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Research Article

SURFACE CHARACTERISTICS OF COMPOSITE RESIN ENHANCED BY NEW ANTIBACTERIAL NANOFILLERS

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A B S T R A C T

Antimicrobial Magnesium oxide (MgO) nanoparticles has recently shown to be homogenously incorporated in dental materials when coated with Zein polymer.
The aim of the study was to investigate the effect of Zein coated MgO nanoparticles on the surface roughness and wettability of flowable resin composite.
Methods: Sixty disc shaped samples were prepared and assigned into six groups (n=10). In the control group, samples were prepared with flowable composite only. In the other five groups, Zein coated MgO nanoparticles of 5 different concentrations (0.3%, 0.5%, 1%, 2% and 5% by weight respectively) were added to the flowable resin. Samples were tested for wettability by drop shape analyzer, while for surface roughness samples were tested using profilometer, followed by scanning electron microscope analysis for particles distribution and surface configuration. Data was collected and analyzed statistically by ANOVA followed by PostHoc test at $p < 0.05$.
Results: For wettability, there was no statistically significant variance among all groups ($p<0.05$), except for 0.3% concentration; where wettability was increased ($p=0.006$). For surface roughness, no statistically significant difference was found between all groups, except for 5% concentration, where roughness increased ($p= 0.0001$).
Conclusion: Incorporating antimicrobial MgO nanoparticles in composite resin does not alter its surface properties.

INTRODUCTION

Nanotechnology is used in dentistry for developing dental materials with superior properties. Different nanoparticles were added to dental materials to improve the properties of the conventional and flowable composite dental resin. They are also promising in inhibiting caries by controlling biofilm acids and enhancing remineralization (1). Active biofilm formation is directly related to caries development adjacent to dental restorations(2,3), it has been reported that ceramics and resin composites show biofilms thicker than that of glass ionomer. Many researchers attributed the accumulation of bacteria to the surface roughness of composite restorations(4,5). However, biofilm formed on composite restorations was not related to certain type of composite resins(6,7).

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loading have been shown to increase resin volumetric shrinkage and consequently, shrinkage stresses(8,9). Such internal stresses may lead to interfacial debonding, marginal deterioration and secondary caries(10).

Studies (11) evaluated the antimicrobial activity of composites containing Zinc Oxide (ZnO) nanoparticles and changes in their physical and mechanical properties.Their study revealed that there is a potential of producing antimicrobial composite while preserving its mechanical and physical properties. Others(12) assessed the effect of adding Silver nanoparticles to composite on its physical properties and antibacterial activity. They concluded that the use of low concentration of silver (0.3%) had better antimicrobial property without affecting its compressive strength and surface roughness. Also, investigators (12) studied the antimicrobial and mechanical properties of composite resins modified by the addition of TiO_2 nanoparticles. Their study showed an antibacterial property added to composite with acceptable shear bond strength values with the low concentrations of Titanium oxide NPs. While other researchers [13)] showed that addition of low concentrations of metal oxide nanoparticlesto compositeinduced an antibacterial activity towards different organisms.

Magnesium oxide (MgO) have shown an excellent antimicrobial properties against streptococcus mutans and many other gram positive and negative bacteria[(14-18)]. It is safe, stable and able to bind and damage bacterial membranes [(19)]. However, it was never incorporated in dental materials to take advantage and benefit of its potent antibacterial properties. That is because the material's properties are exhibited differently at nanoscale level than at the atomic level [(1)]. MgO nanoparticles have the tendency to agglomerate, which hinder their nano properties, due to the lack of dispersion that causes its agglomeration in the material, changing it to a macro scale filler and hence loosing its nano properties and also affecting the stability and properties of the material itself [(20)]. Recently, coating MgO nanoparticles with zein polymer has shown to ease particles dispersion and to prevent agglomeration[(20)]. Also its use as a coating to MgO nanoparticles prolonged their release to 60%[(20)]. Zein is a new biopolymer that has been applied in different fields like food industry, pharmaceuticals and biomedicine. The advantages of Zein polymer that elected it to be widely used are: its biocompatibility and biodegradability with low toxicity; its hydrophobicity and solubility; its slow digestion that makes it perfect for oral route use; and its ease of fabrication[(21)]. That is considered a break through in allowing MgO nanoparticles to be incorporated in dental materials.Incorporating the MgO nanoparticles in flowable composite (FC) will add the antimicrobial property to the composite and will help in reducing carries recurrence. However, its effect on the mechanical and physical properties of composite has not yet been tested.

Thus, this research aims to evaluate some of the physical properties of FC; the roughness and wettability after adding MgO nanoparticles. The null hypothesis of this study was that addition of nanoparticles would not affect the wettability and surface roughness of FC.

MATERIALS AND METHODS

Materials

MgO nanoparticles (spherical, 10nm Nanoparticles, US Research nanomaterialsinc. Texas 77084, USA) , Zein polymer (Sigma Aldrich, Misouri 63103, USA) and Tetric N-Flow (IvoclarVivadent, Liechtenstein) were used in this study.

Methods

Zein coating of MgO nanoparticles

Zein was weighed and dissolved in a blend of ethanol (C2H5OH) and sodium hydroxide (NaOH) solution, sonicated then carefully dropped in polyvinyl alcohol (PVA) at 10 °C. The suspension was stirred at 500 rpm until complete evaporation of ethanol. Zein coated MgO nanoparticles were washed with distilled water to remove the excess PVA, then, centrifuged for two cycles differential centrifugation (3,000 rpm for 45 min). Then, the supernatant was removed and the sediment was dissipated in 5 ml of buffer, followed by addition of trehalose and lyophilized. (Virtis TM, Bench Top model, USA) [(20)].

Samples preparation

Six groups of flowable composite samples (Tetric N-Flow FC) were prepared with 10 samples each (n=10). One control group was with only flowable composite, and five other groups with five different concentrations, (0.3, 0.5, 1, 2 and 5 % respectively) of MgO nanoparticles. Each composite sample was weighed by a sensitive scale. Then the calculated amount of MgO nanoparticles representing each concentration was added by weight. The FC and the MgO nanoparticles were mixed in a double-ended syringe in a dark room to prevent polymerization of the composite.

The FC Tetric N-Flow and five composite mixtures were then poured into disc-shaped molds 10-mm in diameter, 2mm in thickness. A glass slab was held against the mold from one side, and the composite blend was pressed into the mold from the other side using celluloid strips, to obtain a smooth surface. Then, the resin composite was light-cured for 40 seconds from both sides using an LED light-curing device (Paradigm™ 3M, Canada). In this manner, sixty samples were obtained and categorized into six groups (n=10); 0.3% MgO group, 0.5% MgO group, 1% MgO group, 2% MgO group, 5% MgO group and control group without MgO particles.

Profilometer

All specimens were tested for surface roughness by non-contact profilometer (Contour GT, Bruker, USA). The arithmetical mean deviation of the assessed profile (Ra) was recorded for each sample to determine the degree of surface roughness among the different groups.

Drop Shape Analyzer

Wettability of the specimens was also measured using the drop shape analyzer (DSA100, Kruss, Germany). A single water drop is placed on each specimen then the contact angle was measured on both points of the droplet, and the mean was calculated and recorded for each group.

Scanning electron microscope (SEM)

Specimens were platinum coated and analyzed by SEM to determine the distribution of the nanoparticles and the surface configuration of the resin samples.

Statistical analysis

The results for the surface roughness and wettability were evaluated using one-way ANOVA test, followed by Post-Hoc test. All the statistics were done using SPSS (Version 18.0. Chicago: SPSS Inc.), at a significant level of $p<0.05$.

RESULTS

Data were analyzed by one-way ANOVA, the mean value of contact angle and surface roughness are presented in Figure 1 and 2.

One-way ANOVA test for the wetting properties displayed a significant difference between the groups ($p=0.029$). Further testing was performed with the Post Hoc T-test between the control and all study groups. It revealed a significant variance among the control and 0.3% group ($p=0.006$) (Figure 1).

Furthermore, the surface properties were also tested by One-way *ANOVA* indicating no significant variance among the groups of added MgO nanoparticles by 0.3%, 0.5%, 1% and 2% by weight and the control. However, the group with 5%

MgO nanoparticles showed statistically significant increase in surface roughness (P =0.0001) (Figure 2).

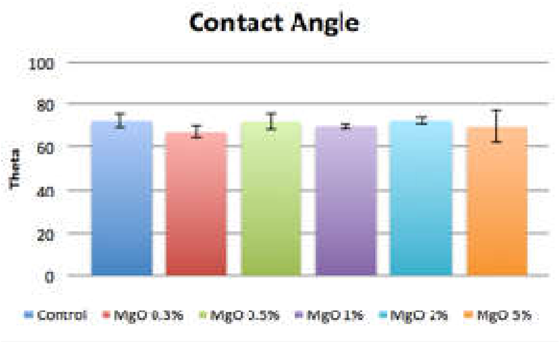

Figure 1 The mean contact angle of each group

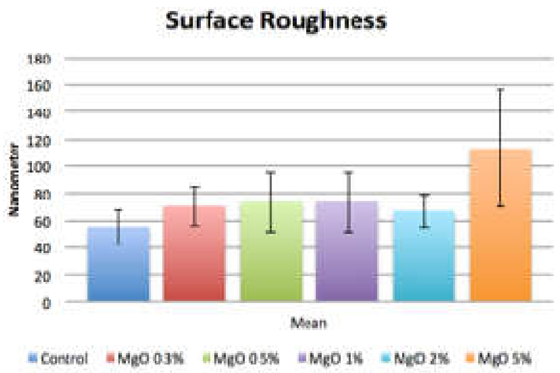

Figure 2 Surface roughness mean values (Ra) of each group

SEM images showed a good dispersion of zein coated MgO nanoparticles in flowable composite and confirmed the results of surface roughness measurement with the profilometer (Figure 3). The MgO nanoparticles were homogenously well dispersed in the resin for groups of 0.3%, 0.5%, 1%, and 2%. While the group of 5% MgO nanoparticles, clusters began to form showing non-homogenous surface. (Figure 4)

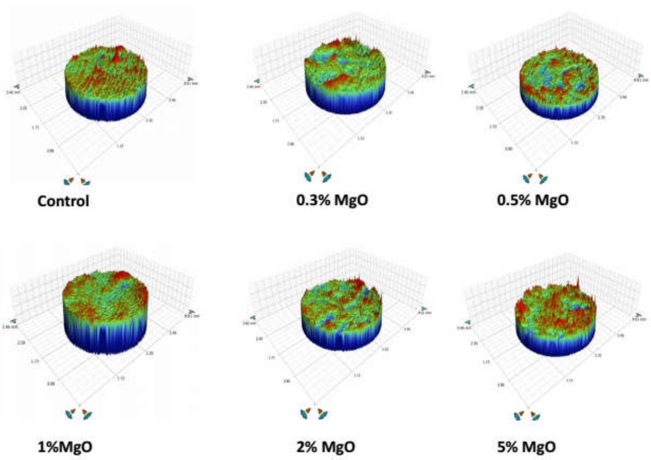

Figure 3 Profilometer images displaying the surface roughness results of flowable composite with different concentrations of MgO nanoparticles

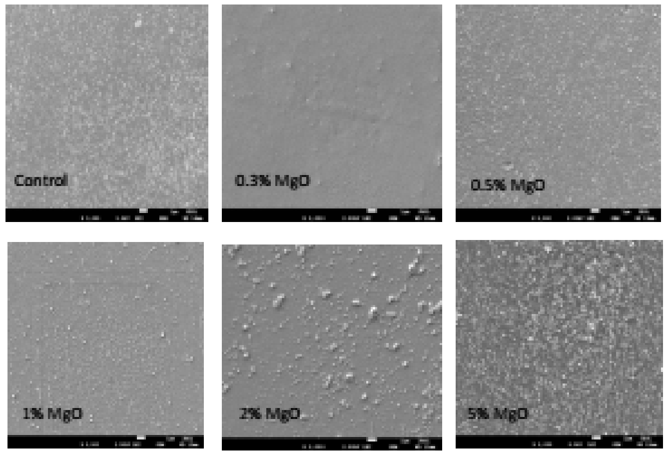

Figure 4 SEM images displaying the dispersion of different concentrations of MgO nanoparticles in flowable composite

DISCUSSION

Caries recurrence is one of the main causes for tooth destruction and eventually resulting in its loss. Nowadays, the use of antimicrobial restorative materials could be a getaway for increasing restorations life span[(22)]. This study investigated the changes in surface properties of FC after addition of Zein coated MgO nanoparticles. As wettability and surface roughness play essential roles in the hydrophilicity and depth of wear of composite restorations. Both of which also have crucial roles in the accumulation of bacteria specially in flowable composite with its low filler content and high volumetric shrinkage leading to marginal deterioration, and secondary caries [(8,9,23-25)].

MgO nanoparticles were proven to be safe and stable antimicrobials (Jin and He 2011) but have not been incorporated in dental materials before, due to the lack of dispersion that causes its agglomeration in the material[(20)]. Coating MgO nanoparticles with zein polymer has shown to ease particles dispersion and to prevent agglomeration[(20)], allowing the MgOnanofillers to perform its antibacterial properties in its nano status when incorporated in dental materials without altering the material stability and properties. Incorporating the MgO nanoparticles in flowable composite (FC) is expected to add the antimicrobial property to the composite and will help in reducing carries recurrence.

Previous studies showed that the addition of any nanoparticles to dental composite restorations in a concentration higher than 10% showed significant decrease in its mechanical properties[(22,26,27)]. However, the use of low concentration of 0.3% of nano particles was found to give the antimicrobial property of nanofillers without affecting its materials [12)]. Based on that, this study used those ranges of MgO nanoparticles concentrations, (0.3%, 0.5%, 1%, 2% and 5% by weight). As those concentrations had also shown effective antimicrobial property, especially against streptococcus[(18)].

Also, the shape, size and amount of filler particles played an important role in the overall structural integrity and surface texture of flowable composite([28-30)]. A spherical and small size nanoparticles have proven to produce less surface roughness compared to other shapes and sizes[31)]. For that reason, small spherical shaped MgO nanoparticles (10nm) were used in our study.

The main factors affecting the wetting of dental composite are: Surface roughness, Surface tension and surface energy. As stated by Tang *et al*. (1991): " the use of smaller particle size leads to an increase in the surface area, accompanied by an increase in the percentage of nonmagnetic to magnetic surface area"[32]. Hence, small particle size is associated with high surface energy leading to a more reactive surface and increasing wettability. In this study, the addition of different concentrations of MgO NPs to flowable composite had no significant difference except for the 0.3% concentration, which increased the surface energy of the composite, decreased the contact angle and increased the wettability of the composite. This is in accordance with previous studies that proved that small sized nanoparticles increased the surface energy of hydrophobic materials like FC, causing a reduction in contact angle, and hence increasing its wettabilit([27,32]. However, contact angle values were still higher than that of Bisphenol-A-glycidyldimethacrylate (65°) (Craig 1979) indicating high hydrophobicity of all tested samples.

This interesting finding can be practically valuable, indicating that the zein coated MgO nanoparticles were effectively dispersed in the composite resin as seen by SEM analysis (Figure 4). Furthermore, this will encourage the use of the 0.3% MgO nanoparticles with Flowable composite in order to obtain a smoother surface with conserving its hydrophobicity and with antibacterial property that will increase the restorations lifespan by preventing recurrent caries.

Composite surface roughness depends on the type of matrix, filler type, size, shape and distribution[33]. Studies showed that addition of silica Nano fillers and silver nanoparticles resulted in an increase in the surface roughness of [26]. Also, the higher the concentration of nanoparticles added to FC, the more the[34]. However, this phenomenon was not observed in our study. The 5% group was the only group with a significantly higher surface roughness. This is probably due to increase of filler to matrix ratio.

As the surface roughness of the restoration will appear optically smooth when their surface Ra value is smaller than 0.1μ [35]. It has been suggested in a previous study that the maximum threshold value of surface roughness below which there is no observed reduction of bacterial retention is Ra=200nm[25]. In this study the surface roughness values were 70-140 nm indicating no change in the surface properties of flowable composite.

Dispersion of nanoparticles is an important factor to ensure that they perform and do their role in their nano structure. SEM analysis of platinum coated samples showed that Zein coated MgO nanoparticles were well dispersed in all concentrations. (Figure 4).

Our results showed that the addition of 0.3%, 0.5%, 1%, 2% and 5% MgO nanoparticles to FC did not change its wettability and surface roughness. This indicates that the use of small (10nm), spherical nanoparticles and coating them with the natural Zein polymer to enhance their dispersion produced more stable antibacterial nanoparticles that can be added safely to FC. Their use in FC will make us benefit from their antibacterial properties without altering the FC surface properties.

Thus, this antibacterial flowable composite will be beneficial not only as a liner under restorations but in use with orthodontic appliances and in prevention as fissure sealant.

CONCLUSION

Within the limitations of this study, it is concluded that low concentrations of Zein coated MgO nanoparticles can be incorporated safely in FC to provide it with a potent antimicrobial effect without altering the physical properties of surface roughness and wettability of the composite resin.

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