

Nanohybrid Resin Composites: Nanofiller Loaded Materials or Traditional Microhybrid Resins?

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Clinical Relevance

Under clinical conditions, nanohybrid resin composites may not perform comparable to nanofilled materials.

SUMMARY

Nanohybrid resin composites present conventional particles to be mixed with nanomeric fillers and, therefore, it is unknown whether they

may perform similarly to nanofilled or microhybrid resins. The current study investigated the properties of nanohybrid resins (TPH³, Grandio, Premise, Concept Advanced) in comparison with a nanofilled (Supreme XT) and a microhybrid (Z250) composite. The inorganic fillers were characterized by SEM/EDS analysis. Diametral tensile strength (DTS), surface roughness before and after toothbrush abrasion, Knoop Hardness (KHN), water sorption and solubility were evaluated. The data were separately analyzed by ANOVA and the Student-Newman-Keuls' tests ($p < 0.05$). The results of all analyses were material-dependent. Noticeable differences in filler size and shape were detected among the materials. Supreme XT generally showed higher DTS and KHN compared to all the nanohybrids and also showed lower surface roughness before and after toothbrush abrasion compared to most of the materials tested. Similar results were generally detected for the nanohybrids compared with the microhybrid material. In conclusion, the nanohybrid resins generally presented inferior properties compared with the nanofilled composite and either similar or slightly better properties com-

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pared to the microhybrid material. Under clinical conditions, nanohybrid resins may not perform similarly to nanofilled materials.

INTRODUCTION

Resin-based restoratives are increasingly being used in dentistry, mainly because of their esthetic quality and good physical properties. A variety of dental composites that could be used in both anterior and posterior areas is available for clinical use, presenting a wide range of organic and inorganic components that may affect both their handling characteristics and clinical service. The introduction of well-dispersed inorganic particles into a resin matrix has been shown to be extremely effective for improving the performance of polymer composites.¹⁻³ The fillers used in dental resins directly affect their radiopacity, properties, wear resistance and elastic modulus.³⁻⁵ Therefore, resin composites have usually been classified according to filler features, such as type, distribution or average particle size.

In addition to traditional microhybrid and microfilled materials, nanofilled and nanohybrid composites were more recently introduced in an endeavor to provide a material presenting high initial polishing combined with superior polish and gloss retention.^{3,5-10} Nevertheless, while nanofilled composites use nanosized particles throughout the resin matrix,⁹ nanohybrids take the approach of combining nanomeric and conventional fillers,¹¹ and this characteristic is similar to microhybrid composites. Therefore, one can speculate that nanohybrids may still suffer from the loss of large particles, as microhybrids do, and it is questionable whether they are really innovative materials and whether they should be defined as nanofiller-loaded resins or traditional microhybrid composites instead.¹²

It is known that the shape, amount and size of the particles reinforcing the composite might affect their prop-

erties;^{2-3,5} finer particle size results in less interparticle spacing, more protection of the softer resin matrix and less filler plucking.¹²⁻¹³ Nonetheless, little is known as regards the properties of nanohybrid resin composites. Turssi and others,¹³ for instance, reported that, in terms of wear and fatigue resistance, nano-structured composites may perform either similarly or comparatively worse than microfilled materials, and Yesil and others¹⁴ reported that nanofillers might not significantly improve wear resistance compared with microhybrid and microfilled composites.

The current study evaluated the properties of commercial nanohybrid resin composites (TPH³, Grandio, Premise, Concept Advanced) and compared them with nanofilled (Filtek Supreme XT) and microhybrid (Filtek Z250) materials in an effort to identify whether nanohybrids should be classified as nanofiller-loaded materials or traditional microhybrid composites. The hypothesis tested was that nanohybrid materials would be shown to have properties similar to those of traditional microhybrid, and not of nanofilled composites.

METHODS AND MATERIALS

Resin Composites Tested

The resin composites evaluated in the current study and their compositions are shown in Table 1. Nanohybrids: TPH³ (Dentsply Caulk, Milford, DE, USA), Grandio (VoCo, Cuxhaven, Germany), Premise (Kerr, Orange, CA, USA) and Concept Advanced (Vigodent, Rio de Janeiro, RJ, Brazil); nanofilled: Filtek Supreme XT (3M ESPE, St Paul, MN, USA); microhybrid: Filtek Z250 (3M ESPE). All the materials were of shade A2 or corresponding color. Although classification criteria among the different manufacturers may vary, the authors of the current study classified all materials presenting range of fillers size above 100 nm as “nanohybrids.”

Table 1: Materials Used in This Study				
Resin Composite	Manufacturer	Classification*	Resin Phase**	Filler**
Filtek Z250	3M ESPE	Microhybrid	Bis-GMA, UDMA, Bis-EMA, TEGDMA	Zirconia, silica (0.01–3.5 µm, 75 wt%)
Filtek Supreme XT	3M ESPE	Nanofilled	Bis-GMA, UDMA, Bis-EMA, TEGDMA	Zirconia, silica (5–20 nm, 78.5 wt%)
TPH ³	Dentsply Caulk	Nanohybrid	Bis-GMA, Bis-EMA, TEGDMA	TiO ₂ , silica, Ba-B-F-Al-Si glass (0.02–1 µm, 75 wt%)
Grandio	VoCo	Nanohybrid	Bis-GMA, UDMA, TEGDMA	Silica, glass ceramic (0.02–1 µm, 87 wt%)
Premise	Kerr	Nanohybrid	Bis-EMA, TEGDMA	Prepolymer, silica, Ba glass (0.02–0.4 µm, 84 wt%)
Concept Advanced	Vigodent	Nanohybrid	Bis-GMA, UDMA	Silica, Ba-Al-Si glass (0.01–2 µm, 77.5 wt%)
*Although classification criteria among different manufacturers may vary, the authors of the current study classified all materials presenting range of fillers size above 100 nm as “nanohybrids.”				
**Information provided by the manufacturers.				
Bis-GMA: bisphenol-A glycidyl dimethacrylate; UDMA: urethane dimethacrylate; Bis-EMA: bisphenol-A ethoxylated dimethacrylate; TEGDMA: triethylene glycol dimethacrylate.				

Filler Characterization

The unpolymerized resin phase of each composite was removed by dissolving 0.2 g of each material in 100% acetone and additionally in chloroform for 24-hour periods. The remaining filler particles were placed in 100% ethanol; the suspension was smeared on a metal stub and dried at 37°C for 24 hours. The materials were coated with gold and examined by scanning electron microscopy (SEM—JSM5600LV; JEOL Inc, Peabody, MA, USA) to investigate their filler morphology and size. An additional batch of fillers was coated with carbon and analyzed using energy dispersive x-ray spectroscopy (EDS, Noran Instruments, Middleton, WI, USA) to assess the elemental composition and percentage distribution of the elements present in the filler particles.

Diametral Tensile Strength

Five cylindrical specimens (4 mm diameter x 2 mm thick) per resin composite were obtained by placing the materials into elastomer molds and light-curing them for 20 seconds from both the top and bottom surfaces using a quartz-tungsten-halogen curing device (XL2500, 3M ESPE—700 mW/cm²). After storage in distilled water at 37°C for 24 hours, the specimens were wet-polished with 1200-grit SiC abrasive papers. The diametral tensile strength (DTS) test was performed in a mechanical testing machine (model 4411; Instron, Canton, MA, USA) at a crosshead speed of 0.5 mm/minute. The specimens were positioned vertically on the base of the machine and subjected to compressive loading until failure. Means were calculated in MPa and the data submitted to ANOVA and Student-Newman-Keuls' test ($p < 0.05$).

Toothbrush Abrasion

Eight conical specimens per composite (5 mm top diameter x 4 mm bottom diameter x 1 mm thick) were obtained using brass molds, following the same photo-activation protocol for the DTS. After storage in distilled water for 24 hours at 37°C, the top surfaces were polished with medium, fine and superfine aluminum oxide discs (Sof-Lex system, 3M ESPE), and the samples were ultrasonically cleaned in distilled water for 10 minutes. Baseline surface roughness readings were taken on the top diameter of the samples by rotating them clockwise at random angles through a surface profilometer (Surfcorder SE1700, Kosaka Lab, Tokyo, Japan) equipped with a diamond stylus (0.5 µm tip radius) accurate to 0.01 µm. The stylus traversed across the diameter of each sample three times, and the mean roughness parameter for each specimen (R_a , µm) was recorded as the mean of the three readings.

Toothbrush abrasion was carried out on a multi-station brushing device. The top diameter of each sample was brushed using a soft, nylon-bristle toothbrush (Johnson & Johnson, Langhorne, PA, USA, 32 tufts, 60

bristles per tuft). The specimens were completely immersed in slurry of dentifrice (Colgate Classic, Hollywood, FL, USA) and distilled water (1:2 weight ratio). In total, 30,000 strokes (complete forward and reverse movement) were performed at a frequency of 4 Hz. The specimens were then cleaned with a one-minute air/water spray, followed by a 10 minute ultrasonic bath, and surface roughness was determined again. The data were submitted to Repeated Measures ANOVA and Student-Newman-Keuls' test ($p < 0.05$).

Hardness

Hardness readings were performed on the non-brushed bottom diameter of the samples that were retrieved from the toothbrush abrasion using an indenter (HMV-2; Shimadzu Corp, Tokyo, Japan). The bottom surfaces were polished with medium, fine and superfine aluminum oxide discs (Sof-Lex system, 3M ESPE), and the samples were ultrasonically cleaned in distilled water for 10 minutes. For each specimen, five indentations were performed under a load of 50 g for 15 seconds. The Knoop Hardness number (KHN, kg/mm²) for each specimen was recorded as the average of the five readings. The data were submitted to ANOVA and the Student-Newman-Keuls' test ($p < 0.05$).

Water Sorption and Solubility

Five cylindrical specimens (4 mm in diameter x 1 mm thick) were obtained for each material following the same procedures described for the DTS test. After calculating the volume (V , in mm³) of the specimens, they were stored in a desiccator at 37°C and repeatedly weighed after 24-hour intervals using an analytical balance (JK-180; Chyo Balance Corp, Tokyo, Japan) accurate to 0.1 mg until a constant mass (m_1) was obtained. The samples were then individually placed in sealed vials, immersed in distilled water and stored at 37°C.

After seven days, the surface water of the specimens was removed by blotting with absorbent paper and waving the specimen in the air for 10 seconds. The weighing procedures were repeated, by which m_2 was recorded. The specimens were then placed in the desiccator again, at 37°C, and reweighed until a constant dry mass (m_3) was obtained. Water sorption (W_{sp}) and solubility (W_{sl}), given in µg/mm³, were calculated as follows: $W_{sp} = (m_2 - m_3)/V$; $W_{sl} = (m_1 - m_3)/V$. The data were separately submitted to ANOVA and the Student-Newman-Keuls' test ($p < 0.05$).

RESULTS

Results for the EDS analysis are shown in Table 2. While both the nanofilled and microhybrid resin composites presented Si and Zr as the main components of the inorganic fillers, the nanohybrids presented Si and Ba as main components and also presented a small amount of Al. SEM pictures of the fillers for each composite are shown in Figures 1 and 2. The microhybrid

Table 2: Elemental Composition (wt%) of the Inorganic Phase of Each Material				
Resin Composite	Ba	Al	Si	Zr
Z250	-	-	66%	34%
Supreme XT	-	-	57%	43%
TPH ³	57%	6%	37%	-
Grandio	11%	12%	77%	-
Premise	56%	5%	39%	-
Concept Advanced	49%	7%	44%	-
Values are relative to the mass fraction of each component present in the filler particles.				

Table 3: Means (Standard Deviations) for DTS, KHN, W _{sp} and W _{sl}				
Resin Composite	DTS (MPa)	KHN (kg/mm ²)	W _{sp} (µg/mm ³)	W _{sl} (µg/mm ³)
Z250	53.7 (5.1) ab	69.6 (6.1) b	30.7 (4.9) a	1.9 (1.2) a
Supreme XT	58.0 (11.0) a	72.4 (7.4) b	29.1 (5.1) a	2.9 (2.1) a
TPH ³	53.4 (4.8) ab	54.9 (2.6) d	26.4 (6.4) a	2.9 (1.9) a
Grandio	54.6 (11.7) ab	111.7 (13.6) a	15.1 (5.3) b	3.1 (1.8) a
Premise	40.1 (11.1) b	62.4 (6.2) c	18.1 (4.5) ab	5.1 (2.5) a
Concept Advanced	38.8 (7.1) b	44.8 (2.4) e	17.3 (4.3) b	3.1 (2.2) a
Means followed by distinct letters in the same column are significantly different at p<0.05.				

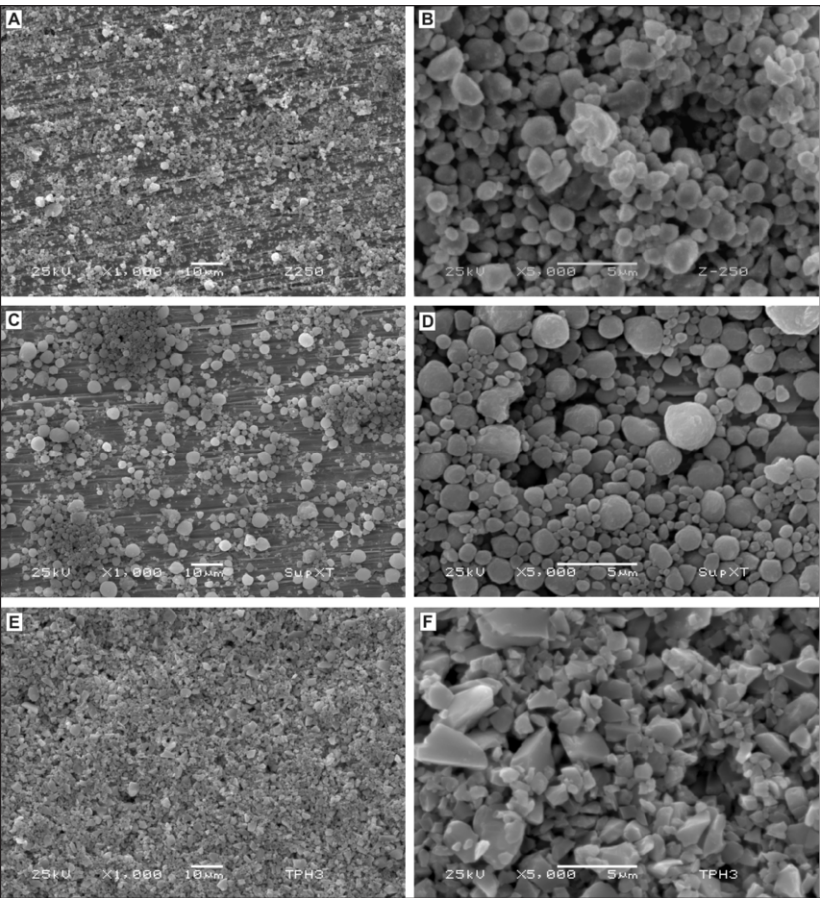


Figure 1. SEM pictures of inorganic fillers. Figures 1A and 1B Z250–microhybrid: round-shaped small and medium particles are predominant; Figures 1C and 1D–Supreme XT nanofilled: round-shaped clusters are observed, although the magnification is not sufficient to accurately observe the individual nanosized particles; Figures 1E and 1F–TPH³ nanohybrid: irregular-shaped small and medium particles are present.

Z250 presented round-shaped small and medium particles, while the nanofilled Supreme XT was shown to have round-shaped nanoclusters. For the nanohybrids, TPH³ presented irregular-shaped small and medium particles, Grandio presented very large irregular-shaped particles mixed with medium fillers, Premise showed very large irregular-shaped clusters formed by small particles and

Concept presented large irregular-shaped clusters, also formed by small fillers. Results for DTS and KHN are shown in Table 3. Supreme XT showed the highest DTS values, which were significantly higher than those of Premise ($p=0.032$) and Concept Advanced ($p=0.026$) but similar to those of the other composites ($p\geq0.063$). As regards hardness, almost all the materials presented significantly different values when compared with each other: Grandio > Z250 and Supreme XT > Premise > TPH³ > Concept Advanced ($p\leq0.022$).

Results for water sorption and solubility are shown in Table 3. Z250, Supreme XT and TPH³ presented significantly higher sorption than Grandio and Concept Advanced ($p\leq0.012$), which were similar. Premise presented intermediary sorption values, with no significant differences compared with any other resin composite ($p\geq0.068$). On the other hand, no significant differences in solubility were detected among all composites ($p\geq0.183$). Results for the surface roughness analysis are shown in Table 4. Before toothbrush abrasion, Supreme XT, Premise and Concept Advanced showed significantly smoother surfaces compared with the other resin composites ($p<0.001$), which were similar ($p\geq0.583$). After toothbrushing, the results were: Supreme XT and Premise < Concept Advanced < TPH³ and Z250 < Grandio ($p\leq0.042$). Comparing the times before and after abrasion, Z250 and

Table 4: Means (Standard Deviations) for Surface Roughness Before and After Toothbrush Abrasion

Resin Composite	Surface Roughness (Ra, µm)	
	Before	After
Z250	0.24 (0.09) A,a	0.27 (0.08) A,b
Supreme XT	0.09 (0.02) B,b	0.12 (0.02) A,d
TPH ³	0.25 (0.08) A,a	0.30 (0.09) A,b
Grandio	0.24 (0.04) B,a	0.36 (0.04) A,a
Premise	0.08 (0.01) B,b	0.11 (0.02) A,d
Concept Advanced	0.09 (0.01) B,b	0.16 (0.01) A,c

Means followed by distinct capital letters in the same line, and small letters in the same column, are significantly different at p<0.05.

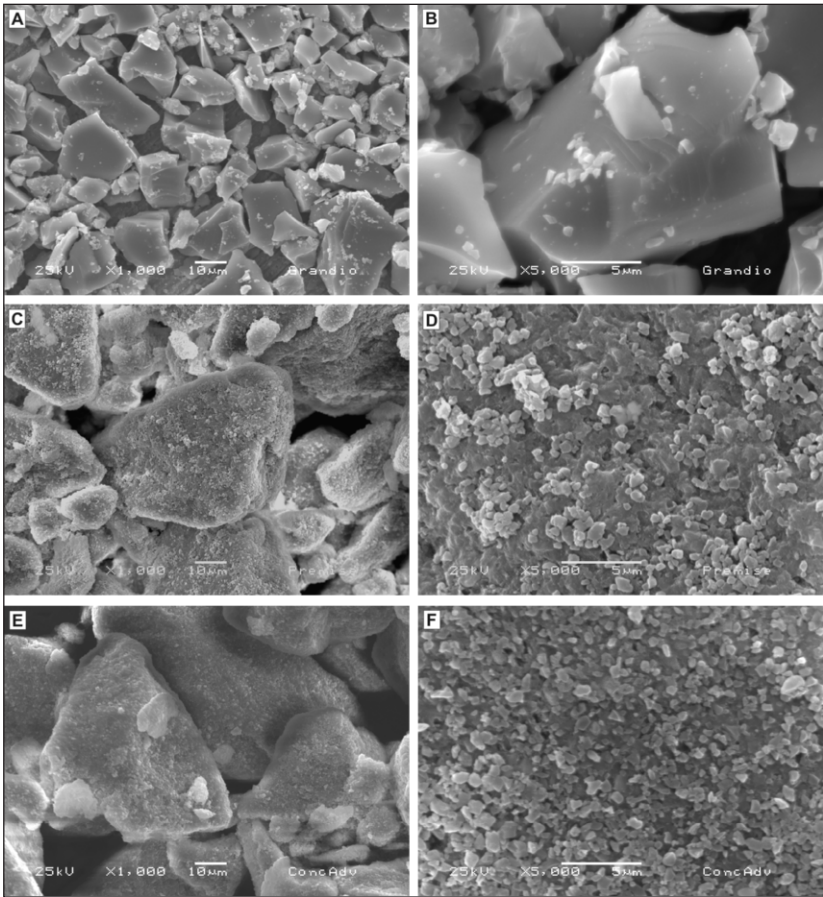


Figure 2. SEM pictures of the inorganic fillers. Figures 2A and 2 B–Grandio nanohybrid: very large, irregular-shaped particles are present, mixed with medium fillers; Figures 2C and 2D–Premise nanohybrid: very large, irregular-shaped clusters were detected, formed by small particles shown in the magnification; Figures 2E and 2F–Concept Advanced nanohybrid: large, irregular-shaped clusters are also present, and the small fillers that form it can be observed in the detail.

TPH³ showed no significant differences ($p \geq 0.431$), whereas all the other materials presented significantly rougher surfaces after toothbrushing ($p \leq 0.006$).

DISCUSSION

The current results indicate that, with regard to filler morphology, some nanohybrid composites presented

large clusters formed by small particles (Premise, Concept), while others presented large fillers (TPH³, Grandio). The nanotechnology concept refers to a field of applied science whose theme is the control of matter in the atomic and molecular scale, generally 100 nm or smaller.^{1,3,5} Therefore, the size of the fillers observed for the nanohybrid resins could *per se* be a reason to not refer to them as nano-structured materials, as microhybrid composites may also mix nano-sized particles with large fillers. In addition, the results of the EDS analysis showed that the composition of the fillers presented in nanohybrids is the same as almost all of the traditional hybrids, namely Ba-Al-Si glass fillers.^{3-5,12}

The shape of the fillers presented in the nanohybrids was also different when compared with the nanofilled material (Figures 1 and 2). The properties of nanoparticles and the resulting nanocomposites are size- and shape-dependent.² In order to produce inorganic nanoparticles, the materials are either mechanically crushed in a conventional manner or synthesized from precursor compounds by controlling the crystal growth kinetics.¹ The top-down approach is when macroscopic particles are downsized by physical means, such as mechanical grinding or high energy milling,¹ which seems to be the approach for the nanohybrids tested here. However, there are limiting factors to this approach, as particles become irregularly-shaped and sizes below 500 nm can hardly be generated.¹ On the other hand, the bottom-up approach starts at the other end: materials and phases are generated by physical or chemical means from precursor compounds forming nanoscale fillers,¹ allowing the production of round-shaped fillers less than 100 nm in diameter, such as those observed for the nanofilled resin.

With regard to the properties of the restoratives, the results were generally dependent on the material evaluated, especially with regard to filler features. Nanofilled Supreme XT showed significantly higher DTS than Premise and Concept, which were precisely the only two nanohybrids whose fillers were clusters rather than large individual particles. This suggests that the entanglement between the resin

components and the nanofillers is better for Supreme XT, enhancing the three-dimensional microstructure of the composite and improving its mechanical strength. The shape of the nanofillers in Supreme XT may also explain this result, as Kim and others¹⁵ reported that composites with round particles may present increased mechanical strength. This is further evidence that nanohybrids may not behave similarly to nanofills. Furthermore, similar DTS values were observed for Supreme XT compared with the remaining restoratives, which might be related to the large particles presented by the other materials.

The results indicated no trend towards the size or shape of fillers affecting hardness. Indeed, all materials generally presented different results in comparison with one another. Grandio, for instance, presented the highest KHN values, probably because of presenting large particles and the highest filler content. The similar results detected for Z250 and Supreme XT indicated that, not only the filler particles, but also the components of the resin phase themselves might interfere with hardness. The microhybrid and nanofilled composites tested here present the same organic matrix components in spite of the noticeable differences in filler size. Similarity in resin components might produce similar polymer network structures and therefore similar hardness results when the filler loading is similar.

When evaluating the surface smoothness before abrasion, the nanofilled material and both nanohybrids presenting clusters showed lower surface roughness than all the other materials, which indicates a beneficial effect of the presence of small fillers forming clusters. However, the great theoretical advantage of nanocomposites should be that they retain their polished surfaces over the course of time. After toothbrush abrasion, the nanofilled composite presented lower surface roughness than all the other materials, except for Premise. Concept also showed lower Ra values after toothbrushing compared with all the remaining materials. These outcomes again indicate that, in general, nanohybrids may not perform in a similar manner to nanofilled composites, but their performance is similar to, or sometimes slightly better than that of microhybrids. In corroboration, a recent study observed that, while nanofills could be expected to withstand the wear caused by brushing, nanohybrids had higher roughness levels.¹⁰ Another point to be highlighted is that the presence of clusters formed by small particles seems to reduce the surface roughness of the materials and possibly increase the retention of smoothness after abrasion, while very irregularly-shaped particles seem to decrease the retention of smoothness.

The results showing lower water sorption for Grandio and Concept are most probably related to the organic matrix components rather than the inorganic matrix

itself. Characteristics of the chemical structure of monomers, especially with regard to the potential for hydrogen bonding and polar interactions, present a strong influence on the hydrophilicity of the polymer network. The filler features would present a significant effect only if the amount of the fillers dispersed throughout the resin phase were different among composites, which is not the case. Furthermore, no significant differences in solubility were observed among the composites tested. This result indicates that the current resin composites are virtually insoluble in water, although it has been demonstrated that the effects of water on dental polymers might be affected by aging.⁴

All evaluations showed a material-dependent effect. The current results indicate that the behavior of nanohybrid resin composites is more closely related to that of microhybrid than nanofilled materials. Therefore, it seems that nanohybrids are not innovative materials and that the "new" classification is probably related to marketing strategies. This is clinically relevant, considering that professionals could opt for using a nanohybrid resin in order to obtain high polish retention with time, but this effect is uncertain. Notwithstanding, the addition of small particles forming large clusters seems to be the most efficient way to obtain the retention of high smoothness, but the shape of the particles forming the cluster may also affect the outcomes. Nonetheless, the results of the current study do not take into account the effects that pH changes, salivary enzymes and mechanical fatigue might have on clinical performance. The clinical evaluation of nanohybrids compared with microhybrids and nanofills would more properly indicate differences in performance among these materials.

CONCLUSIONS

The nanohybrid resins tested generally presented inferior properties compared with the nanofilled composite and either similar or slightly better properties compared with the microhybrid material.

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