Original Article

New Experimental Zirconia-Reinforced Rice Husk Nanohybrid Composite and the Outcome of Its Surface Roughness and Microhardness in Comparison with Commercialized Nanofilled and Microhybrid Composite Resins

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Abstract

Background: An ideal composite resin should demonstrate smooth surface after polishing and high hardness value to provide long-term success. Thus, this study aimed to compare the surface roughness and microhardness of new experimental zirconia-reinforced rice husk nanohybrid composite (Zr-Hybrid) with commercialized nanofilled (Filtek-Z350-XT) and microhybrid composite (Zmack-Comp) resins before and after artificial ageing. **Methods:** One hundred and eighty standardized disc samples were prepared, of which ninety samples each were used for surface roughness and microhardness test, respectively. They were divided equally into: Group 1 (Filtek-Z350-XT), Group 2 (Zmack-Comp), and Group 3 (Zr-Hybrid). For surface roughness test, all samples were polished with aluminium oxide discs and further subdivided into aged and unaged subgroups, in which composite samples in aged subgroups were subjected to 2500 thermal cycles. Next, all the samples were subjected to surface roughness test using a contact stylus profilometer. As for microhardness test, all the aged and unaged samples were tested using a Vickers hardness machine with a load of 300 kgf for 10 s and viewed under a digital microscope to obtain microhardness value. Data were analyzed using two-way ANOVA followed by *post hoc* Tukey's honestly significant difference and paired sample t-test with significance level set at $P = 0.05$. **Results:** In both the aged and unaged groups, Zr-Hybrid showed statistically significantly lower surface roughness ($P < 0.05$) than Filtek-Z350-XT and Zmack-Comp, but no statistically significant difference was noted between Filtek-Z350-XT and Zmack-Comp ($P > 0.05$). A similar pattern was noted in microhardness test, whereby Zr-Hybrid showed the highest value ($P < 0.05$) followed by Filtek-Z350-XT and lastly Zmack-Comp. Besides, significant differences in surface roughness and microhardness were noted between the aged and unaged groups. **Conclusion:** Zr-Hybrid seems to demonstrate better surface roughness and microhardness value before and after artificial ageing.

Keywords: *Composite resin, microhardness, rice husk, surface roughness, zirconia*

Introduction

Composite resins are undeniably one of the most used restorative materials in current dental practice. With the increased esthetic demands and advancement of nanotechnology, nanocomposites have been introduced in which the filler particle size has been progressively decreased to enhance their physical and mechanical properties.[1,2] Moreover, nanocomposites also improve the esthetic features of the restoration by improving their polish capacity and durability.[2,3] Recently, bio-composite has gained attention by incorporating natural products into composite resins. Rice husk as silica in dental composite was first introduced by Noushad *et al.* and has shown to possess several advantages such as being environmental friendly, cost-effective, and of comparable strength with commercialized composite resins.[4] This type of nanohybrid composite was found to have nanosized spherical silica particles widely dispersed in the resin matrix.[4] Adopting an innovative approach, the present study incorporated 10% w/w of zirconia nano-powder into rice husk composite based on the fact that addition of zirconia increases the physical properties and fracture toughness of the material.[5] Hence, it can be anticipated that this new nanohybrid composite would demonstrate better physical properties.

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Finishing and polishing of composite resin restoration are essential steps and routinely used in daily dental practice to enhance both esthetics and longevity of the composite restorations.[6] A rough surface of composite may result in plaque accumulation, gingival inflammation, surface staining, and increased risk of secondary caries.[6-8] Composite resin is a heterogeneous material composed of resin matrix, filler particle, coupling agent, and photo-initiator. Thus, they do not abrade to the same degree due to the different hardness value among its composition.[9] Because of this, composite resins exhibit various degrees of surface roughness after polishing.[10] Besides, various surface defects may appear, such as irregularities and microcracks, as a result of removing some surface particles during finishing and polishing procedures.[9] Microhardness is also another important parameter because composite restorations may suffer from reduced surface microhardness with time, thus increasing their susceptibility to wear.[9] The measurement for microhardness of composite resins using Vickers and Knoop hardness tests is considered an indirect method to evaluate the physical and mechanical strength.[9]

Different types of composite resins do not achieve a comparable surface smoothness and microhardness when they are subjected to the same procedural techniques. Therefore, as part of this bigger project, the present study aimed to investigate and compare the surface roughness and microhardness before and after artificial ageing of a new experimental zirconia-reinforced rice husk nanohybrid composite resin with commercially available nanofilled and microhybrid composite resins. The null hypotheses were as follows: first, all composite resins used in this study show similar surface roughness and microhardness values. Second, no significant difference will be found in terms of surface roughness and microhardness before and after artificial ageing.

Methods

Preparation of zirconia-reinforced nanohybrid composite

The composite filler-to-monomer ratio was 75:25. 25% w/w of the resin monomer was composed of 49% w/w bis-phenol A-glycidyl methacrylate (bis-GMA), 49% w/w of tri-ethylene-glycol-dimethacrylate, and 2% w/w of camphorquinone. They were mixed in a mixing bowl placed on a vortex mixer (Fisherbrand™ Analog Vortex Mixer, Fisher Scientific International, Inc., Hampton, US). Aluminium foil was used to cover the bowl to prevent the resin from prepolymerization by ambient light. The resin mixture was stored in a refrigerator for 48 h to allow the resin monomer to be stabilized. After 48 h, the prepared matrix mixture was added to 65% w/w of nano silica from the rice husk obtained from the previous study[4] and 10% w/w pure zirconia nano powder (MFCD00011310, American Elements, Los Angeles, US). The bowl was placed on the mixing vortex to form a homogenous composite material and kept in the refrigerator for further usage.

Sample preparation

A total of 180 samples were used in the present study. First, standardized round plastic disc of 10 mm diameter and 2 mm thickness were prepared using a round mould, of which ninety disc samples each were allocated for surface roughness and microhardness test. Each set consisted of three groups of thirty disc samples according to the types of composite, as follows:

- Group 1 – Nanofilled composite Filtek-Z350-XT (3M ESPE, Seefeld, Bayern, Germany)
- Group 2 – Microhybrid composite Zmack-Comp (Zhermack, Badia Polesine, 45021, Italy).
- Group 3 – Zirconia-reinforced rice husk nanohybrid composite Zr-Hybrid (Universiti Sains Malaysia, Health Campus, Kubang Kerian, Kelantan, Malaysia).

The composition and manufacturer details of each type of composite resin used in this study are listed in Table 1. Waterproof transparent plastic stretch films (Manual Stretch Film, Scientex, Shah Alam, Malaysia) were placed on the top and below of the composite samples in the mould, and then compressed manually in between two flat glass slides (GLP2 × 2, United Scientific Supplies, Inc., Waukegan, US) using a 2-kg metal weight (abs-sl-weight-set-small, PCS Instruments, 78 Stanley Gardens, London, W3 7SZ, United Kingdom.) to obtain a flat bubble-free surface. The composite samples were then light cured using light-emitting diode (LED) light-curing unit Elipar Free Light 2 (3M™ ESPE™, St. Paul, MN, USA) with a light intensity of 600 mW/cm^2. The distance between the light source and the sample was standardized by placing a 1-mm-thick glass slide (GLP2 × 2, United Scientific Supplies, Inc., Waukegan, US) in between the light source and sample. During light polymerization process, the light source tip was ensured to be in contact with the glass plate. Besides, the LED light cure unit was calibrated using a LED radiometer (Demetron, Kerr, Danbury, CT, USA) before and after used for every two samples to ensure that the output of the emitting light source was standardized throughout the experiment. The polymerization process under LED light cure was performed for 20 s as recommended by the manufacturers. After that, the glass slides and plastic films were removed gently. All the composite samples were stored in an incubator at 37°C for 24 h to allow the composites to be fully matured.

After 24 h, the composite samples were polished and finished with aluminium oxide discs (Sof-Lex™, 3M ESPE, St Paul, MN 55144-1000, USA.) using all the four textures, namely, coarse, medium, fine, and extra-fine, in sequent for 60 s each. These discs were applied with light pressure using a low-speed handpiece running at 12,000 rpm in one straight direction only with continuous water

Table 1: Characteristics of composite resins used in this study

Type of composite	Name	Manufacturer	Type of resin monomer	Type of filler	Filler (weight) (%)
Nanofilled	Filtek Z350 XT	3M ESPE, Seefeld, Germany	Bis-GMA, Bis-EMA, UDMA, TEGDMA, PEGDMA	Zirconia and silica	78.5
Microhybrid	Zmack Comp	Zhermack, Badia Polesine, Italy	Bis-GMA, Bis-EMA, TEGDMA	Bariumglass and silica	77
Nanohybrid	Zr-Hybrid	Universiti Sains Malaysia, Kelantan, Malaysia	Bis-GMA, TEGDMA	Zirconia and rice husk silica	75

TEGDMA: Triethylene glycol dimethacrylate; Bis-GMA: Bisphenol A glycol dimethacrylate; Bis-EMA: Ethoxylated bisphenol A glycol dimethacrylate; UDMA: Urethane dimethacrylate; Bis-EMA: Ethoxylated bisphenol A glycol dimethacrylate; PEGDMA: Polyethylene glycoldimethacrylate

irrigation. Fresh abrasive discs were used for each sample. A single-blinded operator was chosen to polish and finish all samples to reduce experimental bias and variability. All samples used in this study were examined under a digital microscope (Hirox 3D Digital microscope, RH-2000, Hirox Co Ltd., Tokyo, Japan) at a ×20 magnification to ensure that they were free from defects.

Artificial ageing of samples

The thirty disc samples corresponding to each experimental group were further subdivided into aged ($n = 15$) and unaged ($n = 15$) subgroups. The samples in the aged subgroups were placed in a thermocycling machine (TS Series Liquid, Weiss Technik, North America) and subjected to artificial ageing for 2500 thermal cycles in sequential water baths of 5°C, 37°C, and 55°C. The dwell time was set at 30 s with a transfer time of 5 s.

Evaluation of surface roughness

The ninety composite disc samples, aged and unaged, belonging to the three experimental groups, were subjected to surface roughness test using a profilometer (MarSurf M 400, Mahr GmbH, Germany) with a 2-µm diamond stylus, a cutoff length of 0.80 mm, a measuring length of 2 mm, and a speed of 0.3 mm/s [Figure 1a]. Three recordings of surface roughness, R_a, were made for each sample. The average value was calculated.

Evaluation of microhardness

The remaining ninety composite disc samples, aged and unaged, belonging to the three experimental groups, were subjected to microhardness test using a diamond indenter [Figure 1b] of a Vickers hardness (VH) machine (Shimadzu Ltd., Tokyo, Japan). The load of the indenter was set at 300 kgf for 10 s.[11] Four indentations were made for each sample. The specimens were then viewed under a digital microscope (Hirox 3D Digital microscope, RH-2000, Hirox Co Ltd., Tokyo, Japan) at a ×20 magnification to measure the dimension of the indentations digitally. VH values were obtained based on the following equation:

$$VH = 1.8544 \times \frac{P}{d^2}$$

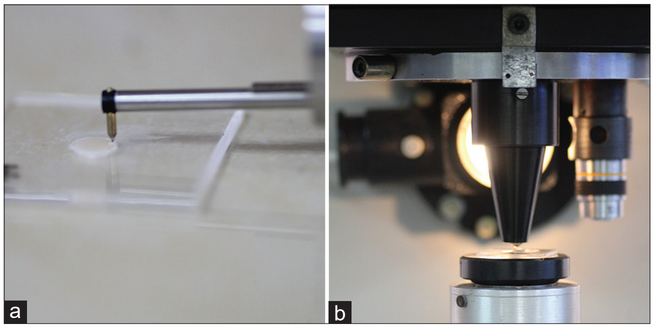

Figure 1: (a) Surface roughness test on composite sample using a contact stylus profilometer. (b) Microhardness test on composite sample using Vickers hardness machine

Where d is the average size of the indentations in mm and P is the load in N.

The values (Nmm^{-2}) of VH were calculated and compared.

Statistical analysis

Statistical analysis was performed using IBM SPSS version 24 for Windows 10 (IBM Corp., Armonk, NY, USA). The level of statistical significance was set at $P = 0.05$. Data were analyzed using two-way ANOVA complemented by *post hoc* Tukey's honestly significant difference multiple comparison test and paired sample t-test to compare the surface roughness and microhardness of different composite resins before and after artificial ageing.

Results

Based on Table 2, a significant difference was observed in which Zr-Hybrid showed statistically significant lower surface roughness than Filtek-Z350-XT ($P = 0.001$) and Zmack-Comp ($P = 0.001$), but no significant difference was noted between Filtek-Z350-XT and Zmack-Comp in unaged ($P = 0.997$) and aged ($P = 0.359$) groups, respectively. Data in Table 3 reveal that Zr-Hybrid showed the highest value of microhardness ($P < 0.05$) followed by Filtek-Z350-XT and lastly Zmack-Comp in both aged and unaged groups. Thus, the first null hypothesis was rejected. Besides, statistically significant differences ($P = 0.001$) were noted in both surface roughness and microhardness of all composite resins before and after artificial ageing according to Table 4. Therefore, the second null hypothesis was also rejected.

Table 2: Surface roughness, R_a (µm) of different composite resins using two-way ANOVA complimented by Tukey's honestly significant difference test

Group	Types of composite	Mean±SD	*F* (*df*)	*P*	Multiple comparisons			
					Groups	Mean differences	SE	*P*
Unaged								
1	Filtek Z350 XT	0.121±0.056	6.73 (2, 57)	0.002*	1 versus 2	0.001	0.031	0.997
2	Zmack Comp	0.122±0.034			1 versus 3	0.047		0.001*
3	Zr-Hybrid	0.106±0.044			2 versus 3	0.046		0.001*
Aged								
1	Filtek Z350 XT	0.248±0.076	4.27 (2, 57)	0.019*	1 versus 2	0.072	0.038	0.359
2	Zmack Comp	0.251±0.057			1 versus 3	0.111		0.001*
3	Zr-Hybrid	0.229±0.068			2 versus 3	0.040		0.001*

*Significant at 0.05. SD: Standard deviation; SE: Standard error

Table 3: Vickers hardness number of different composite resins using two-way ANOVA complement by Tukey's honestly significant difference test

Group	Types of composite	Mean±SD	*F* (*df*)	*P*	Multiple comparisons			
					Groups	Mean differences	SE	*P*
Unaged								
1	Filtek Z350 XT	67.88±1.94	259.71 (2, 42)	0.001*	1 versus 2	6.82	0.741	0.001*
2	Zmack Comp	61.56±1.57			1 versus 3	9.97		0.001*
3	Zr-Hybrid	77.85±2.47			2 versus 3	16.79		0.001*
Aged								
1	Filtek Z350 XT	75.65±1.58	348.19 (2, 42)	0.001*	1 versus 2	8.65	0.814	0.001*
2	Zmack Comp	67.01±1.44			1 versus 3	12.71		0.001*
3	Zr-Hybrid	88.35±3.21			2 versus 3	21.35		0.001*

*Significant at 0.05. SD: Standard deviation; SE: Standard error

Table 4: Comparison of surface roughness and microhardness values of all composite resins before and after artificial aging using paired sample *t*-test

Surface roughness				
Group	Type of composite resin	Mean±SD		*P*
		Unaged	Aged	
1	Filtek Z350 XT	0.121±0.056	0.248±0.076	0.001*
2	Zmack Comp	0.122±0.034	0.251±0.057	0.001*
3	Zr-Hybrid	0.106±0.044	0.229±0.068	0.001*
Microhardness				
Group	Type of composite resin	Mean±SD		*P*
		Unaged	Aged	
1	Control	67.88±1.94	75.65±1.58	0.001*
2	EndoREZ™	61.56±1.57	67.01±1.44	0.001*
3	Sealapex™	77.85±2.47	88.35±3.21	0.001*

*Significant at 0.05. SD: Standard deviation

Discussion

The role of polishing composite resin is to produce restoration with smooth surface similar to that of enamel. A significant purpose to polish composite restoration is to remove the resin-rich layer because it is relatively unstable in the oral environment.[10] However, removing this layer through finishing and polishing causes roughness on the polished surface of composite resins; the surface roughness of composite resin greatly affects the wear resistance, causes discolouration of the composite material, and increases plaque accumulation, which lead to gingival inflammation.[7,8,10] The present study used a two-dimensional profile to calculate the roughness parameter when measuring the surface roughness of composite resins, which is in accordance with previous studies.[12,13]

A significant difference in surface roughness among all the three types of composite resins using the same polishing protocol system was noted, in which the microhybrid composite, Zmack-Comp, showed the roughest surface, while the nanohybrid composite, Zr-Hybrid, showed the smoothest surface after polishing. The result of the present study was in agreement with the previous study that also showed high roughness value in microhybrid composite resin polished with aluminium oxide discs.[9,10] This could be attributed mainly to the filler size and filler particle content of the composite resins.[10,14] Zmack consisted of micro-size particles, whereas Filtek-Z350-XT and Zr-Hybrid consisted of nano-size filler particles in their compositions. Previous studies suggest that materials with larger filler size show greater surface roughness than those with smaller filler size,[3,8,10,15] due to the fact that nanoparticles disperse homogenously in the polymer matrix.[4] Although Reis *et al.* showed that a lower surface roughness can be obtained by increasing the amount of filler content,[16] this is in contrary with the result of the present study because Zr-Hybrid with the lowest filler content demonstrated the smoothest surface as compared to Filtek-Z350-XT and Zmack-Comp.

The critical surface roughness threshold suggested for bacterial adhesion according to a systematic review done by Bollenl *et al.* is 0.2 µm.[17] Surface roughness of any dental material above this threshold will result in increased plaque accumulation and possibly cause periodontal inflammation and increased risk of caries development.[17] In addition, this also compromises the esthetic aspect and longevity of the dental restoration.[18] Surprisingly, the results of the present study showed that all composite resins in the unaged group polished with aluminium oxide abrasive discs demonstrated surface roughness values lesser than the threshold, which is contrary to some previous studies.[8,12,15,19] This can be explained by the differences in polishing time. In the present study, each composite sample was polished for 60 s as compared to 15–20 s in some previous studies.[8,12,19] Thus, it can be postulated that the longer the period of polishing, the smoother the surface of composite resins.[20] Although all aged composite samples presented roughness values slightly higher than the threshold that is, 0.2 µm, the effect of polishing on the surface roughness of different composite resins was clinically relevant. Furthermore, Sof-Lex aluminium oxide discs were used in the present study to polish all composite samples due to the fact that it produces better surface smoothness and does not displace the composite fillers.[6] However, one of the limitations of this polishing system is their geometry, making them difficult to reach countered surfaces, especially in posterior restorations. Another factor that needs to be considered is the difficulty in controlling the manual pressure applied by different operators during polishing, and therefore, a bias might occur when comparing the result with other similar studies. In the present study, only flat surfaces were evaluated; thus, it does not fully represent the actual situation *in vivo*.

With regard to composite resin, an elevated hardness value is a good indicator of its ability to withstand wear and abrasion and prevent surface formation.[11] Several factors that might influence the hardness of composite resin include filler size, filler content, and type of resin monomer used.[21,22] In the present study, the microhardness of microhybrid composite with larger particle size exhibited lower hardness value than nanofilled and nanohybrid composites, which is in agreement with previous studies.[9,23] A smaller filler particle size allows the particles to be dispersed at higher concentrations and produces molecules that are more compatible to be coupled with the resin polymer during polymerization process,[21] thus, improving the physical and mechanical properties of composite resin. A direct correlation has been observed between the microhardness value and and the amount of filler loading in composite resin.[22] However, this trend is contradicted in the present study because Zr-Hybrid with the lowest filler content revealed the highest hardness value. This could be due to the reinforcement of the Zr-Hybrid with zirconia nano powder. Zirconia is widely used nowadays in dentistry due to its extremely high strength to enhance the mechanical properties of composite restoration, which has been reported in literature.[5,24] In addition, rice husk silica incorporated into composite materials has shown to provide good physical and mechanical properties.[4,25,26]

The differences in hardness values can also be explained based on the resin matrix component of the composite resin used. Filtek-Z350-XT and Zmack-Comp contain bisphenol A glycol dimethacrylate (Bis-EMA) in their composition, which is absent in Zr-Hybrid. Bis-EMA was found to have higher molecular weights than Bis-GMA, due to the lack of hydroxyl groups in its chemical structure.[27] Therefore, composite resins based on Bis-EMA can exhibit higher conversion values than Bis-GMA resins.[28] A correlation between the degree of conversion and microhardness of composite resins was noted, in which higher degree of conversion leads to enhanced physical and mechanical strengths.[23,29,30] This is in contrast with the result of the present study because Zr-Hybrid presented with the highest hardness value in the absence of Bis-EMA. However, correlations based merely on resin matrix composition cannot be fully justified because most manufacturers do not disclose proprietary information regarding the specific weightage or volume of the resin matrix used.

The artificial ageing method used in the present study allows a better assessment of the degradation of composite resins over a short period of time by stimulating the physiological ageing of the materials in the oral cavity.[11,31] Although previous studies revealed that the hardness of composite resins decreased after artificial ageing,[32-35] the result of the present study is similar to that done by Gomes *et al.*,[11] which showed increased hardness value after thermocycling process. During artificial ageing process, the resin matrix degrades and at the same time, polymer conversion process also takes place.[11] Therefore, it can be speculated that the rate of additional polymer conversion exceeded the rate of resin monomer degradation in the present study, thus explaining the result. However, the result of the study could have been partially affected by the inability of the artificial ageing process to reproduce all the variables found in the oral environment such as the presence of saliva, masticatory forces during chewing, and different types of food and beverage consumed.

Although the present *in vitro* study attempts to create a clinically relevant testing environment, *in vivo* test and evaluation can provide more reliable outcomes regarding the long-term surface roughness and microhardness of composite resins. Hence, future research should be directed toward the long-term clinical evaluation of this novel zirconia-reinforced rice husk nanohybrid composite.

Conclusion

Within the limitations of the present study, Zr-Hybrid demonstrated the lowest surface roughness and highest microhardness value as compared to commercialized composite resins. Artificial ageing significantly affected the surface roughness and microhardness of all composite types.

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Conflicts of interest

There are no conflicts of interest.

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