Original Article

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Comparison of Microhardness and Surface Roughness of New Nanofiber Filled Flowable Composite

Yeni Nanofiber Dolduruculu Akışkan Kompozitin Mikrosertlik ve Yüzey Pürüzlülüğünün Karşılaştırılması

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ABSTRACT

Objective: This study aimed to compare the microhardness and surface roughness (Ra) of a resin composite, which was recently introduced to be the first flowable composite with the nano-sized fiber filler, with the particle filled composite resins with different properties, used in Class V cavities.

Methods: Totally 100 disc-shaped samples (diameter: 4 mm, height: 2 mm) were prepared and divided into five groups in accordance to the different types of composites (n=20): 1) Flowable composite with nano-fiber filler (Group N: NovaPro Flow, Nanova, USA); 2) Flowable bulk-fill composite [Group Estelite Bulk Fill Flow (EBF): Tokuyama, Japan]; 3) Flowable composite [Group G-aenial Universal Flo (GUF): GC Corp, Japan]; 4) Highly-filled flowable composite [Group G-aenial Universal Injectable (GUI): GC Corp, Japan]; 5) Micro-hybrid composite (Group Z250: Filtek Z250, 3M ESPE, USA). They were polished with aluminum oxide polishing discs. Ra measurements (μm) were made using contact profilometer (MarSurf M 300 C; Mahr GmbH, Germany) (n=10). Vickers microhardness evaluations were made using HMV microhardness tester (Shimadzu, Japan) (n=10). Three dimensional (3D) optic profilometer was used to evaluate the surface topography. One-way ANOVA, Shapiro-Wilk and Tukey tests were used for statistical analysis ($p<0.05$).

Results: At top and bottom surfaces, Group N showed significantly lowest microhardness values while Group Z250 showed significantly highest microhardness values than other groups ($p<0.05$). Group GUI showed significantly higher microhardness values than group

ÖZ

Amaç: Bu çalışmada, nano boyutlu fiber dolduruculu ilk akışkan kompozit olarak yakın zamanda piyasaya sürülen bir rezin kompozitinin mikrosertlik ve yüzey pürüzlülüğünün (Ra), Sınıf V kavitelerde kullanılan farklı özelliklere sahip partikül dolduruculu kompozit rezinlerle karşılaştırılması amaçlandı.

Yöntemler: Yüz adet disk şeklinde numune (çap: 4 mm, yükseklik: 2 mm) hazırlanarak farklı kompozit türlerine göre beş gruba ayrıldı (n=20): 1) Nano fiber dolduruculu akışkan kompozit (Grup N: NovaPro Flow, Nanova, ABD); 2) Akışkan bulk fill kompozit [Grup Estelite Bulk Fill Flow (EBF): Tokuyama, Japonya]; 3) Akışkan kompozit [Grup G-aenial Universal Flo (GUF): GC Corp, Japonya]; 4) Yüksek oranda doldurucu içeren akışkan kompozit [Grup G-aenial Universal Injectable (GUI): GC Corp, Japonya]; 5) Mikro hibrit kompozit (Grup Z250: Filtek Z250, 3M ESPE, ABD). Alüminyum oksit polisaj diskleri ile parlatıldı. Ra ölçümleri (μm) kontak profilometre (MarSurf M300C; Mahr GmbH, Almanya) (n=10) kullanılarak yapıldı. Vickers mikrosertlik değerlendirmeleri ise HMV microhardness tester (Shimadzu, Japonya) (n=10) kullanılarak yapıldı. Yüzey topografyasını değerlendirmek için üç boyutlu (3D) optik profilometre kullanıldı. İstatistiksel analiz için one-way ANOVA, Shapiro-Wilk ve Tukey testleri kullanıldı ($p<0,05$).

Bulgular: Üst ve alt yüzeylerde Grup N anlamlı olarak en düşük mikrosertlik değerlerini gösterirken, Grup Z250 diğer gruplara göre anlamlı olarak en yüksek mikrosertlik değerlerini gösterdi ($p<0,05$). Grup GUI, grup EBF ve GUF'ye göre anlamlı derecede

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ABSTRACT

EBF and GUF ($p<0.05$). Hardness ratio was found lower than 80% in Group N. No significant differences in Ra were found between the groups. 3D optic profilometer revealed that similar scratch appearances were detected in all groups.

Conclusion: Incorporation of flowable composite with nanofiber filler may not be advantageous for micro-hardness, hardness ratio and Ra properties.

Keywords: Class V restoration, flowable composite, nanofiber, microhardness, surface roughness

ÖZ

yüksek mikrosertlik değerleri gösterdi ($p<0,05$). Grup N'de sertlik oranı %80'in altında bulundu. Tüm gruplarda Ra anlamlı bir farklılık bulunmadı. 3D optik profilometrede tüm gruplarda benzer pürüzlü görünümler tespit edildi.

Sonuç: Akışkan kompozitin nanofiber doldurucu maddesi ile birleştirilmesi mikro sertlik, sertlik oranı ve Ra özellikleri açısından avantajlı olmayabilir.

Anahtar Sözcükler: Sınıf 5 restorasyon, akışkan kompozit, nanofiber, mikrosertlik, yüzey sertliği

Introduction

Direct resin composites have gained popularity as the preferred material due to their ability to restore tooth structure, while preserving the natural look of teeth with shape, color, and texture (1). Also, these restorative materials are frequently used for Class V cavities which may be caused by caries, erosion, abfraction and abrasion. However, enhancing restorative materials for Class V cavities presents challenges as they have distinct biomechanical demands compared to occlusal cavities. The stress caused by occlusal forces in Class V lesions can lead to restorative material fractures and debonding (2). To withstand the forces exerted during chewing, enhancing the mechanical properties of restorative materials has been achieved through modifications in filler particle size and morphology. These modifications include incorporating ceramic particles with random orientation, whiskers in single or multi-layer form, or fibers in continuous or discontinuous arrangements in various orientations. These adjustments have led to improved mechanical properties of the materials (3-5). For reinforcement, the fibers can enhance mechanical properties by acting mainly as crack stoppers and mimic the tooth structures (3,6). Microstructural parameters - such as fiber diameter, fiber length, fiber orientation, fiber loading, and the adhesion between fibers and the polymer matrix - play a crucial role in determining the characteristics of fiber-reinforced composite resins. These factors greatly influence the performance and properties of the composite material (7). Inorganic hydroxyapatite (HAp) nanofibers are used as one of the methods to reinforce dental resin composites (8). HAp nanofibers are calcium phosphate fillers that have been shown to offer more stress transfer by interaction between nanofibers and matrix and reduced polymerization shrinkage so improving the marginal integrity. These aesthetic materials that utilize glass fibers are used in various dental clinical procedures with a primary focus on restorative dentistry (9). Although novel restorative materials are launched in the markets, there is no gold standard procedure on the choice of best materials for Class V restorations.

Attaining a smooth and durable surface is of great clinical significance for composite resin restorations as it directly impacts the long-term survival (10). The filler particles of composite resin materials can influence the material's ability to be polished and its hardness, consequently affecting the surface roughness (Ra) and hardness of the restoration. To our knowledge, limited data are present for evalauting the microhardness and Ra of a resin composite, which was recently introduced to be the first flowable composite with the nano-sized fiber filler. Thus, the aim of this study was to compare the microhardness and Ra of new nanofiber reinforced flowable composite with flowable bulk-fill, highly-filled flowable composite, flowable composite and microhybrid composite resins used in Class V cavities.

The null hypothesis of this *in vitro* study was:

There would be no differences in microhardness and Ra of new nano-fiber reinforced flowable with other composite materials used in Class V cavities.

Methods

Sample Size Calculation

A power analysis was performed to establish the specimen size according to the literature (11). In this study, for each group, minimum 10 specimens were required to gain a medium effect size ($d=0.50$), with 95% power and a 5% type 1 error rate.

Sample Preparation

A list of the composite resins used in this study is given in Table 1. The colors of composites were selected as A2. Five different composite resins were used: Group N: NovaPro Flow (Nanova, USA); Group Estelite Bulk Fill (EBF): (Tokuyama, Japan); Group G-aenial Universal Flo (GUF): (GC Corp., Japan); Group G-aenial Universal Injectable (GUI): (GC Corp., Japan) and Group Z250: Filtek Z250 (3M ESPE, USA). Hundred disc-shaped samples, for each tested materials (n=20) (height: 2 mm and diameter: 4 mm) were prepared using teflon molds. To achieve a smooth, polymerized surface, the samples were sandwiched between two transparent polyester matrix strips (Mylar Strip, SS White Co., Philadelphia, PA, USA) and glass slides. The excess material was then removed by applying pressure using the glass slides. Then, the samples were polymerized with a light-emitting diode light-curing unit for 20 s according to the manufacturer's guidelines (light emitting diode, light curing unit) (Valo, Ultradent, South Jordan, UT, USA) (irradiance of 1000 mW/cm^2). The top surfaces of the samples underwent polishing using a sequence of aluminum oxide polishing discs. (Sof-Lex XT, Pop-On, 3M ESPE, USA) with a slow hand-piece.

They were stored in distilled water at 37 °C for 24 h in a dark vial.

All restorative procedures were done by a single operator (Z.C.O.) in accordance to the manufacturers' instructions.

Microhardness Measurement and Calculating Bottom/Top Hardness Ratio

Ten disc-shaped samples of each composite resin were used (n=10) and Vickers microhardness test was peformed with HMV microhardness tester (HMV-G, Shimadzu Corp., Japan) according to the ASTM E384-17 standard (12). Three measurements were obtained on the top (upper) and bottom (lower) surfaces of each specimen (200 g load and 10 s dwell time). Vickers Hardness values of each surface was recorded as the average of these measurements. The hardness number of the bottom surface was divided by the hardness number of the top surface to establish the hardness ratios (%), which were subsequently converted to a percentage. A second operator, who was unaware of the type of composite resin, performed all of the microhardness measurements (R.H.E.O.).

Surface Roughness Measurement

Ten disc-shaped samples of each composite resin were used (n=10) and Ra test was perfomed with a contact profilometer (MarSurf M 300 C; Mahr GmbH, Göttingen, Germany) in accordance with EN ISO 4288 (stylus tip Radius: 5 µm, a stylus driving speed: 0.5 mm/s, traversing length (Lt): 1.75 mm and five cut-off lengths: 0.250 mm) (13). Three measurements were performed in 4 different locations (in each quadrant in a clockwise direction) of the polished surface and arithmetic mean of the measurements (µm, Ra) were recorded. A second operator, who was unaware of the type of composite resin, performed all of the Ra measurements (B.O.).

Statistical Analysis

A software program (SPSS 22.0 Windows, SPSS Inc., IL, USA) was used for statistical analysis. Shapiro-Wilk test, and Levene's test were to determine the normality of variables and homogeneity of variances for microhardness and Ra data. Since the data were normally distributed, one-way analyses of variance (one-way ANOVA) were used to compare the the materials. All pairwise comparisons were performed with the Tukey HSD test at a significant level of 0.05.

Surface Topography Analysis

One specimen from each group was subjected to surface pretreatment to evaluate the three dimensional (3D) surface topography with an optic profilometer (Nanomap 1000WLI,

Table 1. The composite resins used in this study

Material	Filler type	Organic marix
Filtek Z250 (Z250) 3M ESPE, St Paul, MN, USA	Zirconia, silica 78 wt %, 60 vol % 0.01 µm to 3.5 µm with an average particle size of 0.6 µm	Bis-GMA, Bis-EMA, UDMA, TEGDMA
NovaPro Flow Nanova Inc, Missouri, USA	Barium borosilicate glass (0.7 µm), hydrophobic amorphous silica (40 nm), hydroxyapatite fibers. 60% wt	Bis-EMA, TEGDMA, UDMA
Estelite Bulk Fill Flow Tokuyama Dental Corp, Ibaraki, Japan	Spherical Silica-zirconia (200 nm) 70 wt %, 56 vol %	Bis-MPEPP, TEGDMA, Bis-GMA
G-aenial Universal Flo GC Corp., Tokyo, Japan	SO_2 (16 nm), Strontium glass (200 nm) 69 wt %, 50% vol	UDMA (15-20 wt %), TEGDMA (5-10 wt %), Bis-MEPP (5-10 wt %)
G-aenial Universal Injectable GC Corp, Tokyo, Japan	150 nm Barium glass, silica 69 wt %	Dimethacrylate monomers

BIS-GMA: Bisphenol A glycidyl methacrylate, UDMA: Urethane dimethacrylate, TEGDMA: Triethylene glycol dimethacrylate, Bis-MPEPP: Bis-methacryloxyethoxy phenyl propane, Bis-EMA: Bisphenol A diglycidyl methacrylate ethoxylated, µm: Micrometer, wt %: Weight percentage, vol %: Volume percentage, nm: Nanometer

Table 2. Mean microhardness values, hardness ratio and standard deviations (± SD) for all groups

Groups	Top	Bottom	Hardness ratio (%)
Group N	40.603±3.378^{A}	20.633±3.183^{A}	50.9±7.4
Group EBF	54.454±1.748^{B}	50.917±2.442^{B}	93.6±4.6
Group GUF	51.389±3.19^{B}	40.946±5.55^{B}	79.6±9.4
Group GUI	65.168±4.222^{C}	57.671±5.662^{C}	88.5±6.2
Group Z250	113.023±8.416^{D}	95.834±5.873^{D}	85.0±5.4
p-value	<0.001	<0.001	

*Different capital letters show the significant difference between the groups (p<0.05). N: NovaPro Flow, EBF: Estelite Bulk Fill Flow, GUF: G-aenial Universal Flo, GUI: G-aenial Universal Injectable, Z250: Filtek Z250, SD: Standard deviation

AEP Technology, Saratoga, CA, USA). The scan range was adjusted to 232 mm, the vertical dynamic range was adjusted to 500 mm and the stylus loading force was set to 12 mg. A color scale and graphics were used for interpretation of the images. Different values are represented with different colors. The negative values indicate the pits while the positive values resemble the peaks.

Results

Microhardness Measurement and Calculating Bottom/Top Hardness Ratio

Mean microhardness values, hardness ratio and standard deviations of all groups are presented in Table 2. At top and bottom surfaces, Group N showed significantly lowest microhardness values while Group Z250 showed significantly highest microhardness values than other groups ($p<0.05$). Group GUI showed significantly higher microhardness values than group EBF and GUF ($p<0.05$). However, no significant differences were determined in microhardness values between Group EBF and Group GUF ($p>0.05$).

Group Z250, Group GUI, Group GUF and Group EBF showed hardness ratio equal or exceeding the 80% threshold values although Group N exhibited lower hardness ratio (50.9%) than theshold values (80%).

Surface Roughness Measurement

Mean Ra values and standard deviations of all groups are shown in Table 3. There were no signicant differences in Ra between the groups ($p>0.05$).

Surface Topography Analysis

In all groups, similar micro-scratches and irregularities were detected (Figure 1).

Discussion

In this study, the microhardness and Ra of new nanofiber reinforced flowable composite with flowable bulk-fill, highly-filled flowable composite, flowable composite and microhybrid composite used in Class V cavities, were compared. Based on the results, the null hypothesis that there would be no differences in microhardness and Ra of new nanofiber reinforced flowable with other composite materials used in Class V cavities, was partially rejected since the differences in microhardness values of the composite resins used were significant while no significant differences in Ra were found among all composite resins.

Vickers hardness is a method used to determine the hardness value by measuring the depth or area of an indentation left by an indenter with a specific shape, force, and time of application. The hardness value is indicative of a material's ability to withstand applied loads (14). It represents a combination of deformation and elastic behavior. Several factors related to resin composites can influence hardness, including the size, shape, and fraction of fillers in the inorganic phase. Generally, hardness increases with a higher amount of fillers (15). This can be explained by the fact that as the volume fraction of fillers increases, a point is reached where particles come into contact with each other within the matrix. At this point, stress is transferred primarily through the interactions between hard particles (16,17). In this study, NovaPro Flow exhibited significantly the lowest microhardness values while Filtek Z250 showed significantly the highest microhardness values at the top and bottom surfaces. This finding could be attributed

Table 3. Mean surface roughness values and standard deviations (± SD) of all groups (µm, Ra)

Groups	Surface roughness (± SD)
Group N	0.198±0.06
Group EBF	0.161±0.053
Group GUF	0.126±0.044
Group GUI	0.145±0.053
Group Z250	0.166±0.055
p-value	0.051

N: NovaPro Flow, EBF: Estelite Bulk Fill Flow, GUF: G-aenial Universal Flo, GUI: G-aenial Universal Injectable, Z250: Filtek Z250, SD: Standard deviation

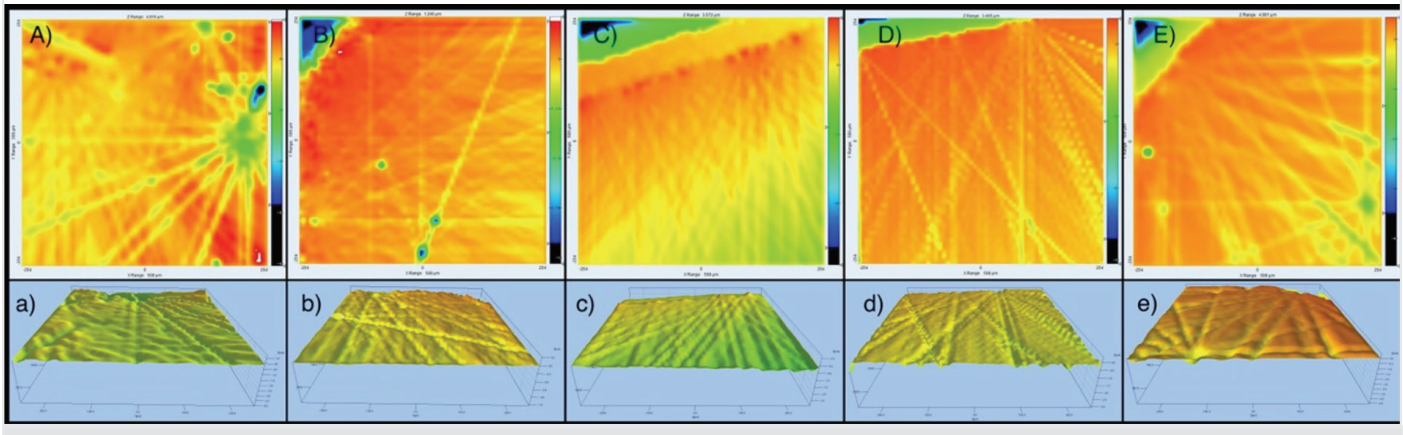

Figure 1. Optic profilometer images showing the 2D (A-E) and 3D (a-e) surface topography of all composite resins. A, a: NovaPro Flow, B, b: Estelite Bulk Fill Flow, C, c: G-aenial Universal Flo, D, d: G-aenial Universal Injectable, E, e: Filtek Z250, 2D:Two dimensional, 3D: Three dimensional

to the differences in inorganic filler amount and filler types of the composite resins used in this study. NovaPro Flow is a low-viscosity, visible-light cured, radiopaque, nanohybrid composite that contains 60% by weight HAp nanofiber filler (lowest filler load) while Filtek Z250 is a high-viscosity, microhybrid composite that contains 78% by weight silica-zirconia fillers (highest filler load). Besides, this is in line with McCabe and Wassell (18), who reported that microhardness of composite materials enhanced with increasing filler content. However, in this study, when G-aenial Universal Injectable, G-aenial Universal Flow and EBF, which have similar filler amount, were compared, it was determined that G-aenial Universal Injectible showed higher microhardness values than the other two composites at the top and bottom surfaces. It was indicated that composite resins with small filler particles increased surface microhardness (19). So, this finding could be explained by the fact that G-aenial Universal Injectable (150 nm) had smaller filler particles than the G-aenial Universal Flow (200 nm) and EBF (200 nm).

As light passes through the composite resins, light intensity is clearly reduced due to light absorbtion and attenuation (20). Hardness values of bottom/top surfaces generally can be used to measure the degree of polymerization (21). Direct methods, such as infrared and Raman spectroscopy, are not commonly employed in routine procedures due to their complex, expensive, and time-consuming nature (22). Thus, in this study, the Vickers microhardness measurement was preferred to determine the restorative material's degree of polymerization, in view of its ease of use, popularity and relative efficiency (23). Degree of polymerization is influenced by many factors, such as the chemical structure of the monomers, filler composition, curing time and light intensity (21). In the literature, it is indicated that an acceptable degree of polymerization is achieved if the bottom hardness corresponds to at least 80% of the top surface hardness (24). However, in this study, hardness ratio lower than 80% was found in only NovaPro Flow. This finding could be attributed to its lower translucency.

The esthetics of a restorative material may be compromised due to its Ra, leading to negative impacts on abrasion and wear resistance, plaque buildup, and the potential development of secondary caries (25). Especially in Class V restorations, plaque accumulation is very important in terms of gingival health in this region (26).

Various polishing systems are such as polishing discs, rubber wheels, cups, discs, and pastes, that can be used to finish and polish composite resin restorations (27). To ensure consistency and eliminate any potential variations caused by different polishing systems, the same polishing procedure was employed for all materials in the current study, despite individual manufacturers typically recommending specific polishing systems for each material evaluated. Previous studies indicated that smoothest surfaces were obtained with multistep aluminum oxide polishers (27,28). Thus, in this study, the multistep polishers with higher flexibility (Sof Lex) were preferred. In the literature, it was reported that 2D Ra above 0.2 μm resulted in an increase of plaque accumulation and higher risk for caries and periodontal inflammation (29). It was found that the majority of patients were capable of discerning differences of approximately 0.3 μm in terms of mean roughness (30). In this study, all composite resins exhibited lower Ra values than 0.2 μm.

Profilometers are commonly utilized to obtain roughness values, providing a quantitative assessment of surface irregularities. Mechanical profilometry, a widely used method for evaluating surface properties, offers a 2D representation of the surface, yielding limited information (31). On the other hand, optical profilometry, which is also employed to measure Ra after polishing composites (32), captures the 3D surface topography, thereby reflecting the natural characteristics of the surface (33). By utilizing 3D measurements, optical profilometry offers a more comprehensive and detailed description of surface topography compared to 2D measurements, providing a more complete understanding of the surface (31). In this study, the Ra of the composite resins was measured with a mechanical profilometer, then their surface topograpy was evalauted with an optical profilometer. NovaPro Flow offers optimal handling and finishing properties that does not require any special polishing tools that enable the dentist to achieve the desired finish and esthetics expected from a flowable composite. In this study, NovaPro Flow showed similar Ra values when compared to the other composite resins. Besides, no significant differences in Ra values were observed for other tested composites. The results of 3D optic profilometer images revealed that similar scratch appearances were detected in all groups, were consistent of the Ra values.

Study Limitations

In this study, multistep polishers were used and oral conditions such as bacteria, saliva or pH and temparatures changes were not considered. Another limitation of this study was that it was not accomplished using spectral analysis. Thus, further studies should focus on the effect of different polishing systems and clinical conditions on the surface properties of this new restorative material, additionally using different test techniques such as spectral analysis.

Conclusion

Despite the limitations of this study, it can be concluded that: At the top and bottom surfaces, new nanofiber reinforced flowable composite showed significantly lowest microhardness while microhybrid composite showed highest microhardness than other composites. At the top and bottom surfaces, highly-filled flowable composite showed significantly higher microhardness than flowable bulk-fill and flowable composite resin. Hardness ratio of a new nanofiber reinforced flowable composite was found lower than 80% threshold value that was the acceptable degree of polymerization. Similar Ra and surface topography were obtained for all composite resins.

Ethics

Ethics Committee Approval: Ethics committee approval is not required.

Informed Consent: Informed consent is not required.

Footnotes

Authorship Contributions

Concept: Z.C.O., Design: R.H.E.O., Z.C.O., Data Collection or Processing: R.H.E.O., Z.C.O., B.O.O., Analysis or Interpretation: B.O.O., E.E.D., Literature Search: R.H.E.O., B.O.O., Writing: R.H.E.O., B.O.O.

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