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Original Article

Mechanical properties of resin composites containing bioactive glass and experimental nano zinc-silica complex

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Abstract

Aim: Secondary caries is an important problem in dental composite restoration, and nanoparticles are commonly added to the structures of resin composites to improve their antimicrobial properties. The aim of this study is to evaluate the mechanical properties of composite materials containing bioactive glass (BAG) and an experimental nano zinc-silica (NZS) complex.

Methodology: An experimental resin composite containing 70 wt% filler was produced and used as a control sample. This experimental resin composite was then modified by adding different amounts of BAG (10%), NZS (10%), and both BAG and NZS (10% + 10%). NZS was synthesized in situ by milling zinc and silica to nanoscale level. Compressive strength and flexural strength were investigated using a universal testing machine. Data were analyzed using one-way ANOVA and the Tukey post-hoc test.

Results: There were no statistically significant differences in compressive strength caused by the filler amount, but statistically significant changes were found in flexural strength. Although the addition of antimicrobial agents to resin composites reduces their physical properties, this is not a clinically unacceptable limit.

Conclusion: NZS exhibits better mechanical properties than does BAG, but both materials can be used safely in restorative materials.

Keywords: bioactive glass, experimental composites, mechanical properties, resin composites, nano zinc-silica

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Introduction

Resin composites are frequently used in dentistry because of their ease of application, ability to enable bonding to the tooth structure, high aesthetic quality, adequate strength, and reasonable cost. An additional reason for the increase in the use of resin composites is the risk of release of trace amounts of Hg from amalgam restorations (1).

Resin composites can provide good sealing of cavities, but polymerization shrinkage or functional loading may lead to interface failure and gap development. In addition, resin composites cause more plaque accumulation than do other restorative materials or human enamel. Bacterial accumulation in marginal gaps or plaque formation increases the risk of secondary caries (2, 3).

Agents that have antimicrobial effects can be added to increase resistance against secondary caries, or remineralization can be performed after damage has occurred (4). Bioactive glass (BAG), which includes oxides of calcium, sodium, phosphorus, and silicon (5), is a popular alternative because of its potential remineralization-promoting effects, antimicrobial properties, and biocompatibility (6). In this regard, many studies have examined the effects of the antimicrobial properties of different BAG compounds on various bacterial species. However, there are several concerns about the development of bioactive dental composites (BACs). BAG fillers are unsuitable for resin matrices or leach ions over time, resulting in poor mechanical properties (2).

Another important advancement in this field is the development of nanoparticles (NPs) that can be added into resin matrices and exhibit antimicrobial properties. Nanotechnology mainly involves the generation and application of materials and structures sized 0.1–100 nm by diverse physical or chemical methods. Metal oxide NPs sized 5–100 nm can be added into resin matrices (1). The mechanism of NPs against bacterial toxicity is still being investigated, but the cause may be free metal ion toxicity from the NP surface and/or oxidative stress caused by reactive oxygen species (ROS). When ROS, such as OH, $^1O_2$, and O^-_2, or free metal ions directly contact the cell wall, they attack unsaturated phospholipids in bacteria and damage the cell membrane by electrostatic interaction, failure of metal–metal ion homeostasis, protein and enzyme dysfunction, genotoxicity, and photokilling (7,8). Direct contact of zinc oxide NPs (ZNPs) with cell walls results in the liberation of antimicrobial Zn(2+) ions and causes ROS formation, thereby compromising bacterial cell integrity. Silicium oxide NPs (SNPs) reduce the bacterial penetration of dental plaque. They also have a less toxic mechanism, reduce adhesion to plaque, and decrease the natural proliferation of bacteria (8, 9).

The mechanical properties of conventional resin composites deteriorate differently in aqueous environments depending on the material composition. Such properties degrade with age in BACs because of hydrophilic characterization and the soluble fillers in structure. Besides, the opacity of metal oxide NPs (MONPs) in visible light may reduce light curing and thus the mechanical properties of composites (10, 11).

In the addition of MONPs into the composition of a resin composite for improved curing depth and aesthetic appearance, the added quantity should be minimized because of the opacity of MONPs (11). The compression forces occuring at occlusal loading are concentrated on filler particles. Then, cracks begin to form in the filler particles and diffuse into the resin matrix (12). Therefore, the mechanical properties of the material are affected by the particle size and distribution, and a decrease in particle size increases the fracture strength. The dilemma is how to improve the mechanical and antimicrobial properties of the material without weakening the aesthetic properties. Experimental nano zinc-silica (NZS) complexes have been developed to take advantage of the combined effects of ZNPs' antimicrobial properties and SNPs' reduction of the bacterial penetration of dental plaque.

Previous studies on this topic have examined the effect of the mechanical properties of restorative materials when added into the resin composites of similar antimicrobial agents in different ratios. The purpose of this study is to determine whether the addition of NZS and BAG into a resin composite would affect its mechanical properties. Thus, the null hypothesis is that the compressive strength and flexural strength (FS) of resin composite samples are not affected by the addition of BAG or NZS.

Materials and Methods

Production of Resin Composites

For this study, resin composites containing two antimicrobial agents (BAG and NZS) were produced. The contents of the produced composites are shown in Table 1.

Silanized inorganic fillers with different particle sizes were used. The inorganic fillers were milled to the required size in aqueous media by using ytterbium-stabilized zirconium oxide balls in the attritor (01-HD/HDDM Lab Attritor, Union Process, Ohio, USA).

Table 1. Compositions of investigated materials

GROUPS	COMPOSITIONS
CONTROL	• %30 Resin matrix (%70 Bis-GMA; %30 TEG-DMA) • %1 photoinitiator (camphorquinone) • %2 co-initiator Ethyl 4-(dimethylamino)benzoate; • %70 BG filler (0,7 µm)
BAG	• %30 Rezin Matrix (%70 Bis-GMA; %30 TEG-DMA) • %1 photoinitiator (camphorquinone) • %2 co-initiator Ethyl 4-(dimethylamino)benzoate; • %60 BG;

	• %10 BAG (2 µm)
NZS	• %30 Rezin Matrix (%70 Bis-GMA; %30 TEG-DMA) • %1 photoinitiator (camphorquinone) • %2 co-initiator Ethyl 4-(dimethylamino)benzoate • %60 BG • %10 NZS (20-40 µm)
BAG+NZS	• %30 Rezin matrix (%70 Bis-GMA; %30 TEG-DMA) • %1 photoinitiator (camphorquinone) • %2 co-initiator Ethyl 4-(dimethylamino)benzoate; • %50 BG filler • %10 BAG, %10 NZS

∗ Bis-GMA: bisphenol A glycidyl methacrylate (Sigma-Aldrich Chemie Gmbh, Steinhelm, GERMANY); TEG-DMA: triethylene glycol dimethacrylate (Sigma-Aldrich Chemie Gmbh, Steinhelm, GERMANY); BG: Baryum Glass; BAG: Bioactive Glass; NZS: Nano-Zinc Silicium

They were then silanized in an inert nitrogen atmosphere reactor (SS 316, Amar Equipment, Mumbai, India) using methacryloxy prophyl trimethoxysilane. The silanized inorganic fillers were dried in a vacuum incubator (VDL 53, Binder Gmbh, Tuttlingen, Germany) at 120 ° C for 3 hours. Inorganic fillers weighed in precision scale and slowly added into the monomer matrix at the desired amount and mixed with specialized production triaxial mixer (001 Mixer, İntermak Inc, Konya, Turkey) at room temperature for 3 hours. The air bubble was detract from the product by stirring again under vacuum for 30 minutes after mixing. Red illumination light was used in the laboratory lighting to prevent photoinitiators from starting the reaction.

In this study we had four experimental groups. Group 1: Control; Group 2: BAG (10%); Group 3: NZS (10%); Group 4: BAG+ NZS (10%+10%).

Compressive Strength

According to ISO 9917 for the compressive strength CS tests, the stainless-steel cylindrical molds with diameter of 4 mm and height of 6 mm were placed on a glass slide and then overfilled with the resin composites (Fig. 1). After complete filling of the mold, another glass slide was pressed on the top side and the whole materials cured for 40 s from each end. The lateral sides of the cylindrical resin specimens were cured for further 40 s in order to achieve higher polymerization. The specimens of each group were stored in water at 37◦C for 24 h prior to test. The CS was then determined with the universal testing machine (Instron, Canton, MA, USA) at a cross-head speed of 1 mm/min. CS was determined in megapascals (MPa) by dividing the failure load (N) with the specimen cross-section area (mm^2).

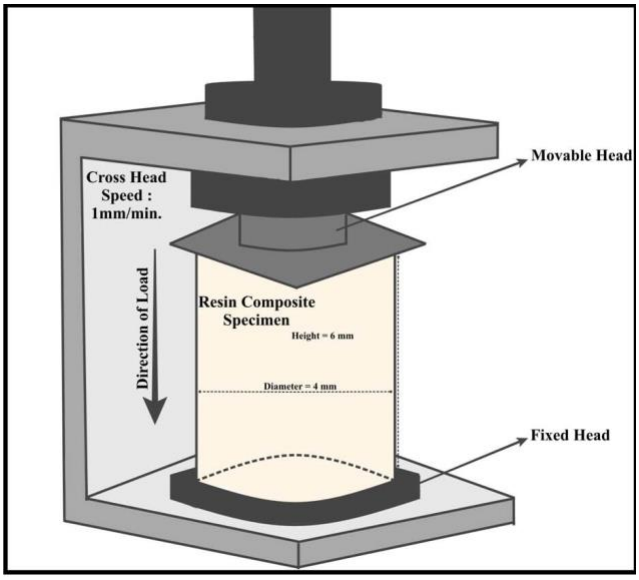

Figure 1. Compressive Strength Application

Flexural Strength

FS is one of the most important mechanical tests for assessing the performance of dental resins. According to ISO 4049, the resins were inserted in a rectangular stainless-steel mold with 2 mm × 2 mm × 20 mm dimensions, which was placed on a glass slide (Fig. 2). Then, the mold was covered with another glass slide and specimens were cured from both top and bottom sides by a light-curing unit irradiated for 40 s in each spot using an overlapping regime. The specimens were removed from the mold and stored in distilled water for 24 h at 37◦C prior to the test. Both surfaces of all specimens were polished using a 600 grit silicon carbide paper in a moist environment. A three-point bending test was performed using a universal testing machine at a cross-head speed of 0.5 mm/min. The FS in MPa was calculated as:

$$FS = \frac{3 \times P \times L}{2 \times b \times d^2}$$

where P stands for load at fracture (N), L is the span length (20 mm), and b and d are, respectively, the width and thickness of the specimens in millimeter.

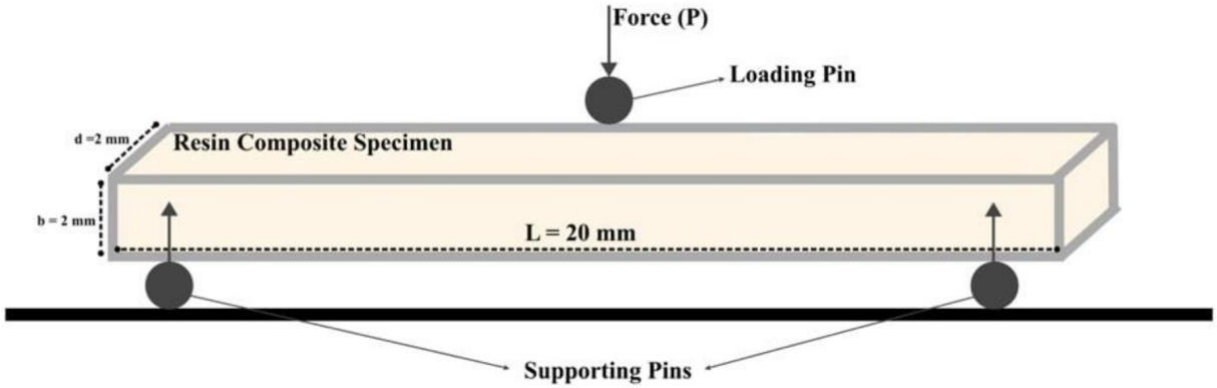

Figure 2. Flexural Strength Application

Statistical analysis

Analysis of the data was carried out with SPSS software version 22.0 (IBM Corp., Armonk, NY, USA) by 1-way analysis of variance (ANOVA) and the Tukey post hoc HSD multiple comparison test at p<0,05.

Results

The compressive and flexural strength values of resin composites with different antimicrobial agents are shown in Table 2. These values indicated that there was no significant difference between experimental composites and control group on CS values (p>0,05). A statistical comparison of FS values, significantly difference between Control and BAG groups was detected (p<0,05; p=0,002). There was no significant difference between BAG, NZS, and BAG + NZS groups (p>0,05).

Discussion

This study examined the comparison of mechanical properties of two different antimicrobial agents under different conditions and the null hypothesis was partially accepted.

MONPs is being investigated as an antibacterial agent in both microscale and nanoscale formulations and in case particle size decreased, contact with bacterial cell wall is increased thus exhibits significant bactericidal mechanisms (8). Different metal oxides have been examined for microbial toxicity, and promising results have emerged. Besinis et al. conducted the study examined the efficacy of titanium dioxide, silicon dioxide, and silver nanoparticles (TNP, SNP, AgNP) against S. Mutans; AgNP has been found to be the most effective antimicrobial agent, but SNP and TNP have limited or no effect against it (9). However, the fact that silver ions have a coloring effect on the resin composites limits their use in the structure. Adams et al. carried out a study with TNP, SNP, and ZNP against B. Subtilis, and they mentioned the ZNP exhibit effectiveness at lower concentrations. TNP or SNPs effectiveness has been revealed at higher concentrations or presence of light (13). Tavassoli Hojati et al. reported that the highest value of the CS at 1 wt.% ZNP content group and no significant difference for FS in their studies which examined the mechanical properties of the resin composites with different ratios of ZNP (11). Chen et al. investigated the reinforcement of dental resins without any silica particles and conventional glass filler composites with various mass fractions, and they reported that the values of biaxial FS were significantly increased by the addition of smaller rates (14). Hosseinalipour et al. examined the influence of filler particle size and filler loading on mechanical properties of composite resins and showed FS increased with filler mass fraction up to 40%, and then decreased sharply (1). Stencel et al. investigated the impact of the rate of silver releasing nanofiller into resin-based composites for its mechanical properties and they found no significant difference for CS but for FS, control group has the highest mean value, and increasing antimicrobial filler concentration caused significantly decrease for this situation they stated that the reason could be an error in one of the samples, and the average value did not

differ significantly if the related sample was ignored (15). Khvostenko et al. compared the mechanical properties of the control group and the experimental group with BAG, and they reported that the group containing BAG showed superior FS than the control group (2). Par et al. investigated the effect of systematically varying BG amount in experimental composites on mechanical properties of resin composites and they reported that lower rates of BG showed higher FS than the commercial reference, whereas increasing the BG amount beyond 10 wt% resulted in significantly lower FS (16). Korkut et al. evaluated the mechanical properties of resin composites containing different amounts of microparticulate BAG and they stated that the both of compressive and flexural strength decreased significantly of resin composites which include 30 wt.% BAG (17).

After all that studies, both of the compressive and flexural strength of resin composites containing antimicrobial agents, even if it is not significant, would provide to mechanical improvement up to a threshold beyond which more antimicrobial fillers will no further increase the mechanical properties but none of them evaluated to which material has better mechanical properties.

All restorations determine an internal defect such as pores and filler agglomerates, even if in small amounts, so it is an obligation to evaluate material strength when evaluating these materials (1). CS of the composites is higher than the tensile strength, and it is more affected by the internal defects, which is probably the most suitable strength test (18). The compressive force has an important role in the chewing process because most of the masticatory forces have a compressive effect. The maximum CS is calculated using the cross-sectional area and the maximum forces of the sample.

FS has been reported as a method for determining the clinical wear indicators or tensile failure of composites (15). Furthermore, composite restorations are subjected to FS, particularly in stress-bearing areas like class I, II, and IV restorations (19). FS of composite materials should be at least 80 MPa for occlusal surface restorations and 50 MPa other restorations (20).

According to our study, there was no significant difference between the CS of the investigated composites; however, BAG has the lowest, and the highest CS was observed in BAG + NZS groups. While BAG addition influenced the CS values, this problem could be compensated when added with NZS.

Another result of this study is the addition of any antimicrobial agent to the restorative material reduces its FS. The samples added BAG exhibited dramatically lower FS than the others, and the difference between the control group was significant. Korkut et al. reported that the FS values were lower in the groups with 5-10% BAG compared to the control group and the statistical difference was in the group with the addition of 30% BAG (17). Khvostenko et al. also reported FS values would not be affected by BAG up to 15% (2). After all, the FS of all samples is above the clinically acceptable limits.

Conclusions

The limitations of this study are this in vitro study may not fully simulate the oral environment because of no biofilm formation on the samples; also, adding different ratios of various nanoparticles in resin composites could derived the diversity of results. In future studies, the use of more diverse antimicrobial agents with different ratios would be appropriate.

Within the limitations of this study, the addition of antimicrobial agents to the resin composites reduces the mechanical properties, but this decrease will not be clinically unacceptable besides NZS exhibit better mechanical properties than BAG.

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