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Influence of the mechanical properties of composites for indirect dental restorations on pattern failure

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SUMMARY

This study evaluated the fracture pattern of four composites for indirect dental restoration relating to three-point flexural strength, compressive strength and modulus of elasticity (Solidex, Artglass, belleGlass, and Targis). Ten specimens of each composite were tested in a universal testing machine at 0.5 mm/min crosshead speed for flexural strength and 1mm/min for compressive strength. Fracture pattern was classified as complete or partial fracture. Modulus of elasticity was calculated from flexural strength data. Composites polymerized under high temperatures (belleGlass and Targis) had higher flexural strength and elastic modulus values than composites polymerized by light (Artglass and Solidex). However, they failed earlier under compression because they were more rigid and showed partial fracture in the material bulk.

Key words: flexural strength, compressive strength, dental composites, indirect restoration.

INTRODUCTION

Photo-polymerized dental composites for indirect restorations have been used as an alternative esthetic material for ceramic single restorations, multi-unit fixed partial dentures, and implant-supported prostheses [1,2]. The first generation of composites for indirect restoration was introduced to the dental market in the 1980-decade but they showed poor *in vitro* and clinical performance. Deficient bonding between organic matrix and inorganic fillers was the main problem leading to unsatisfactory wear resistance, high incidence of bulk fracture, marginal gap, microleakage, and adhesive failure in the first attempts to restore posterior teeth [3]. Efforts to solve these problems included the increase of inorganic filler content, reduction of filler size, and modification of the polymerization system [3,4].

The use of different polymerization methods may result in variation of mechanical properties, *e.g.*, the application of heat for additional polymerization increases the conversion rate of monomers, reflecting in improvement of surface hardness, compressive and flexural strength [5]. However, few independent and standardized studies on physical properties and clinical performance of composites for indirect restorations are available, and the literature is controversial. For example, in relation to flexural strength of belleGlass system (Kerrlab), one study [4] reported 142 MPa while other [2,6] reported 221,7 MPa. Also, manufacturers' information about the products cannot be directly compared because they often use different methodologies to evaluate mechanical properties.

The purpose of this study was to compare three-point flexural strength, compressive strength, elastic modulus, and fracture pattern of four indirect composites polymerized by different methods: 1) light – Solidex (Shofu) and Artglass (Heraeus-Kulzer); 2) light and heat – Targis (Ivoclar); and 3) light, heat, and pressure – belleGlass (Kerrlab). The null hypoth-

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esis was that there is no difference in mechanical properties among the tested composites.

MATERIAL AND METHODS

Table 1 displays the brand names, manufacturers, and filler content of the composites tested.

Flexural strength test

Ten specimens of each composite system were made using a 25 x 2 x 2 mm metallic matrix, according to the ISO Specification No. 4049 (1988) [8] for flexural strength test . The composite was packed into the metallic matrix in one increment. A transparent plastic stripe was positioned over the metallic matrix, and a glass slab was pressed against the matrix-composite. The glass slab was removed for initial composite polymerization for 20 s (curing unit XL–1500, 3M-ESPE, Seefeld, Germany) with light intensity above 400mW/cm^2, which was monitored by a radiometer (Curing Radiometer, model 100, Demetron/Kerr, Danbury, CT, USA). After this step, the specimen was removed from the metallic matrix and received additional polymerization according to the composite system.

Solidex specimens were submitted to additional polymerization in the Solidilite system (Shofu, California, USA) at a wavelength of 420-480 nm and temperature of 55 ºC for 3 minutes. Artglass specimens were placed inside the stroboscopic light unit UniXs (Heraeus-Kulzer, Hanau, Germany) for 180 s. BelleGlass specimens were treated in the curing unit (KerrLab Corporation, West Collins Orange, CA, USA) under 60 psi nitrogen pressure at 140 °C for 20 minutes. Targis specimens were coated with glycerin gel (Targis Gel) to prevent formation of oxygen-inhibited surface layer and were placed in the curing unit Targis Power (Ivoclar Vivadent, Liechtenstein - Switzerland) for the following cycle: light emission in the first 10 min, increase of temperature to 95 °C for 10 s, and cooling for 5 min. After this, the specimens were rinsed in running water and dried with air blasts. All specimens were stored in individual light-protected plastic tubes with distilled water (1 design group per vial) at 37 °C for 1 week.

Specimens were placed on a 25 mm-length supporting base and assembled in a universal testing machine (EMIC DL-2000, EMIC, São José dos Pinhais, PR, Brazil). A customized device was adapted to the upper holder to allow vertical loading of the specimens according to a three-point bending test design. Axial load was applied until failure at a crosshead speed of 1.0 mm/min. Flexural strength data were obtained in kgf and transformed in MPa using the following ISO 4049 formula: $\sigma = 3\,F\,L\,/\,2\,b\,h^2$, where σ is the flexural strength (MPa), F is the recorded force (kgf), L is the length between the supporting points (21 mm), b is the width of the prism (2 mm), and h is the thickness of the prism (2 mm). The load-deflection curves were recorded with computer software (MTest, EMIC).

Table 1. Specifications of the composite systems for indirect restoration evaluated in this study

Material and Manufacturer	Filler size *	Filler content (% vol) *	Color	Batch number
Solidex, Shofu, California, USA	1µm silicon dioxide, aluminum oxide	53%	C3, dentin	030220
Artglass, Heraeus-Kulzer, Hanau, Germany	1µm barium glass, colloidal silica	68%	C3, dentin	040104
belleGlass, KerrLab Corporation, West Collins Orange, CA, USA)	25µ particles (blend of resin, barium glass and discrete nanofiller), 0.4µ structural filler and small discrete silica nanoparticles	87%	C3, dentin	107373
Targis, Ivoclar, Liechtenstein - Switzerland	30nm-1µm barium glass, silicon dioxide	55%	210 O2/S2, dentin	D38031

* Information from Miranda et al.7 and manufacturers.

Table 2. Flexural strength, compressive strength, and modulus of elasticity of the tested indirect composites

Group (n=10)	Flexural strength (MPa)		Compressive strength (MPa)		Modulus of elasticity (GPa)		Pattern of fractures (%)	
	Mean	SD	Mean	SD	Mean	SD	Bulk	Partial
Solidex	76.95^{c}	14.86	206.70^{a}	34.91	15.61 ^{b}	2.91	100%	
Artglass	94.76^{bc}	13.51	224.00 ^{a}	17.40	14.03 ^{b}	3.32	100%	
BelleGlass	132.48^{a}	22.19	163.02^{b}	18.42	21.55 ^{a}	2.23		100%
Targis	111.23^{b}	17.02	163.39^{b}	32.04	19.48 ^{a}	3.45		100%

Means followed by the same letter were not statistically different (ANOVA and Tukey test, α=0.05).

Compressive strength test

Compressive strength test was performed according to previous studies [9,10]. Samples were made with 2 mm thick increments of each composite resin using a cylindrical Teflon matrix with 3 mm diameter and 6 mm height. Polymerization method for each system followed the procedures previously described for the flexural strength test. After storage for 24 h, specimens were tested in a universal testing machine at a crosshead speed of 1mm/min. Data were obtained in kgf and transformed in MPa using the following formula: $R_C = F \times 9.807 / A$, where R_C is the compressive strength (MPa), F is the recorded force (kgf) multiplied by the constant *9.807* (gravity), and A is the base area (7.06 mm^2).

After compressive strength testing, the specimens were classified according to the type of fracture: complete fracture if the specimen had rupture into multiple fragments, or partial fracture when indicated by the specimen deformation percentage at 50% in the software Mtest of the universal testing machine.

Modulus of elasticity

From flexural strength data, modulus of elasticity was calculated using the following formula E (GPa) [20]: $Ef = l^3 Fl / 4b f h^3$, where *Ef* – flexural modulus; *l* – support width (mm); *Fl* – load (N) at convenient point that is in straight line portion of the trace; *f* – deflection of the test specimen at load *Fl* (mm); *b* – breadth of the test specimen (mm); and *h* – height (mm). Flexural strength, compressive strength, and modulus of elasticity data were analyzed by ANOVA and Tukey test at the level of significance of 0.05 (two-tailed).

RESULTS

Mean values (MPa) of flexural strength, compressive strength, and modulus of elasticity are shown in Table 2. BelleGlass and Targis had higher flexural strength and modulus of elasticity than Artglass and Solidex, but lower compressive strength.

DISCUSSION

As flexural strength reflects resistance to compressive and tension stresses that act in the material simultaneously [11], the evaluation of this property is important for materials used in posterior teeth, particularly in multi-unit fixed partial dentures. In our study, the composite polymerized by light, heat, and pressure (belleGlass system) had the highest flexural strength, followed by the composite polymerized by light and heat Targis (Ivoclar Vivadent). The composite system with additional polymerization under stroboscopic light (Artglass) had intermediate values of flexural strength and was not different from Targis and Solidex. High flexural strength for belleGlass may be related to its polymerization under nitrogen environment and pressure, which decreases porosity and oxygen inhibition, and increases adhesion of fillers to resin matrix [5,12]. This combination of high temperature and pressure for additional polymerization increases flexural strength, and may improve wear resistance [13], hardness, and diametral tensile strength [14] because of high monomer conversion rate [5,12]. It has been reported that systems that only use light-polymerization have lower flexural strength even with increased light intensity and longer polymerization [15]. However, Artglass (only light polymerization) exhibited flexural strength similar to the composite additionally polymerized by heat (Targis) probably because of the presence of monomer with multifunctional groups [16].

BelleGlass and Targis showed higher modulus of elasticity than Artglass and Solidex, with values ranging from 15.61 to 21.55 GPa. It can be speculated that additional polymerization and increase of monomer conversion rate result in higher modulus of elasticity, which also may be influenced by filler size and volume [5,17]. Both filler morphology and filler loading are shown to influence flexural strength, flexural modulus, hardness, and fracture toughness of dental composites [18]. Parallel conclusion was drawn by another study [7] with the same composites tested here, which reported that Targis showed higher microhardness than Artglass and Solidex.

Contrary to our expectations that the resin additionally treated with heat would have higher compressive strength, Artglass and Solidex showed higher values than Targis and belleGlass. The opaque composites Targis and belleGlass have more Bis-GMA in the organic matrix and higher elastic modulus. On the other hand, Artglass and Solidex have high content of multifunctional monomers in the organic matrix and are more resilient. Artglass manufacturer claims that the material is more resistant to fractures because it is more resilient than the resins with large amount of Bis-GMA. The compressive strength test is easy to perform but its interpretation is complex as tension and shear forces act concurrently inside the material. Brosh et al. [19] stated that compressive resistance cannot predict the capacity of the composite resin to support stress, and that this relationship is limited to frail materials. Composite resins would suffer a "barrel" effect when submitted to a compressive test and expand until plastic deformation occurs [11].

Fig. 1. Partial fracture pattern observed for belleGlass and Targis specimens

In relation to the fracture pattern, 100% of the belleGlass and Targis specimens had partial fracture mode. Solidex and Artglass specimens showed 100% of complete fracture mode (Figures 1 and 2).

Observation of fracture pattern under compressive force is important for the understanding of material behavior. Failure pattern may be dependent on the volume and distribution of inorganic particles or the organic matrix components, but the interpretation of the failure mechanism is very complex as several forces are interacting and competing simultaneously. BelleGlass and Targis had 100% partial fractures and

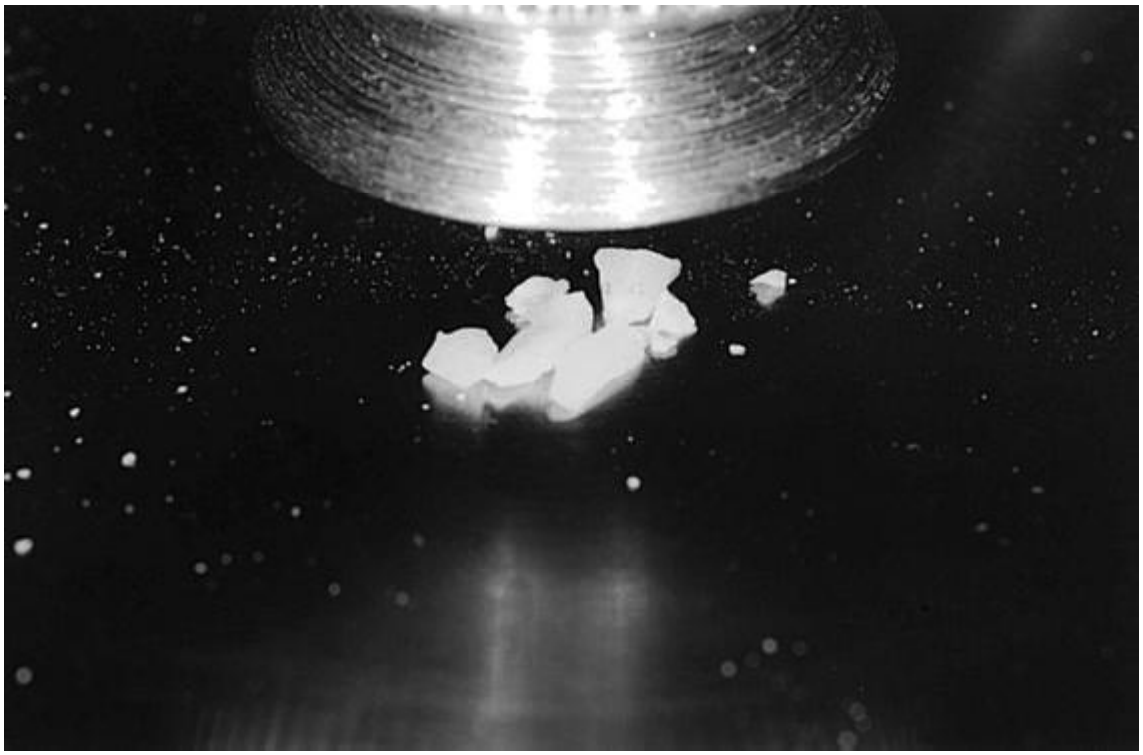

Fig. 2. Complete fracture of Solidex and Artglasss pecimens

exhibited a pattern of homogeneous fracture propagation with longitudinal rupture of the specimen into two or three large fragments. Ferracane and Condon [5] speculated that composites submitted to heat might present internal stress relief, specifically at the interface between organic matrix and inorganic particles. This would increase the adhesion between both phases and the cross-linking between the methacrylate groups. The occurrence of large fragments denotes this great adhesion between phases. However, during the compressive test, when the first longitudinal fracture occurred, the testing machine automatically stopped its movement preventing the total rupture of belleGlass and Targis specimens. This did not happen for Solidex and Artglass, which presented 100% of complete fracture and were reduced to minute fragments without the longitudinal fracture pattern observed for belleGlass and Targis.

Within the limitations of an in vitro study, clinicians must be aware that indirect composites are essentially direct composites in their composition. However, the additional curing process seems to be the most relevant point in each system. The results of the indirect composites belleGlass and Targis confirm this hypothesis, as their flexural behavior and elastic modulus were superior compared to the other tested composites. Nevertheless, only clinical investigations are able to confirm if post-polymerized composites have higher success rate than simple photocured resins used for indirect restorations or if the adhesive cementation would sustain those differences.

CONCLUSIONS

These results suggest that composites polymerized under high temperatures (belleGlass and Targis) have higher flexural strength and modulus of elasticity than composites polymerized by light (Artglass and Solidex). However, they fail earlier under compression because they are more rigid and show partial fracture in the material bulk.

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