Published online: 2019-09-30

Mechanical properties of direct and indirect composites after storage for 24 hours and 10 months

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ABSTRACT

Objective: The objective of this study was to evaluate the diametral tensile strength (DTS) and Knoop hardness (KH) of direct (Filtek Z350-3M/ESPE and Charisma-Heraeus Kulzer) and indirect composites (Sinfony-3M/ESPE and Signum-Heraeus Kulzer) kept in storage for two periods of time, 24 hours and 10 months, in distilled water.

Methods: Twenty-five specimens of each material were prepared. DTS (n=10) was tested using a universal testing machine (Versat, model 2000) at a crosshead speed of 1.0 mm/min. KH (n=5) was measured using Knoop micro-hardness (HMV-2000; 50 gf for 15 s). All tests were performed 24 hours after polymerization and after 10 months of storage in distilled water at 37°C. The data were statistically analyzed using Kolmogorov-Smirnov, ANOVA and t-Student (P=.05).

Results: Filtek Z350, Sinfony, and Signum showed higher DTS values than Charisma after 24 hours. After storage, Sinfony and Signum showed higher DTS values because the storage did not influence the DTS values of the indirect composites. Filtek Z350 showed higher KH values after 24 hours and after storage than other composites; the storage influenced the KH of all composites except Sinfony.

Conclusion: Storage for 10 months did not influence the properties of the indirect composite Sinfony. In general, the indirect composites showed higher DTS values than direct composites, especially after 10 months storage. The direct composite Filtek Z350 obtained the highest KH values regardless of storage. (Eur J Dent 2013;7:117-122)

Key words: Composite resin; dental materials; hardness

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INTRODUCTION

Composites may be prepared in posterior teeth by direct or indirect techniques. Several investigations have shown that many properties associated with direct composite resins are inadequate when used as posterior restorative materials in extensive cavities.[1,2] Clinical disadvantages of direct composites include polymerization shrinkage and inadequate contour. Because of major clinical problems that clinicians have experienced with the use of direct posterior composite resins, the indirect composite system was introduced.[3] Indirect composites have revealed several clinical advantages compared with the direct technique, including improved contour and interproximal contact and the potential for less postoperative sensibility because restoration is generated on a die rather than directly in the cavity preparation.[4] In addition, as the restoration may be fabricated outside the mouth, increased temperature, pressure, light intensity, or a combination of these factors may be used to improve curing.[5,6]

However, the composition of indirect composite resin systems is similar to that of direct systems, differing in terms of the use of different methods of additional polymerization, which allows a higher radical conversion. These additional polymerization procedures can involve photo-activation, heat, pressure, and a nitrogen atmosphere, as previously described.[6,7] Therefore, it is expected that indirect composites show better properties than direct composites because of the possibility of better activation of polymerization reactions.[8]

The possibility of a higher radical conversion can improve the immediate properties of these materials, along with the longevity of the restoration. This is because most conversions can reduce the degradation and leaching of the monomeric components.[9]

Aging in water, on the other hand, may have a beneficial effect on dental composites, as the water is absorbed into the resin matrix, making the composite more flexible, resulting in an apparent increase in its mechanical properties. However, over time, the leaching of the components and the swelling and degradation of the cross-linked matrix in the dental composite and hydrolysis of the filler-matrix interfaces eventually lead to a decrease in the mechanical properties.[9,10] Thus, it is important to evaluate the properties of the composites after a certain storage time.

The objective of this study was to evaluate the diametral tensile strength (DTS) and Knoop hardness (KH) of direct and indirect composites after storage for two periods of time, 24 hours and 10 months, in distilled water at 37°C.

The hypothesis tested were:

1. The indirect composites could show higher mechanical properties (DTS and KH) than direct composites both in the immediate test and after 10 months of storage;

2. The storage will not influence the mechanical properties (DTS and KH) of indirect composites, but will influence the mechanical properties of direct composites.

MATERIALS AND METHODS

The materials used in the study are shown in Table 1.

Four composites were analyzed. Two direct composites with higher (Filtek Z350) and lower (Charisma) content of filler particles and two indirect composites with higher (Signum) and lower (Sinfony) content of filler particles.

Diametral Tensile Strength - DTS

Cylindrical brass molds (4 mm inner diameter and 2 mm thick) were used for the preparation of specimens. The molds were kept on transparent strips on glass plates. The composite resin was packed into each mold and a second transparent strip was kept on top and covered with a second glass plate. The molds and strips of film between

Table 1. Materials name, type, composition and manufacturers of composites.

Material	Type of composite	Composition	Manufacturer
Filtek Z350®	Direct	Bis-GMA, Bis-EMA, UDMA and TEGDMA - Nanofillers of silica (5-20 nm) and nanoclusters of zirconia/silica (0.6 and 1.4 μm) - 78.5 wt%	3M ESPE St. Paul, MN, USA
Charisma®	Direct	BIS-GMA and TEGDMA - Fillers: Ba-Al-F – 64 wt%	Heraeus Kulzer, South Bend, Ind
Sinfony®	Indirect	Aliphatic and cycloaliphatic monomers - Fillers: Al-B-Si and B-SiO2 (0,6 μm) - 50 wt%	3M ESPE St. Paul, MN, USA
Signum®	Indirect	Bis-GMA and TEGDMA - SiO2, Ba-Al-Si (1,0 μm) - 70 wt%	Heraeus Kulzer, South Bend, Ind

the glass plates were pressed to displace excess material. The plates were removed and the composite resin was exposed to visible light for a predetermined time using the manufacturer's recommendations in accordance with Table 2. Only the photo-activation of direct composites, a light emitting diode - LED Freelight II (3M-ESPE, St. Paul, MN, USA) was used. The activation of indirect composites was made according to Table 2. After polymerization, in accordance with Table 2, both top and bottom surfaces were wet-polished with 1200-grif SiC paper to obtain a planar and parallel surfaces. The specimens were kept in distilled water at 37°C, some for 24 h and others for 10 months before testing.

DTS was determined using a Universal Testing Machine (Versat 2000, Panambra, São Paulo, Brazil) with a crosshead speed of 1.0 mm/min (Figure 1). The load at which break occurred (Kgf) was noted and DTS was calculated using the following formula:

DTS (MPa) = 2P / DL where P is the maximum value in Kgf, D is the diameter of the sample in mm, and L is the thickness of the sample in mm. Mean and standard deviations of ten specimens (n=10) of each group were calculated.

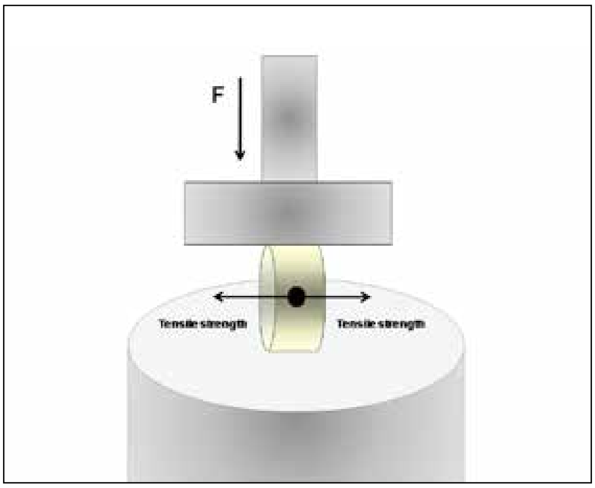

Figure 1. Diametral Tensile Strength (DTS).

Knoop hardness – KH

For the KH test, five specimens of each composite were prepared exactly as described for the DTS test. After the light-curing procedure, the specimens were stored in distilled water at 37°C for 24 h. The surfaces of samples were wet-polished with 1200-grit SiC paper to obtain a planar surface. Knoop hardness measurements were taken using an indenter (HMV-2, Shimadzu, Tokyo, Japan) under a load of 490N (equivalent to 50 gf) for 15 s. Five readings were performed. The KH number (KHN, Kgf/mm^2) was recorded as the average of the five indentations.

After, these specimens were stored again in distilled water at 37°C for 10 months and new KH readings were performed as describe above.

Statistical Analysis

Data involving DTS and KH was statistically evaluated using Kolmogorov-Smirnov for evaluations of normality and then a two-way analysis of variance-ANOVA (composite and storage time) was performed. Comparisons were conducted using t student test (P=.05).

RESULTS

Table 3 shows the mean DTS values of composites after 24 hours and after 10 months of storage.

The direct composite Filtek Z350 and the indirect composites Sinfony and Signum showed higher DTS values after 24 hours than Charisma. After 10 months, Sinfony showed the highest DTS values, followed by Signum. In this period (10 months), the direct composites Filtek Z350 and Charisma showed the lowest DTS values.

The direct composites Filtek Z350 and Charisma were affected by storage, as shown by virtue of higher DTS values after 24 hours than after 10 months, unlike the indirect composites Sinfony and Signum, which showed no difference between

Table 2. Photo-activation protocol used for each material.

Material	Polymerization method
Filtek Z350®	20 seconds at 1200 mW/cm^2 (radiant exposure – 24 J/cm^2)
Charisma®	20 seconds at 1200 mW/cm^2 (radiant exposure – 24 J/cm^2)
Sinfony®	First cyclo: photo-activation (Visio Alpha), 400 mW/cm^2, 15 s; Second cyclo: photo-polymerization (Visio Beta) under vacuum at 40ºC, 15 min.
Signum®	180 seconds at 1100 W using xenon strobe light

the periods of storage (24 hours and 10 months).

Table 4 shows the mean KH values of composites after 24 hours and after 10 months of storage.

The direct composite Filtek Z350 showed the highest KHN after 24 hours, followed by Charisma and Signum. Sinfony showed the lowest KHN after 24 hours. After 10 months of storage, Filtek Z350 continued showing the highest KHN, followed by the other composites, which showed no statistical differences between them.

The direct composites Filtek Z350 and Charisma and only the indirect composite Signum were affected by storage, as shown by higher KHN in 24 hours than 10 months, unlike the indirect composite Sinfony, which showed no difference between the periods of storage (24 hours and 10 months).

DISCUSSION

The advantages of the use of indirect materials for the restoration of compromised teeth are well known.[11] Most often, the material of choice for indirect restoration is ceramic due to its excellent mechanical properties and aesthetics.[5,11,12] However, when ceramics cannot be used, for example, in patients with temporomandibular disorders, indirect composites are used.[2]

Composite resins have better mechanical properties, such as compressive strength, than other restorative composites such as conventional or resin-modified glass ionomers, suggesting a longer clinical life in regions submitted to occlusal loads.[13]

The literature reports a positive correlation between compressive strength and diametral tensile strength. In both types of testing, specimens are submitted to a compressive load applied at different planes, and fracture occurs as a result of tensile and complex shear stresses within the material.[13] Diametral tensile strength testing was developed to investigate brittle materials with little or no plastic deformation. In this test, a cylindrical specimen is submitted to a compressive load in the diametral plane, which is perpendicular to the longitudinal axis (Figure 1).[13,14]

There is a close relationship between fatigue resistance, hardness, elastic modulus, compressive strength and diametral tensile strength of materials. A restorative material with high compressive strength, flexural strength and diametral tensile strength may be clinically applied and should be resistant to masticatory forces.[13,14]

Indirect composites represent an alternative that can be used to overcome some deficiencies in direct composite restorations, such as polymerization shrinkage stresses, inadequate polymerization in interproximal areas, restoration of proximal contacts, and adequate dental contour.[3] Moreover, as the composition of indirect composites is similar to that of direct composites, but with more efficient methods of polymerization, some manufacturers believe that indirect composites have improved properties. Mechanical properties such as KH and DTS, may also be affected by the polymerizing system. The indirect composites were polymerized using proprietary curing units. These units combine heat, vacuum, and high in-

Table 3. Means (MPa) and standard deviation of diametral tensile strength (DTS) of direct and indirect composite after 24 hours and after 10 months of storage.

Material	24 hours	10 months of storage
Sinfony	35.9 (5.4) Aa	33.2 (4.8) Aa
Signum	36.5 (6.1) Aa	29.8 (7.5) Ba
Charisma	29.7 (5.0) Ba	23.0 (6.3) Cb
Filtek Z350	40.1 (5.3) Aa	24.4 (4.5) Cb

Means followed by different capital letters in the same column and small letters in the same line were significantly different (P<.05).

Table 4. Means (KHN – Kgf/mm^2) and standard deviations of Knoop hardness (KH) of direct and indirect composite after 24 hours and after 10 months of storage.

Material	24 hours	10 months of storage
Sinfony	27.9 (1.5) Ca	27.6 (2.6) Ba
Signum	39.7 (5.2) Ba	31.2 (8.0) Bb
Charisma	41.2 (4.2) Ba	31.1 (5.0) Bb
Filtek Z350	78.3 (6.3) Aa	58.5 (6.6) Ab

Means followed by different capital letters in the same column and small letters in the same line were significantly different (P<.05).

tensity light to enhance the degree of conversion of the resin matrix. In performing post-cure procedures on a resin based composite material, the expectation is that the conversion of the methacrylate group will be enhanced, increasing the cross-link density of the set material.[5,7] Several investigations have shown that a material may undergo changes in properties following post-curing and that some of these changes may be beneficial.15 Previous studies revealed significant improvement in mechanical properties following post-curing.[7,15]

However, according to the results of this study, the direct composite Filtek Z350 showed higher KH values than those for the indirect composites Signum and Sinfony. When the composites remained in distilled water for 10 months, Filtek Z350 maintained the highest values, followed by the other composites, but the indirect composites Sifony and Signum showed the same KH values of Charisma. Thus, the first hypothesis tested was rejected. The higher KH values observed for Filtek Z350 in this study could have been influenced by the higher filler content (78.5 wt%) of this material. The results corroborate this finding because Charisma (64 wt%) and Signum (70 wt%) showed intermediate KH values, and Sinfony (50 wt%) the lowest KH values after 24 h. In addition to the high contend of filler particles, the direct composite Filtek Z350 contains particles of zirconia instead of barium glass as filler, differing from the other composites studied. Thus, the filler type (zirconia) may also have influenced the highest KH values of Filtek Z350.

Unlike hardness, the indirect composites Sinfony and Signum showed the same DTS values of the direct composite Filtek Z350, after storage for 24 hours and higher DTS values than Filtek Z350 and Charisma, after storage of 10 months. This probably occurred due to the probable increase in the polymerization of indirect composites because of additional activation. Unlike KH which is more influenced by the type and amount of filler particles.

Apart from factors associated with material composition and curing, the conditions of the oral environment are an important factor in reference to considering the mechanical strength of composite materials. Water or other chemicals present in the oral cavity could, with time, decrease the mechanical properties of composites.[16,17] Hydrolytic degradation is a diffusion rate-dependent process, influenced by polymer type, filler particle type, and surface treatment of the filler particle. Aging in water appeared to increase filler particle pull out on the fractured surface, possibly due to breakdowns of the silane bond between the resin and the filler particle.[9,10]

In the current study, storage in water for 10 months caused no reduction in hardness for the Sinfony and DTS for the Sinfony and Signum, whereas the direct composites showed decreases in KH and DTS, respectively. It is hypothesized that water causes a softening of the polymer resin component by swelling the network and reducing the frictional forces between chains. However, only Sinfony was not influenced by storage. The second hypothesis was rejected.

The objective of a secondary polymerization is to maximize the degree of conversion of composites in order to improve mechanical and physical properties, durability, solvent resistance, and biocompatibility.[7,15,18,19] The presence of unpolymerized monomers in the matrix negatively affects the properties of composite materials and may induce surface degradation and discoloration.[9,10,16,17,20] Additional curing allows the higher mobility and reactivity of free radicals formed by light irradiation or by thermal decomposition with an increase in collision probability among the unreacted active groups.[7,8,14] When the composite is heated to a temperature above its glass transition, there is an increase in the molecular mobility of the polymer chains. Therefore, it may be possible to further the chemical reaction by enhancing the molecular mobility of existing free radicals and other reactive species.[21]

Thus, this process probably increased the degree of conversion and cross-link density of the indirect composites, and the long storage time did not influence the properties of the indirect composites.

CONCLUSION

Considering the limitations of study, the two hypotheses were not accepted:

Indirect composites did not show higher mechanical properties than direct resins, especially after storage for 24 hours.

Storage for 10 months in distilled water decreased the properties of the direct composites and the KH of the indirect composite Signum, but did not decreased the properties of the indirect composite Sinfony.

Clinical Conclusion

When properly indicated, indirect composite restorations may exhibit improved mechanical properties over time and therefore to increase the longevity of the composite restorations.

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