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Evaluation of diametral tensile strength and knoop microhardness of five nanofilled composites in dentin and enamel shades

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SUMMARY

The purpose of this study was to evaluate and compare the mechanical behavior of five nanofilled composites with dentin and enamel shades trough diametral tensile strength and knoop microhardness. 4 Seasons (Ivoclar Vivadent), Esthet X improved (Dentsply), Filtek Supreme (3M), Grandio (Voco) and Palfique Estelite (Tokuyama Dental Corp.) were used in this research. Twenty samples (6 mm diameter and 3 mm thickness) of each material were used. Ten samples were submitted to knoop microhardness and the others to diametral tensile strength examinations. The results were submitted to ANOVA and Tukey statistical tests (a=0.05). There were significant differences between the tested groups ($p<0.05$). The diametral tensile strength ranged from 36.08 (4 Seansons dentin) to 49.24 (Grandio enamel). The knoop microhardness ranged from 54.45 (Esthet X improved enamel) to 123.90 (Grandio dentin). A great variability in the mechanical behavior of the nanofilled composites was observed. However it was not recorded with dentin and enamel shades with the exception of Grandio.

Key words: nanofilled composite, diametral tensile strength, knoop microhardness.

INTRODUCTION

Composite resin technology has continuously evolved since its introduction by Bowen (1963) [1] as a reinforced Bis-GMA system. A major breakthrough in composite technology was the development of photo-curable resins [2]. A continued development resulted in materials with reduced particle size and increased filler loading that significantly improved the universal applicability of light-cured composite resins [3].

Resin composites are widely used in dentistry and have become one of the most commonly used esthetic restorative materials because of their adequate strength, excellent esthetics, moderate cost compared with ceramics, ability to be bonded to tooth structure [4], improvements in composition, simplification of the adhesive procedures and the decline in amalgam usage due to fear of mercury toxicity [5] represent additional advantages. During the last decades, the increasing demand for esthetic dentistry have led to the development of resin composite materials for direct restorations with improved physical and mechanical properties, esthetics and durability. The latest development in the field has been the introduction of nanofilled materials by combining nanometric particles and nanoclusters in a conventional resin matrix. Nanofilled materials are believed to offer excellent wear resistance, strength and ultimate esthetics due to their exceptional polishability, polish retention and lustrous appearance [6].

The essence of nanotechnology is in the creation and utilization of materials and devices at the level of atoms, molecules, and supramolecular structures, and in the exploitation of unique properties and phenomena of particles [7] with size ranging from 0.1 to 100 nanometers. The compressive and diametral strengths and the fracture resistance of the nanocomposite materials are equivalent to or higher than those of the other commercial composites tested (hybrids, microhybrids and microfill) [8]. Nanofilled resin composites show mechanical properties at least as good as those of universal hybrids and could thus be used for the same clinical indications as well as for anterior restorations due to their high aesthetic properties [9]. Therefore, the aim of this study was to evaluate and compare the mechanical behavior of five nanofilled composites with dentin and enamel shades applying diametral tensile strength and knoop microhardness.

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MATERIAL AND METHODS

The nanofilled composites used in this study were: 4 Seasons (Ivoclar Vivadent, Schaan, Liechtenstein), Esthet X improved (Dentisply Caulk, Milford, DE, USA), Filtek Supreme (3MEspe, St. Paul, MN, USA), Grandio (Voco, Cuxhaven, Germany), Palfique (Tokuyama Dental Corp., Tokyo, Japan), in dentin and enamel A2 shades (Table 1).

Table 1. Descriptive table of materials used in the research.

Group	Manufacture	Organic matrix	Filler
4 Seasons	Ivoclar Vivadent	Bis-GMA, TEGDMA, UDMA	76 wt% of barium glass filler, ytterbium trifluoride, Ba-Al-fluorsilicate glass and high dispersed silica
Esthet X improved	Dentsply	UDMA, Bis-GMA, TEGDMA	60 vol% of barium boron fluoroalumino silicate glass with particle sizes of 0.6–0.8 mm and silica nanofiller (0.04 mm)
Filtek Supreme	3M Espe	Bis-GMA, Bis-EMA, UDMA, TEGDMA	78.5 wt%, combination of aggregated zirconia/silica cluster filler with primary particle size of 5-20 nm, and nonagglomerated 20-nm silica filler
Grandio	Voco	Bis-GMA, TEGDMA	87 wt% of spherical silicium dioxide 20-50 nm and glass ceramic fine particles
Palfique Estelite	Tokuyama Dental Corp.	Bis-GMA, TEGDMA	82 wt% of 50-100nm spherical silica-zirconia filler and silica-zirconia prepolimerized

Table 2. Descriptive table of results and statistical differences between groups

Group	Diametral tensile strength		Knoop microhardness	
	MPa	Std. deviation	KHN	Std. deviation
4 Seasons dentin	36.08 B	8.71	61.61 b	1.51
4 Seasons enamel	39.26 AB	8.09	57.98 b	1.29
Esthet X improved dentin	44.44 AB	10.95	73.61 b	1.97
Esthet X improved enamel	45.12 AB	8.09	54.45 b	1.47
Grandio dentin	45.23 AB	5.41	123.90 a	8.19
Grandio enamel	49.24 A	7.10	74.89 b	9.48
Filtek Supreme dentin	46.86 AB	11.51	101.30 a	2.58
Filtek Supreme enamel	48.25 AB	9.83	123.10 a	3.51
Palfique Estelite dentin	38.69 AB	6.59	55.89 b	9.67
Palfique Estelite enamel	38.58 AB	5.59	61.70 b	10.92

A, B, a, b – averages followed by different letters are statistically different ($p < 0.05$)

Twenty samples of each material were made using a bipartite PTFE mould (6 mm diameter and 3 mm thickness). The composite resins were inserted in two increments and lightcured according to each manufacturer's directions. The samples were stored in distilled water at 37°C for 24 hours prior to testing. After that, ten samples were mounted in a universal testing machine EMIC DL-2000 (Emic, São José dos Pinhais, PR, Brazil) and tested with 0.5 mm/min of cross-head speed. The diametral tensile strength (MPa) was converted using the following formula: $(2\times p)/(\pi\times d\times t)$. Were p is the ultimate tensile strength (N), d is the diameter (6 mm) and t is the thickness (3 mm).

The other ten samples were flattened and polished with carbide sandpaper #1000, #1500 and #2000 and then submitted to knoop microhardness tester (Shimadzu HMV, Shimadzu, Kyoto, Japan) using a load of 100 g for 15 s. The results of each test were analyzed with ANOVA and Tukey statistical tests (a=0.05).

RESULTS

The results are summarized in Table 2. A significant difference was observed ($p = 0.006$) when diametral tensile strength of nanofilled composites where compared (Grandio enamel ≥ Filtek Supreme enamel = Filtek Supreme dentin = Grandio dentin = Esthet X Improved enamel = Esthet X Improved dentin = 4 Seasons enamel = Palfique Estelite dentin = Palfique Estelite enamel ≥ 4 Seasons dentin). The diametral tensile strength results ranged from 49.24 (±7.10, Grandio enamel) to 36.08 (±8.71, 4 Seasons dentin).

The microhardness results were statistically different applying ANOVA (p=0.001). By using Tukey's multiple comparison test, it was possible to verify that Grandio dentin (123.90), Filtek Supreme enamel (123.10) and dentin (101.30) showed the highest average values, however without statistical difference (p=0.142). All other groups did not show statistically significant differences (p=0.252). The knoop microhardness values ranged from 123.90 (±8.19, Grandio dentin) to 54.45 (±1.47, Esthet X improved enamel).

DISCUSSION

First of all, the clinician must understand the nanometer concept. This is a scale that is equal to 10^{-3} µm, in other words, 1 µm is equal to 1000 nm. Everything might be measured in nanometers, however it would not be practical. In dentistry, nanofiller is a inorganic particle with average size of 40 nm or 0.04 µm. This size, however, is not an innovation in dental composites, because the microfilled composites have been reached the same filler size (40 nm) since 70's. The real innovation is the nanofiller's possibility to improve the load of the inorganic phase. Microfilled composites have 50 wt% of inorganic phase compared to 80 wt% for the nanofilled. A higher amount of filler content implies better mechanical behavior [10].

The objective of nanotechnology is to develop a dental filling material that might be used in all areas of the mouth with high initial polish and superior polish retention (typical of microfills), as well as excellent mechanical properties suitable for high stress bearing restorations (typical for hybrid composites) [8]. To make filler particles, milling procedure is used, but this procedure usually cannot reduce the filler particle size below 100 nm. Nanotechnology or molecular manufacturing may provide composite resins with filler particles that are smaller, that can be dispersed in higher concentrations, and are polymerized into the resin system with molecules designed to be compatible when coupled with a polymer, and provide unique characteristics (physical, mechanical and optical). Optimizing the adhesion of restorative biomaterials to the mineralized hard tissues of the tooth is a decisive factor for enhancing the mechanical strength, marginal adaptation and seal, in order to improve the reliability and longevity of the adhesive restoration. The particle size of conventional composites are so dissimilar to the structural sizes of the hydroxyapatite crystal, dentinal tubule, and enamel rod, that there is a potential for failures in adhesion between the macroscopic (40 nm to 0.7 nm) restorative material and the nanoscopic (1nm to 10 nm in size) tooth structure [3].

The diametral tensile strength is a mechanical property used to understand the behavior of brittle materials when exposed to tensile stress commonly observed in anterior restorations. The results (in MPa) obtained in this study is similar to the average previously recorded as 38.69 for Palfique Estelite [11], 44.6 for Esthet X [12] and 44.42 for Supreme [13]. However, different averages for the same materials has been reported as 76.6 for Esthet X, 87.6 for Supreme translucent (enamel) and 80.7 for Filtek Supreme Standard (dentin) [8].

Restorations in functional areas are exposed to attrition and wear and microhardness may determine the abrasion resistance. The knoop microhardness observed to Esthet X 54.45 (±1.47) comply with 57.8 (±2.5) previously registered in the dental literature [14] validating the used methodology.

The differences between groups could be explained by the nanofiller content (Wt%). Nanofillers have higher contact surface with the organic phase when compared to minifilled composites, consequently improving the material hardness [15]. Unfortunately, full information is not available about weight filler content, i.e., Palfique Estelite has 82 Wt% according to the manufacturer which is different from 68.55 (±0.01) Wt% recorded by an independent research [16]. The weight filler content is directly correlated to microhardness values [17] and a strong positive correlation ($0.88 < r < 0.96$) was registered [18]. Therefore, the higher averages knoop microhardness test values observed with Grandio and Supreme could be explained by their filler content.

Within the limitations of any in vitro study, the results show a great variability in the mechanical behavior of nanofilled composites ($p<0.05$). However, when enamel and dentin shades were compared inside the same material, it was possible to conclude that there was no statistical difference with the exception of Grandio.

CONCLUSIONS

A great variability in the mechanical behavior of nanofilled composites was observed. However, this was not recorded between dentin and enamel shades with the exception of Grandio. Nanofilled composites do not behave similarly in diametral tensile strength and knoop microhardness tests. The highest diametral tensile strength was observed with Grandio dentin while 4 Seasons dentin showed the lowest average value for that test ($p<0.05$). For knoop microhardness, Filtek Supreme in both shades and Grandio dentin showed the highest average in comparison to all other groups ($p<0.05$). Further studies should be carried out to improve the knowledge of the mechanical behavior of nanofilled composites.

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