

Laboratory Research

Properties of a New Nanofiber Restorative Composite

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Clinical Relevance

There may not be any significant advantage to the use of the new nanofiber composite resin restorative material (NovaPro Fill) compared to use of the hybrid composite resins (Filtek Z250, Esthet-X HD).

SUMMARY

A new nanofiber-reinforced hybrid composite (NovaPro Fill, Nanova) was recently introduced with reportedly improved mechanical properties. The purpose of this study was to compare the properties (flexural strength/modulus, degree of conversion [DC], depth of cure, and polymerization shrinkage) of the nanofiber composite to those of traditional

hybrid composites (Filtek Z250, 3M ESPE; Esthet-X HD, Dentsply). To determine flexural strength and modulus, composite was placed in a rectangular mold, light-cured, stored for 24 hours, and then fractured in a universal testing machine. For degree of conversion, composite was placed in a cylindrical mold, light-cured, and stored for 24 hours. Measurements were made at the top and bottom surfaces using Fourier Transform Infrared Spectroscopy. To determine depth of cure, composite was placed in a cylindrical mold and light-cured. Uncured composite was scraped until polymerized resin was reached. Remaining composite was measured and divided by two. Polymerization shrinkage was determined by placing the composite material on a pedestal in a video-imaging device while light-curing. Shrinkage was determined after 10 minutes. Data were analyzed with one-way analysis of variance and Tukey post hoc test per property ($\alpha=0.05$). Compared to Filtek Z250, NovaPro Fill had significantly lower flexural strength and modulus, greater volumetric shrinkage, and similar depth of cure, but greater top and bottom DC. Compared to Esthet-X HD, NovaPro Fill had similar flexural strength, shrinkage, and top and bottom DC, but significantly greater depth of cure and flexural modulus.

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INTRODUCTION

Resin composites have become an integral part of the clinical discipline of operative dentistry. A meta-analysis by Heintze and others¹ found that more than 500 million direct dental restorations were placed each year worldwide, of which about 55% were composites or compomers. According to the National Institutes of Health, composites have an average survival time of 5.7 years, and failures are mainly due to secondary caries and fracture of the restoration.² Historically, composite restorations were advocated for use in areas of minimal stress.³ However, demands for naturalistic esthetics from patients as well as clinicians are increasing and have led to the popular use of resin-based materials on posterior teeth, where considerable mechanical challenges occur under function.⁴ To withstand these stresses, modification of filler particle size and morphology resulted in improved mechanical properties.⁵

Most composite materials are composed of a polymeric matrix (typically dimethacrylate), reinforcing fillers (typically radiopaque glass), a silane coupling agent to bind the filler to the matrix, and chemicals that promote or modulate the polymerization reaction. Because of the major influence of fillers on the physical properties of composites, their classification is primarily based on the type and size of filler particles.⁶

Composites have continued to improve via incorporation of various sizes and shapes of filler particles, starting with the macrofills, up to today's microfills, nanofills, and nanohybrids.⁶ Recently, a novel composite restorative material containing calcium-phosphate (hydroxyapatite) nanofibers (NovaPro Fill, Nanova, Columbia, MO, USA) has piqued many interests in the dental community, which seeks to understand the role of nanofiber inclusion and its impact in order to influence composite strength and reliability. The manufacturer claims⁷ that the nanofibers provide superior mechanical strength and greater degree of conversion (DC) than do most conventional composites that are without these reinforcements. Furthermore, past studies have shown that the inclusion of the nanofibers allows one to significantly increase composite physical properties.

In a study by Vidotti and others,⁸ experimental methacrylate composite beams were created by infiltrating polyacrylonitrile nanofiber mats, and greater tensile properties were observed when the beams were tested perpendicular to the direction of

the nanofiber mats. In another study by Guo and others,⁹ zirconia-silica and zirconia-yttria-silica ceramic nanofibers at various concentrations were placed in experimental composite formulations and compared to control composites without nanofillers. The incorporation of the ceramic nanofillers significantly enhanced the mechanical properties compared to the control. Additionally, impregnation of hydroxyapatite nanofibers into methacrylate resin has been shown¹⁰ to significantly improve the flexural strength of resin formulations. However, other studies^{9,10} have found that there was a limit to the amount of nanofiber content by weight before mechanical properties started to decrease. In addition, the DC decreased with the inclusion of nanofiber content.⁹

EverX Posterior (GC 3-2-14 Hongo, Bunkyo-ku, Tokyo, Japan), although not commercially available in the United States, is one example of a short-fiber-reinforced composite that is designed to be used as a dentin replacement in medium- to large-size Class II preparations (ie, sandwiched technique). A study by Abouelleil and others¹¹ found that with the addition of fibers to the methacrylate-based matrix, EverX Posterior could result in comparable or superior mechanical properties to other bulk-fill materials tested, whereas, a recent study by Fronza and others¹² found that the mechanical performance of EverX Posterior was intermediate in comparison to that of other bulk-fill materials.

No research has been published evaluating the new commercially available nanofiber-reinforced composite NovaPro Fill, designed to be incrementally placed to the cavosurface margins in posterior stress-bearing areas. The purpose of this study was to evaluate the properties of the new nanofiber composite resin compared to those of traditional hybrid composite resin restorative materials, Filtek Z250 (3M ESPE, St Paul, MN, USA) and Esthet-X HD (Dentsply, York, PA, USA). The null hypothesis was that there would be no difference in properties among the composite materials.

METHODS AND MATERIALS

Flexural strength and modulus, DC, depth of cure, and volumetric polymerization shrinkage were evaluated for the composites NovaPro Fill, Filtek Z250, and Esthet-X HD in shade A2. Filtek Z250 is a traditional microhybrid composite that has demonstrated excellent mechanical properties in multiple laboratory studies and was used as a control in this study to compare various new restorative materials.¹³ Esthet-X HD is a nanohybrid composite that is

Table 1: Components of Composite Materials Used in the Study								
Composite	Type	Manufacturer	Lot No.	Resin	Filler	Weight, %	Volume, %	Filler Size, μm
Esthet-X HD	Nanohybrid	Dentsply, York, PA, USA	1510141	Bis-GMA, Bis-EMA, TEGDMA	BAFSG, silica dioxide	77	60	0.02-2.5
Filtek Z250	Microhybrid	3M ESPE, St Paul, MN, USA	N830674	Bis-GMA, Bis-EMA, UDMA, TEGDMA	Zirconia, silica	78	60	0.01-3.5
NovoPro Fill	Nanofiber	Nanova, Columbia, MO, USA	UC033116A	Bis-EMA, UDMA, TEGDMA	BAFSG, amorphous fumed silica, titanium dioxide, hydroxyapatite	Not available	Not available	Not available
Abbreviations: BAFSG, barium aluminofluorosilicate glass; Bis-EMA, bisphenol A ethoxy dimethacrylate; Bis-GMA, bisphenol A diglycidyl methacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.								

marketed as having an ideal combination of polish and strength.¹⁴ See Table 1 for a description of the components of the tested materials.

Flexural Strength/Modulus

To prepare each specimen, a 2 mm × 2 mm × 25 mm stainless-steel mold (Sabri Dental Enterprises, Downers Grove, IL, USA) was placed on a plastic strip-covered glass slide. Each of the composite materials was inserted into the mold. The top surface of the mold was covered with a second plastic strip. The irradiance from the curing light was measured with a laser power meter (FieldMax II, Coherent Inc, Santa Clara, CA, USA) before each group preparation and found to be 1200 ± 10 mW/cm². One side of the specimen was then exposed to a light-polymerization unit (Bluephase G2, Ivoclar Vivadent, Amherst, NY, USA) for 20 seconds each in five separate overlapping increments. Next, the mold was turned, and the opposite side of the specimen was exposed to the light in a similar manner. Then the specimens were removed from the mold and stored in distilled water at 37°C in an incubator (Model 20 GC, Quincy Lab Corp, Chicago, IL, USA). After 24 hours, the specimens were placed on a three-point bending test device, which was constructed with a 20-mm span length between the supporting rods. A central load was applied using a universal testing machine (Model 5943, Instron, Norwood, MA, USA) at a crosshead speed of 0.25 mm/min. The flexural strength was calculated using the following equation:

$$\sigma_{FS} = \frac{3Fl}{2bd^2},$$

where *F* is the loading force at the fracture point, *l* is the length of the support span (20 mm), *b* is the

width, and *d* is the depth (thickness). Measurements were made using a new electronic digital caliper (GA182, Grobet Vigor, Carlstadt, NJ, USA) calibrated by the manufacturer to industry standards. Flexural modulus was determined from the slope of the linear region of the load-deflection curve using the analytical software (Instron). The mean flexural strength and modulus with the standard deviation were calculated for each of the materials.

Degree of Conversion—Cylindrical molds measuring 2.0 mm in height and 8.0 mm in diameter (Sabri Dental Enterprises) were placed on a plastic strip-covered glass slide on a standard white background. The composite was inserted into the mold. Another plastic strip was then placed on top, and a microscope glass slide was used to flatten the top surface. The glass slide was removed. The light-curing unit was positioned with a clamp so that it was flush with the top surface of the plastic strip-covered composite. The composites were light-cured for 20 seconds. The irradiance from the curing light was measured with a laser power meter, as before.

Following light-curing, specimens were stored in the dark at 37°C in 100% humidity for 24 hours in the incubator. Measurements were made at the top and bottom surface of each sample. DC was determined using Fourier Transform Infrared Spectroscopy (FTIR: Spotlight 400 FTIR Imaging System, PerkinElmer, Bacon, UK). To measure top post-conversion DC, the top surface of each specimen was applied to the attenuated total reflectance (ATR) attachment of the FTIR. The specimen was then turned over, reapplied to the ATR, and conversion was measured for the bottom surface. To measure the DC of uncured composite, the cylindrical mold was placed directly over the ATR. The composite was inserted into the mold as before but was not light-cured.

To determine DC, the spectra were taken under the following conditions: 4000-600 cm^{-1} wave numbers range and 16 scans per spectrum at a 4 cm^{-1} spectral resolution. The stretching vibrations of the aliphatic C=C bonds (1635 cm^{-1}) and the aromatic C=C bonds (1608 cm^{-1}) were used as the analytical and internal-reference absorption bands, respectively.

The DC was calculated from the ratio of the peak heights of the analytical and reference absorption bands normalized by the ratio of the uncured monomers, as shown in the following equation:

$$\text{DC}\% = \left(1 - \frac{(P_1/P_2)_{\text{polymer}}}{(P_1/P_2)_{\text{monomer}}} \right) \times 100.$$

The P_1 and P_2 values represent the absorbance intensities of the aliphatic C = C_(1634 cm^{-1}) and aromatic C = C_(1608 cm^{-1}) bonds, respectively. The mean top and bottom DC and standard deviation were determined for each material. In addition, the percent bottom/maximum DC ratio was calculated by dividing the bottom surface DC by the maximum DC per material and multiplying by 100.

Depth of Cure—To determine depth of cure, the composites were tested using the scraping technique (ISO 4049).¹⁵ A 4-mm-diameter by 8-mm-long stainless-steel split mold (Sabri Dental Enterprises) was placed on a plastic strip-covered glass slide on a standard white background. The composite was injected into the mold, a plastic strip was placed, and the composite was condensed with a glass slide to displace excess resin. The glass slide was removed, and the composite was immediately polymerized with a curing light for the manufacturer's recommended curing time of 20 seconds. The irradiance from the curing light was measured with a laser power meter, as before. The uncured resin was then scraped with a plastic instrument starting from the deepest point on the underside of the mold until polymerized resin was reached. The length of the remaining polymerized material was measured with an electronic digital caliper and divided by two, according to the ISO standard. The specimens were visually inspected and discarded if any voids were noted. The mean depth of cure and standard deviation for each composite material were calculated.

Volumetric Polymerization Shrinkage—The composites were placed on a pedestal in a video-imaging device (AcuVol, Bisco, Schaumburg, IL, USA). Ten specimens of each composite per group were imaged from the side at a distance of 10 cm. The video

camera digitized and analyzed the images with the provided image-processing software. The specimens were cured for 20 seconds using the aforementioned light-curing unit. Polymerization shrinkage was recorded continuously for 10 minutes after the light initiation. The mean percent shrinkage and standard deviation were calculated for each of the restorative materials.

Scanning Electron Microscopy Imaging

The nanofiber-reinforced composite was imaged with a scanning electron microscope (SEM: Vega TC, Tescan, Warrendale, PA, USA). To prepare specimens for imaging, the composite was placed in the same stainless-steel mold used for flexural strength testing and polymerized as before. The specimens were removed from the mold, placed on a three-point bending test device, and fractured as before. The specimens were sonicated in deionized ultra-filtered water for 10 minutes and then placed in a vacuum desiccator. The specimens were sputter-coated for 60 seconds (108 Auto Sputter Coater, Cressington, Watford, UK) with gold and imaged on the fractured surface.

Statistical Analysis

Ten specimens (n=10) were created per group for flexural strength/modulus and volumetric polymerization shrinkage. A sample size of 10 specimens per group in three groups provided 80% power to detect a moderate effect size of 0.6, or approximately 1.2 standard deviation difference among means when testing with a one-way analysis of variance (ANOVA) at $\alpha = 0.05$ (NCSS PASS v11.0.8 2011). Five specimens (n=5) were created per group for DC and depth of cure. A sample size of five specimens per group in three groups provided 80% power to detect a moderate effect size of 0.75, or approximately 1.5 standard deviation difference among means when testing with a one-way ANOVA at $\alpha = 0.05$ (NCSS PASS v11.0.8 2011). Data were analyzed with a one-way ANOVA and Tukey post-hoc test to determine the effect of composite type (three levels) on each of the individual properties ($\alpha=0.05$) using statistical software (IBM SPSS, version 24, Chicago, IL, USA).

RESULTS

Significant differences were found between groups per property. The results are summarized in Table 2. Filtek Z250 had the greatest flexural strength (160.9±24.2 MPa), a value that was significantly greater ($p=0.004$) than that of Esthet-X HD (130.5±12.5 MPa) and NovaPro Fill (135.0±21.4

Table 2: Properties of the Composite Materials ^a			
Property	Properties, Mean (SD)		
	NovaPro Fill	Filtek Z250	Esthet-X HD
Flexural strength, MPa	135.0 (21.4) B	160.9 (24.2) A	130.5 (12.5) B
Flexural modulus, GPa	11.7 (0.6) B	15.5 (0.8) A	10.7 (0.2) C
Shrinkage, %	3.01 (0.08) B	2.17 (0.16) A	3.00 (0.16) B
Depth of cure, mm	3.52 (0.04) A	3.55 (0.12) A	2.74 (0.03) B
DC top, %	58.0 (2.5) A	50.6 (3.8) B	54.7 (3.3) AB
DC bottom, %	49.6 (3.1) A	45.4 (1.5) B	49.0 (1.5) A
DC bottom/maximum ratio, %	81.2 (4.4) A	80.1 (4.7) A	84.2 (2.5) A
Abbreviations: DC, degree of conversion; SD, standard deviation.			
^a Groups With the Same Letter per Row Are Not Significantly Different ($p>0.05$).			

MPa), which were not significantly different from each other ($p=0.90$). The flexural modulus of Filtek Z250 (15.5 ± 0.8 GPa) was significantly greater than that of NovaPro Fill (11.7 ± 0.6 GPa), which was significantly greater than that of Esthet-X HD (10.7 ± 0.2 GPa) ($p<0.001$). Filtek Z250 had significantly less shrinkage ($2.17\%\pm0.16\%$; $p<0.001$) than did Esthet-X HD ($3.00\%\pm0.16\%$) and NovaPro Fill ($3.01\%\pm0.08\%$), which were not significantly different from each other ($p=0.99$). Filtek Z250 exhibited the greatest depth of cure (3.55 ± 0.12 mm), but it was not significantly different from that of NovaPro Fill (3.52 ± 0.04 mm; $p=0.83$), and both had significantly greater ($p<0.001$) depth of cure than did Esthet-X HD (2.74 ± 0.03 mm). NovaPro Fill had the greatest DC on the top surface ($58.0\%\pm2.5\%$), which was significantly greater than that of Filtek Z250 ($50.6\%\pm3.8\%$; $p=0.005$). The DC of the top surface of Esthet-X HD ($54.7\%\pm3.3\%$) was not significantly different than that of NovaPro Fill or Filtek Z250 ($p=0.22$). On the bottom surface, NovaPro Fill ($49.6\%\pm3.1\%$) had the greatest DC, but it was not significantly different ($p=0.95$) from that of Esthet-X HD ($49.0\%\pm1.5\%$). Both were significantly greater than that of Filtek Z250 ($45.4\%\pm1.5\%$; $p=0.008$). However, the DC bottom/maximum ratio was not significantly different ($p=0.27$) among the three groups: Esthet-X HD ($84.2\%\pm2.5\%$), NovaPro Fill ($81.2\%\pm4.4\%$), and Filtek Z250 ($80.1\%\pm4.7\%$).

The SEM images of the nanofiber-reinforced composite were reviewed. As shown in Figures 1 and 2, the nanofibers in NovaPro Fill were found to be bundled and not well organized or aligned.

In summary, compared to Filtek Z250, NovaPro Fill had significantly lower flexural strength and modulus, greater volumetric shrinkage, similar depth of cure, but greater top and bottom DC. Compared to Esthet-X HD, NovaPro Fill had similar flexural strength, shrinkage, and top and bottom

DC, but significantly greater depth of cure and flexural modulus.

DISCUSSION

There are limited published studies in the literature regarding nanofiber-reinforced restorative composites. This is the first study to examine a commercially available restorative composite (in the United States) reinforced with hydroxyapatite nanofibers. Based on the results of this study, the null hypothesis was rejected. Significant differences were found between groups based on property ($p<0.05$). The manufacturers of NovaPro Fill claim that it has superior mechanical properties to other products available on the market. However, in this study, the flexural strength of NovaPro Fill was not significantly greater than that of the other two composites tested.

Despite the significant improvement of restorative composites, these materials still suffer from two key shortcomings: deficiency in mechanical strength and polymerization shrinkage. These shortcomings may contribute to a shorter survival time of composites when compared to amalgams.² The vast majority of restorative composites consist of ceramic-based particles surrounded by a light-curable methacrylate matrix. Monomer solutions typically used are blends of bisphenol A diglycidyl ether dimethacrylate (Bis-GMA), triethylene glycol dimethacrylate (TEGDMA), and urethane dimethacrylate (UDMA).⁸

Recently there has been an increasing interest to reinforced composites with nanofibers. The addition of electrospun nanofibers has been examined utilizing different materials, from nylon and polyacrylonitrile–poly(methyl) methacrylate to hydroxyapatite.¹⁶ In initial laboratory studies, researchers were observing a decrease in flexural properties

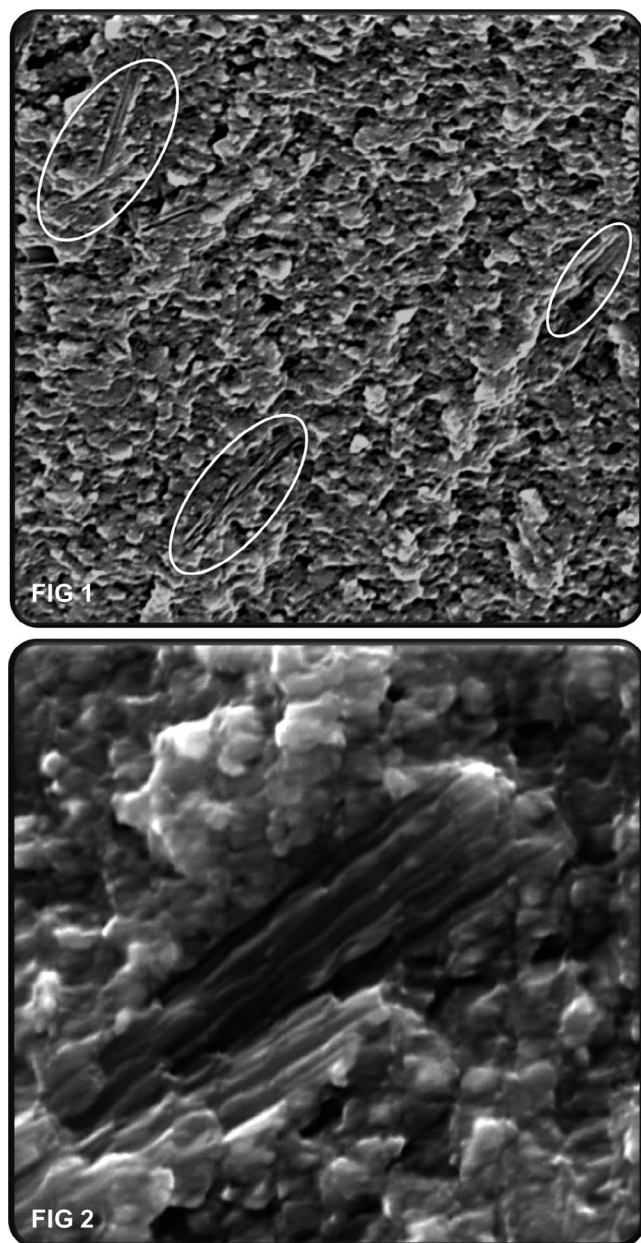


Figure 1. Scanning electron microscope (SEM) image of the nanofibers in the NovaPro Fill composite at 1000 \times magnification. Figure 2. Scanning electron microscope (SEM) image of a bundle of nanofibers in NovaPro Fill composite at 2100 \times magnification.

with certain fiber-to-polymer ratios and resin concentrations. The decrease was due to the limitations of bonding between the fibers and the resin matrix or to the incomplete infiltration of the resin to wet the nanofibers, which resulted in voids that compromised the overall material strength.¹⁷ Many of the studies of nanofiber-reinforced composite focus on the orientation and distribution of the fibers. Of these studies, several have found that when uniform distribution of the fibers is achieved

within the resin, there is a reported increase in toughness of the composite.¹⁸ Vidotti and others⁸ evaluated the influence of different concentrations and mass ratios of nanofiber composites. They found that different ratios of fibers did not affect the flexural strength and modulus of the composites, but the direction of the fibers did affect the tensile properties. However, Fong¹⁹ reported that the addition of 5 wt% relative low-strength polymer (Nylon 6) nanofibers could lead to a 36% increase in flexural strength and a 26% increase in flexural modulus. Recently, Uyar and others²⁰ demonstrated that the mechanical properties of dental composites are improved when using aligned nanofibers. They also found improvements in nonaligned nanofibers; however, the improvement was not statistically significant.²⁰ Nonalignment of nanofibers could have contributed to the strength properties recorded in this study for NovaPro Fill. As shown in the SEM image in Figure 1, the nanofibers in NovaPro Fill were not organized or aligned.

According to Chen and others,¹⁰ the impregnation of the hydroxyapatite nanofibers into Bis-GMA/TEGDMA dental resins may lead to double-sided effects—reinforcement due to a uniform distribution of hydroxyapatite nanofibers, but undermining if the nanofibers amass during processing to form bundles. Vidotti and others⁸ showed that as the hydroxyapatite nanofiber mass fraction increased to 10 wt%, a small portion of the hydroxyapatite nanofibers started to form bundles. When the hydroxyapatite nanofiber mass fraction increased to 20 wt%, more bundles were formed in the dental resin matrix, creating mechanical weak points that led to lower flexural strength values.⁸ A weakening effect due to bundling of nanofiber may have occurred in this study as well. A nanofiber bundle in the NovaPro Fill is shown in a SEM image (see Figure 2). Bundles of the nanofibers in a composite may also decrease the translucency of composites.¹⁰ Therefore, the DC of composites may decrease with a reduction in polymerization, a secondary phenomenon to an increased curing-light attenuation. A reduction in DC may lead to lower mechanical properties.

Nanova⁷ reports that NovaPro Fill has greater DC than do other composite restorative materials. The DCs of both the top and bottom surfaces of NovaPro Fill were significantly greater than those of Filtek Z250, but not significantly different from those of Esthet-X HD. However, when the bottom/maximum DC ratios were calculated, no significant difference was found among all three composites. When

evaluating depth of cure, NovaPro Fill performed similarly to Filtek Z250, with no significant difference between the two composites. Alternatively, both Filtek Z250 and NovaPro Fill had significantly greater depth of cure than did Esthet-X HD.

Finally, this study examined the volumetric shrinkage of the three composites. Polymerization shrinkage has been a restorative challenge for resin-based composites. Residual stress can be generated from shrinkage during curing.¹⁰ A study by Moszner and Salz²¹ showed the polymerization shrinkage of Bis-GMA to be 6.1% and that of TEGDMA to be 14.3%. Commercial composites have included inorganic filler to minimize the total shrinkage rate and to improve marginal adaptation. In 2008, Anttila and others²² showed that fibers could reduce polymerization shrinkage. In this study, NovaPro Fill performed comparably to Esthet-X HD, with no significant difference between the two composites. However, both Esthet-X HD and NovaPro Fill had significantly greater polymerization shrinkage than did Filtek Z250.

CONCLUSIONS

NovaPro Fill had similar flexural strength, shrinkage, and top and bottom DC, but significantly greater depth of cure and flexural modulus when compared to Esthet-X HD. When compared to Filtek Z250, NovaPro Fill had significantly lower flexural strength and modulus, greater volumetric shrinkage, and similar depth of cure, but greater top and bottom DC. Based on the properties tested, there may not be any significant advantage to the use of the new nanofiber composite restorative material (NovaPro Fill) when compared to the use of traditional hybrid composites.

Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

Disclaimer

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