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Research Article

The evolution of contemporary nano-filled composite materials for dentistry clinical applications

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Abstract

Bulk-fill composites have emerged, possibly, as a new “class” of resin-based composites, which are claimed to enable restoration in thick layers, up to 4 mm. The most recent innovation in composite resin technology is the application of nanocomposite theories in restorative materials. Contemporary nanocomposite materials deliver increased aesthetics, strength, and durability, combining scientific principles for increased longevity. Direct applications of a nanocomposite resin materials will address the clinical applications of such a system in the anterior region. This study was to quantify and compare the mechanical properties of some present filling materials in particular two commercially available nanocomposite restorative materials. Z350 TM (Filtek™ Z350) and Grandio™, were polymerized with a LED light for 20 seconds and subjected to mechanical tests. Properties are tested for Grandio exhibited significantly higher mean flexural strength values (σ_f) compared to Z350 (89.1 MPa vs 61.9 MPa), that is significantly higher top microhardness values respect to Z350. Additionally, microhardness for the top surfaces of each composite were compared with their corresponding bottom surfaces, the bottom surfaces demonstrated significantly lower readings. Under optimal curing conditions, the physico-mechanical properties of most currently available bulk-fill composites to those of two conventional composite materials chosen as references, one highly filled and one flowable “nano-hybrid” composite. Tetric EvoCeram Bulk Fill (Ivoclar-Vivadent), Venus Bulk Fill (Heraeus-Kulzer), SDR (Dentsply), X-tra Fil (VOCO), X-tra Base (VOCO), Sonic Fill (Kerr), Filtek Bulk Fill (3M-Espe), Xenius (GC) were compared to the two reference materials. The materials were light-cured for 40 s in a 2 x 2 x 25 mm³ Teflon mould.

Keywords: Dental nanocomposites; resin; Microhardness; Polymer network density; Filtek Z350; Grandio.

Introduction

Aesthetic dentistry continues to evolve through innovations in bonding systems, restorative materials, function-based treatment, and minimally-invasive preparation designs. Such advances have increased opportunities available to discriminating patients and have provided solutions to many of the restorative and aesthetic

challenges faced by clinicians. A major breakthrough in composite technology surfaced with the development of photo-curable composite resins. These light-initiated composite resins were more color stable than the earlier self-cured composites and had smaller filler particles that improved the material’s wear resistance.[1,2].

The effect of a high concentration carbamide peroxide-containing home bleaching system (Opalescence PF) and a hydrogen peroxide-containing over-the-counter bleaching system (Treswhite Supreme) on the microhardness of two nanocomposites (Filtek Supreme XT and Premise) and leucite reinforced glass ceramic (Empress Esthetic), glass ceramic (Empress 2 layering), and feldspathic porcelain (Matchmaker MC), [3]. Then the specimens were polished with SiC paper and 1µm alumina polishing paste. After polishing, porcelain specimens were glazed in accordance with the manufacturer's instructions, and the specimens were treated with either Opalescence PF or Treswhite Supreme. The microhardness of the specimens before bleaching (baseline) and after bleaching was determined using a digital microhardness tester. Data were analyzed using the Mann-Whitney U-test and the Wilcoxon test. Opalescence PF significantly influenced the hardness of all the restorative materials. Considerable improvement since the use of photopolymerizable resin-based composite restorative materials are more frequently extended to large and deep cavities even if variable success, [4-5]. In such cases, incremental build-up of multiple thin layers are required because of the limited cure depth, [6-7] and second to potentially reduce the consequences of shrinkage stress, [8] although the latter theory has been disproved, [9]. Since the inception of resin based composite materials for use as restorative dental materials, continuous research and development has occurred to improve their mechanical properties, clinical handling and performance, [10]. These developments have focused primarily on reducing polymerization shrinkage and stresses by manipulating resin formulation and improvement of mechanical properties such as hardness, flexural strength, fracture toughness, and compressive strength by manipulating the filler factors such as size, shape and concentration of fillers or by the development of novel filler particles. It is generally accepted that an increase in the filler concentration of resin composites is associated with an increase in certain properties such as elastic modulus, flexural strength, hardness and compressive strength, [11-12]. In

studies on the effect of filler loading on the mechanical properties of hybrid composites it was determined that composites with the highest filler by volume exhibited highest values of flexural strength, flexural modulus, hardness and fracture toughness, [13]. Nanotechnology with composite resin, particle size and quantity are crucial when determining how to best utilize the restorative materials. Alteration of the filler component remains the most significant development in the evolution of composite resins, [14] because the filler particle size, distribution, and the quantity incorporated dramatically influence the mechanical properties and clinical success of composite resins, [15]. A modern example has seen the increasing popularity amongst dental practitioners of so-called "bulk-fill" materials, which are claimed to enable the restoration build-up in thick layers, up to 4 mm. This new material class includes flowable and higher viscosity paste material types. There currently exists a growing trend in the use of bulk-fill materials amongst practitioners due to a more simplified procedure, [16]. Hardness is an indirect measure of the degree of conversion of the material and gives useful information on the depth of polymerization when such measurements are performed on the top and bottom surfaces of cured samples, [17-19]. Hardness can also give some indication of the material's polishability and abrasion resistance, [20]. Flexural strength and fracture toughness are the properties that characterize the fracture behavior of composites. Flexural strength is the material property that gives an indication on the quantity of flaws within the material that may have the potential to cause catastrophic failure once subjected to loading whilst fracture toughness is a measure of the stress intensity at the tip of a flaw which may propagate in an unstable manner, [21-22].

Materials and Methods

Filtek™ Z350, an easy-to-use nanofilled restorative for techniques requiring more fluid handling and flow. The compound exhibits great compressive and tensile strength and show high wear resistance make it ideal for a variety of

indications, including as a base/liner, pit & fissure sealant. Grandio is a dual-curing, radiopaque, flowable composite for core build-ups, with very good mechanical properties and contains 77% w/w inorganic fillers in a methacrylate matrix. The main currently available bulk-fill composites as well as a dual-cure composite in a single study were summarized in Table 1,[23]and to compare their physico-mechanical properties under optimal curing conditions to those of two conventional composite materials chosen as references, one highly filled and one flowable nano-hybrid composite: Grandio and Grandio Flow (VOCO) and the second Z350 with excellent compressive and tensile strength. The null hypothesis was that there are no differences in physicomachanical properties between neither of the so-called bulkfill composites, nor with two conventional composite materials chosen as controls. The materials used in the present investigation are presented in Table 1. They were placed in a 2 x 2 x 25 mm³. Teflon mould and light-cured by four 40s overlapping irradiations on the upper sample side to ensure optimal

mechanical properties. The light tip of the polywave LED light BluePhase G2 (Ivoclar-Vivadent, Schaan, Liechtenstein) was placed against a polyester film at the upper sample surface in order to minimize the effects of oxygen inhibition an dipolymerization was initiated using the high-power irradiation mode (1050 mW/cm² , measured by Bluephase Metre). After photopolymerization, the samples were carefully removed from the mould and stored dry for 24 h in the dark at room temperature (23 ± 1 °C) before analysis, to ensure that the polymerization process was complete prior to analysis,[24-25]. Vickers microhardness (VHN) measurements were carried out on The fractured samples. Since that surface was in direct contact with a polyester film providing a uniform surface lustre, no polishing was performed. The length of the diagonal of each indentation was measured directly using a graduated eye-lens. The elastic modulus (Emod) and flexural strength (*f*) were measured using a three-point bend test.

Table 1 – Characteristics of tested materials, [23]

Materials	Abbreviation	Manufacturer	Composite Type	Shade	Batch
Tetric Evo Ceram Bulk Fill	TECBF	Ivoclar-Vivadent (Schaan, Liechtenstein)	Bulk-fill paste composite	IVA	P63316
Venus Bulk Fill	VenusBF	Heraeus-Kuzer, (Hanau, Germany)	Bulk-fill flowable	U	10100
Surefil SDR Flow	SDR	Dentsply, (Konstanz, Germany)	Bulk-fill flowable composite	A3	120 3000 624
X-tra fil	X-traF	Voco (Cuxhaven, Germany)	Bulk-fill paste composite	U	1209605
X-tra base	X-traB	Voco (Cuxhaven, Germany)	Bulk-fill flowable composite	U	1208392
Sonic Fill	SonicF	Kerr (Orange, CA, USA)	Bulk-fill paste composite with sonic hand-piece	A3	3851500
Filtek Bulk Fill (Z350)	FiltekBF	3M-Espe (St. Paul, MN, USA)	Bulk-fill flowable composite	U	N370958
Xenius (previous version of Ever-X posterior)	Xenius	GC Europe (Leuven, Belgium)	Bulk-fill paste composite with glass microfibres	B	20071108
Coltene Dual-cure Bulk-Fill	Col DCBF	Coltene-Whaledent (Altsta tten, Switzerland)	Dual-cure Bulk-fill flowable composite	U	20071108
Grandio	Grandio	Voco (Cuxhaven, Germany)	Hybrid paste composite	A3	120983
Grandio Flow	GrandioF	Voco (Cuxhaven, Germany)	Hybrid flowable composite	A3	1208317

Since the particle concentration depends on the viscosity, the filler loading that can be attained is 69% by volume and 84% by weight, which results in reduced polymerization shrinkage and shrinkage stress. The polymerization shrinkage is reported to be 1.4% to 1.6%. As the interparticle dimension decreases, the load bearing stress on

the resin is reduced, inhibiting crack formation and propagation, [26]. The spheroidal shape provides smooth and rounded edges, distributing stress more uniformly throughout the composite resin. This phenomenon has been termed the “roller bearing” effect, and is said to improve the sculptability and handling characteristics (Figure 1).



Figure 1; a) A small increment of translucent-shaded nanoparticle hybrid resin (Premise, Kerr/Sybron, Orange, CA) was placed over the previous layer. b) The translucent layer of resin was then sculpted, adapted, and smoothed cervicoincisally and mesiodistally to obtain an anatomically correct profile, [27].

It is suggested that the long-term polishing retention arises from the exposed nanoparticle fillers in the resin matrix during wear, tooth

brushing, or polishing. These fillers may act as a nano-polishing medium on the surface of the composite (Figure 2).



Figure 2: a) To reproduce the natural form and texture, the initial contouring was performed with a 30-fluted needle-shaped finishing bur. b) Postoperative appearance demonstrates aesthetic form and color developed using a nanocomposite system for optimal results, [27].

Five specimens of each composite were fabricated using a rectangular brass mold (31 X 2 X 2 mm³) and Mylar strips. A mask of aluminum foil with a circular window cut to the diameter of the curing tip and the width of the specimen (2 mm) was employed to reduce the effects of overcuring. The mask was laid right up against the rectangular mold and after curing the first segment for 20 seconds the window was moved to the new location adjacent to the first section where curing was repeated. Immediately following curing the specimens were placed in a three point bending fixture on two parallel supports, 25 mm apart, in a Hounsfield H50KS TM tensometer and loaded at a crosshead speed of 0.5 mm/min until catastrophic failure occurred. Flexural strength, *f*, was calculated using the formula, [28]:

$$(f) = \frac{3Fl}{2bh^2} \quad (2)$$

where *F* is the load at failure, *l* is the distance between supports (i.e. 25 mm), *b* is the width of the sample and *h* is the height.

Results and Discussion

The mean values of flexural strength together with their significances for Z350 and Grandio are 61.9 and 89.1 MPa, respectively. This particular study evaluated mechanical properties immediately following specimen preparation. It is common to employ a storage regimen prior to assessing mechanical properties. Generally mechanical properties of resin composite are known to be affected by the presence of water, [10, 29]. Specifically, water storage has been shown to affect hardness of composite samples, with flexural modulus of certain composites being affected by more prolonged storage, [30]. In microscopic analysis of a nanofilled composite

following water storage, microcracks were observed at the interface between filler particles and the resin matrix with a reduction in fracture strength even after 24 hours, [10]. Additionally specimens stored dry but under room light may continue to polymerize due to post-irradiation polymerization, albeit at slower rates, which may affect certain physical properties such as hardness, [31].

Grandio displays significantly higher VHN (dry and ethanol) than all other materials, while a group of three materials display very low dry and ethanol VHN: FiltekBF, SDR and VenusBF. Only SonicF and X-traF compete with the values of GrandioFlow, the values of the other materials being significantly lower. As for the ratio between dry and ethanol VHN, half of the materials including both controls display high ratios (82.2–90%), while the ratios of the other half ranges from 68.7% down to 19.2% for SDR. The linear correlation coefficients of multivariate correlations between the investigated variables are reported in Table 2. The latter indicates several good linear correlations, notably between mechanical properties and filler fraction (*R* > 0.8). On the contrary, DC was poorly correlated with the mechanical properties (0.09 < *R* < 0.41). Large and significant differences (*p* < 0.001) were observed for all considered physico-mechanical properties (Filler mass fraction, DC, Emod, *f*, Vickers microhardness dry (VHN), ethanol (VHN) and their ratio) within the bulk-fill composite category as well as with the two conventional composites chosen as controls, which led to the rejection of the null hypothesis.

Table 2 – Multivariate correlation coefficients (Restricted maximum likely hood), [23].

	Filler mass fraction	Dry	Ethanol	Emod	<i>f</i>	DC
Filler mass fraction	1.00	0.86	0.84	0.84	0.83	0.43
Dry (VHN)	0.86	1.00	0.96	0.97	0.65	0.19
Ethanol (VHN)	0.84	0.96	1.00	0.92	0.65	0.09
Emod (Elastic modulus (GPa))	0.84	0.97	0.92	1.00	0.66	0.20
<i>f</i> (Flexural strength (MPa))	0.83	0.65	0.65	0.66	1.00	0.41
DC(The degree of conversion (DC, in %))	0.43	0.19	0.09	0.20	0.41	1.00

Conclusion

Although the nano scale is small in size, its potential is vast. Recent advances by scientists and engineers in manipulating matter at this small magnitude indicate potential contributions for applications of this nanoscience through developments of advanced restorative biomaterials. The continual development of this technology will improve the ability of scientists, manufacturers, and clinicians to create a more ideal composite. Within the limitations of this study, it can be concluded the restorative Grandio has greater observed values for the properties of flexural strength and hardness when compared with Z350. The reduction of time and improvement of convenience associated with bulk-fill materials is a clear advantage of this particular material class. However, a compromise with mechanical properties compared with more conventional commercially-available nano-hybrid materials was demonstrated by the present work. Given the lower mechanical properties of most bulk-fill materials compared to a highly filled nano-hybrid composite, their use for successful restorations under high occlusal load may be controversial. Besides, the significant decrease in surface hardness after ethanol storage of some of the bulk-fill materials investigated raises concern regarding long-term stability and suggests that these materials should be better prevented from direct contact with the oral cavity, which then, of course, reduces their convenience.

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