05 were considered to be statistically significant in all tests. DC data obtained from the top and bottom surfaces of the ICRs were analyzed by 2-way ANOVA.  $P$  values less than 0.05 were considered to be statistically significant in all tests.

## 3. Results

Both the ICR material type ( $P < 0.05$ ) and layering time points ( $P < 0.05$ ) significantly affected the bond strength results (2-way ANOVA). Interaction terms were also significant ( $P = 0.002$ ) (Tukey's test).



**Fig. 2.** Mean bond strength and standard deviations of ICR-ICR combinations, where the substrate and the adherend were of the same material (Sinfony-Sinfony, Targis-Targis), after immediate (control), 5 min, 24 h and 1-week delay in layering.

While ICR1-ICR1 adhesion was not significantly changed when layering was performed at the tested timepoints (Immediate, 5 min, 25 h, 1 week) ( $34.5 \pm 5.3$ – $32.2 \pm 4.4$  MPa) ( $P = 0.939$ ), ICR2-ICR2 adhesion showed significant decrease after 24 h ( $30.9 \pm 12.7$  MPa) ( $P = 0.0028$ ) and 1 week ( $25 \pm 5.7$  MPa) ( $P = 0.0025$ ) delay of layering compared to immediate layering (control:  $38.9 \pm 6.9$  MPa). For ICR2, 5 min ( $38.7 \pm 5.5$  MPa) delay in layering did not show significant difference compared to the control group (Fig. 2).

DC of ICRs did not show significant differences between the experimental groups (top surface:  $75 \pm 2$ ,  $75 \pm 11$ , bottom surface:  $73 \pm 8$ ,  $74 \pm 7$ , for Targis, Sinfony respectively) ( $P > 0.05$ ).

## 4. Discussion

The reported clinical failures of ICRs were related to chipping or pitting occasions ranging between 9–29% [4–6]. Since ICRs are fabricated based on layering principles, adhesion between the incremental layers is important in order to overcome such delamination or chipping problems clinically [38]. When working with ICR materials, the general principle is to minimize shrinkage during polymerizing a bulk of material. Therefore, incremental layering techniques should be implemented. Although controversial opinions exist [39,40], it is generally accepted that with the incremental layering, shrinkage stress may be reduced since bulk volume and configuration factor decrease [41]. Incremental layering gives the clinician or the dental technician the chance to build-up the restoration on solid base as well as controlling the color effect during layering [7]. However, this procedure may cost time and the working process may be delayed. Therefore, this study compared the effect of delayed layering with immediate layering simulating the time that is needed to complete a multiple-unit ICR FDP on the incremental adhesion of two types of ICRs. While ICR1 was not affected when layering was performed at the tested timepoints, the bond strength results decreased for the ICR2 with the increase in relayering time. Thus, since ICR1-ICR1 adhesion was not affected from delayed layering, the hypothesis that adhesion between ICR layers can be impaired when layering is performed at delayed timepoints compared to immediate incremental layering can only be partially accepted.

Although the polymerization methods for the two materials studied are not identical, the working principles of these methods are similar. The significant differences between the bond strengths of ICR1 and ICR2 may be referred to several other factors. One reason for this could be attributed to the slight variations between the polymerization devices. Currently available ICRs are often polymerized utilizing polymerization devices in which higher degree of conversion could be expected as a result of heat and light combination [42]. In this study, ICR1 was polymerized in such a device in total 20 min with the second polymerization cycle being at 40 °C. On the other hand, ICR2 was polymerized in total for 30 min at 68 °C. The additional heat and prolonged time of polymerization might have contributed to the better polymerization thereby, less adhesion with the subsequent adherend layer in the ICR2 material. However, the DC of both materials was statistically similar. Therefore, different polymerization regimes used to polymerize materials with varying constituents in this study make it difficult to reliably explain the differences in results. Further studies are needed to explain the time dependent bond strength decrease between aged and fresh ICRs with other surface characterization and modeling methods after aging them at different timepoints.

Polymerization characteristics of ICRs also vary depending on the power output of light source at polymerizing wavelength, type and geometry of light transmission system, and the type of

composite resin. Laboratory processed composite resins use photo-polymerizing units that combine intense visible light (320–400 nm) and heat that increase the conversion of monomer to polymer. Reduced degree of conversion may decrease the mechanical properties of composite resins [29,43]. The more intense the light source, the more photons available for absorption by photosensitizers [15–26]. Heat polymerizing in addition to visible-light polymerizing has been shown to substantially increase the degree of conversion [37] and reduce the oxygen inhibition of polymerization [31,37,44]. Adequate polymerization is a crucial factor in obtaining optimal physical properties and clinical performance of ICR restorative materials [6]. It is therefore desirable for a dental composite resin to convert most of its monomer to polymer during the polymerization reaction to achieve the best physical and biological properties [29–43]. However, this may then result in a more brittle material.

In this study, satisfactory bond strengths were obtained from both ICR systems tested at all time points, however, with some variations between ICR types. Bond strength is dependent on unconverted C=C double bonds on the resin, which may be attributed to either to a low conversion rate or a high matrix portion [45]. The bond between the substrate and new resin is also based on unreacted C=C double bonds of the functional groups on the surface of the polymer matrix [46]. A high degree of conversion that resulted from the use of heat and light used for polymerization causes improvement in mechanical strength and hardness but on the other hand makes the attachment of the new composite resin more difficult [17–21]. As stated earlier, adhesion between two composite resin layers is achieved in the presence of an oxygen-inhibited layer of unpolymerized resin [29–31]. Although, there are controversial studies [32–35], changes in the oxygen-inhibited layer may have significantly decreased the adhesion between the layers of ICR2 after one week. The thickness and quality of the oxygen-inhibited layer requires further research. Since similar conversion values were obtained for both ICRs, silane coupling agent and the wettability of the adhering resin seems to have more importance in the obtained results. Furthermore, the radical half-life, surface roughness, viscosity of the materials should also be taken into consideration. Further studies are needed to test the wettability, surface roughness, and viscosity as well as oxygen-inhibited surface layer of ICR materials.

The delay in working time of 5 min, 24 h or 1 week did not affect the adhesion of the new layer to the processed substrate significantly in the ICR1 group. This indicated that ICR1 must have a surface more suitable for chemical bonding. One reason for the non-significant difference between the delayed versus immediately layered control group, may be related to the function of adhesive resin that may bond covalently to the pendent, unreacted methacrylate groups. Intermediate resin has been reported earlier to cause swelling of the composite substrate surface with different solvents and the use of low-viscosity adhesive resin influence the bond between two composite resins [46]. The functions of adhesive resin, prior to new resin application during layering, are to achieve better wetting of the substrate surface and dissolve and swell the polymer surface of the substrate to some degree. According to the manufacturer's information, the Sinfony activator monomer system contains more than 90% by weight of monomer (octahydro-4,7-methano-1H-indenediyl)bis(methylene)diacrylate, which is also the same monomer used in the chemical composition of ICR1. This molecule contains two diacrylate functional groups and an indenediyl group. The existence of indenediyl group increases rigidity of the molecule that might have influenced both the dissolving capability of Sinfony activator monomer system on the substrate surface. Advantages of this monomer are its low polymerization shrinkage and its hydrophobic middle part that is indenediyl. It can be also assumed that the viscosity properties were close to that of the unfilled activator monomer that resulted in high bond strength.

The hydrophilic adhesive joint in ICR2 seemed to be weaker than that of ICR1. The wetting agent used in ICR2 system was a 3-methacryloxypropyltrimethoxy silane coupling agent. Silane treatment improves the wettability of the surface, affects the surface energy, hence its dispersion in the matrix. However, when little unconverted C=C double bonds are available, these functions of silane could obviously not be profited in ICR2 group.

It was supposed that a certain percentage of unconverted C=C double bonds are available even after laboratory processing [44]. The differences in the results between the two systems could be then also due to differences in polymerization and monomer formulation. In high molecular weight monomers such as bis-GMA or UDMA there is always an incomplete and significant concentration of unreacted C=C remaining within the resin when it is polymerized with visible light and under heat [47,48]. In addition to the unreacted monomer, further unreacted C=C structures may be present from the diluents such as TEGDMA or similar substances [47]. The insignificant but higher bond strength results, except for 1 week, obtained from ICR2 group could be based on UDMA matrix monomers existing in this ICR type. In FTIR (Fourier transformation infrared spectroscopic) evaluations, it was found that the UDMA/TEGDMA phase had a conversion rate of 70% and exhibited superior wear resistance, while the bis-GMA/TEGDMA had a conversion rate of 55% [49]. In ICRs with such matrix content, the possibility to obtain free radical polymerization bonding during layering is low because of relatively small number of unreacted C=C double bonds on the polymer surface [29,49].

It has been also previously shown that there is slow continuous increase in conversion of conventional photo-polymerized composite resins after initial photo activation [15,43]. Loza-Herrero et al. [15] reported that the delay more than 6 h changed the conversion rate from 58% to 68%. In the current study, the decrease in Targis group after 1-week delay in layering could also be due to continuation of conversion over 24 h or one week.

It would be favorable to maintain a chemically active composite resin surface to secure the chemical bond, but the light polymerization devices may overrule this advantage. The ICR products may profit from the use of an intermediate adhesive resin between layers after each polymerization cycle than the use of a silane coupling agent alone.

## 5. Conclusions

From this in-vitro study, the following conclusions were drawn:

1. Degree of conversion of the two ICRs tested was similar.
2. The effects of aging before bonding additional layers of ICRs varied depending on the chemical formulation and polymerization method of the ICR as reflected in the different brands.
3. Delay in relayering did not affect the incremental adhesion between the layers of ICR1 (Sinfony) but ICR2 (Targis) showed decreased bond strength after 24 h and more dramatically after 1 week.
4. Restorations made of ICR1 (Sinfony) can be relayered at delayed time points up to 1 week but ICR2 (Targis) should be processed preferably in one day in order not to experience decreased adhesion between the incremental layers which may clinically induce chipping problem.

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