

RESEARCH ARTICLE

Synthesis of Zinc Oxide (ZnO) and Study on Mechanical Properties of Polymeric Dental Filling

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ABSTRACT

Polymeric dental filling (light-cured dental filling) has many problems like polymerization shrinkage and weakness in mechanical properties. Recently, scientists found that nanoparticles have good solutions to many problems in dentistry. Nanoparticles were prepared from ZnO, by sol-gel, laser ablation, chemical, and sol-gel methods according to papers published earlier. These nanoparticles were then added to the light-cured dental filling as filler.

The polymeric dental filling containing the nanoparticles prepared were then examined by X-ray diffraction, depth of cure, compression strength, hardness (lower and upper surfaces), degree of cure, the relation between the depth of cure and degree of cure, the diameter of samples, and polymerization shrinkage. The examinations and results showed that polymeric dental filling containing silver nanoparticles has maximum compression strength and hardness and no polymerization shrinkage. The most important result was that ZnO nanoparticles filler canceled polymerization

Keywords: Dental filling, Laser, Nanoparticles, Light-Cured, Polymer.

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INTRODUCTION

Using resin composite in treating teeth began when Joseph Redenbacher discovered acrylic acid and used acrylic resin as a dental restoration filling¹ and afterwards, polymethyl methacrylate was used as a resin denture in 1930.¹

In 1951, another type of dental filling, which was known as resin-based composite, was discovered where it consisted of polymer plus ceramic but suffered from some problems like polymerization shrinkage (reducing the size of dental filling after polymerization process) and weakness in mechanical properties, and thus polymeric dental filling falls.² For this reason, some of inorganic materials were then added as fillers to this resin composite to solve these problem.³

After advent of nanotechnology, researchers tried to enter it in dental restorative materials where they introduced nanoparticles as filler to dental restorative filling.⁴

Inserting nanotechnology in dentistry has great perspective hopes to the dentist and patient. no dentistry increased health care to tooth and mouth by treatment and diagnosis.⁵

Constituents of Polymeric Dental Filling

Polymeric dental filling (resin-based composite) has three components:

1. Organic phase like the monomer bisphenol –A- glycidyl methacrylate (BIS-GMA).
2. Inorganic phase which is an inorganic filler.
3. Bonding agent that bond the organic phase to the inorganic filler.

Polymeric Dental filling, for this reason, is called a composite because it includes three different materials.

Polymerization Process

The organic phase, the monomer (BIS-GMA), is in the fluid state, but when it is cured by light, the monomers convert to polymer where the polymer is in a solid state. The curing process is a transformation of fluid material to solid material.

Light types used in the curing process are halogen, laser, LED, and ultraviolet. When any one of these types of light fall on organic phase or polymeric dental filling, it will convert to solid state, and monomers convert to polymer.

The polymerization process is a transformation of monomers to polymer using light. The more monomers convert to polymer, the higher the polymerization degree will be, i.e., polymerization enhances and becomes better; thus the fluid convert to solid-state.

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Aim of this Work

This work aims to improve polymeric dental filling with nanoparticles.

MATERIALS AND METHODS

The base material used in this work is the polymeric dental filling “COMPOSAN LCM” with three phases mentioned above; this dental filling was assumed to be the organic phase.

Four nanomaterials were prepared, as explained in 2.1, and then added as filler to the base material (COMPOSAN LCM); they are the inorganic phase.

Preparation of the Nanoparticles

5 g of finely grounded plant powder was extracted with 100 mL of Milli Q water on boiling water bath for 30 minutes and were filtered with Whatmann no. 1 filter paper. An aliquot of 10 mL of aqueous plant extract was titrated with 100 mL of 5 mM ZnSO₄.5H₂O to reduce ZnO NPs at 50 C for 2 hours. The obtained mixture was centrifuged at 10,000 RPM for 15 minutes to separate agglomerated, broad-sized particles and plant admixtures. X-Ray Diffractometric (XRD). The morphology was monitored by a scanning electron microscope (SEM). Chemical properties were investigated by Fourier transform infrared spectroscopy

ZnO nanoparticles (ZnO NPs) by sol-gel method according to the method that is mentioned in paper.⁶

Preparing the Dental Filling Samples

The nanoparticles (inorganic phase) that were prepared in 2.1 were added as fillers to the base material (the organic phase), the dental filling “COMPOSAN LCM”.

The new mixture from the organic phase and inorganic phase is called composite. It is a resin-based composite.

This composite with three ratios of the fillers was then put in templates to be cured by light with three periods of exposure 10 seconds, 15 seconds, and 20 seconds except with silver filler, where they were cured at 10, 20, and 30 seconds.

The light that was used in this work is LED. The composite samples that were done were characterized by several tests as follows.

Characterizations of the Samples Prepared

The composite resin samples prepared were examined by the depth of cure, compression strength, upper surface hardness, lower surface hardness, degree of cure, the relation between degree of cure and depth of cure, diameter of samples, and polymerization shrinkage tests.

RESULTS AND DISCUSSION

Results of Compression Strength (Σ) Test

To achieve this examination, resin composite mixture samples prepared were template in cylindrical template of 3mm diameter and 6 mm thickness and then cured by LED. The samples produced were then tested by “micrometer controlled electronic universal testing machine”. All the samples and tests obey the criteria (ISO 9917).

From Figure 1, it seems that compression strength (σ) increases with increasing light time; increasing light exposure time increases monomers involved in the organic phase converted to polymers and enhances polymerization degree,¹⁰ and it could be seen that when filler load increases, composite compression strength increases,¹ that is because the filler is inorganic solid material.¹²

Results of Upper Surface Hardness Test

Composite samples were template in a cylindrical template with 6 mm diameter and 2 mm height to do this test. After samples had been cured by LED they were tested by PS-2006 VIDRO MEASURING DEVISE; the model of the device is TH-717 digital micro hardness.

From the figures above, it can be concluded that upper surface hardness increases with increasing filler ratio. The reason for that is the solidity of the fillers¹¹. Besides that, the fillers are nanomaterials, and seldom does nanomaterial include inter distances between particles; this increases solidity, and thus upper surface hardness increases with increasing filler ratio.¹³

Results of Diameters (D) Samples

Diameter (D) in mm of samples was calculated to find polymerization shrinkage. The template that was used to do this test is a 6x8mm cylinder. It was assumed that the diameter of sample before curing is D₁. D₁ was equal to 6 mm for all samples. The diameter of sample after curing is D₂. The results of diameter after curing (D₂) are displayed in the figures below:

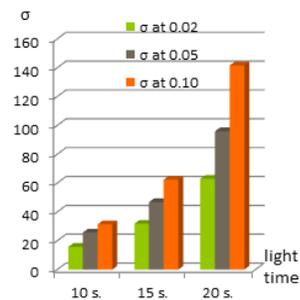


Figure 1: compression strength (σ) in MPa values of ZnO filler-containing resin composite Samples in three exposure times of light at the filler ratios 0.02, 0.05, 0.10 of ZnO.

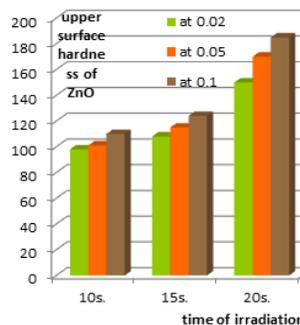


Figure 2: values of upper surface hardness in MPa of ZnO filler-composite samples at the ratios 0.02, 0.05, and 0.01 versus LED exposure time.

From Figure 3, it could be seen that the diameter of the samples increases with increasing light exposure time; the reason is when light time increases, more monomers convert to polymers; thus, polymerization degree enhances. It could also be seen from the figures that are increasing filler load increases the diameter of the samples, i.e., increasing filler ratio improves the polymerization process.

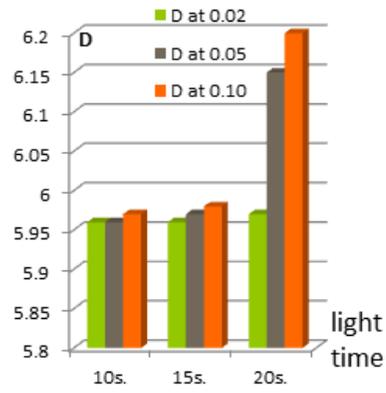


Figure 3: diameter of the samples containing ZnO filler at the ratios 0.02, 0.05, and 0.1 versus LED period time.

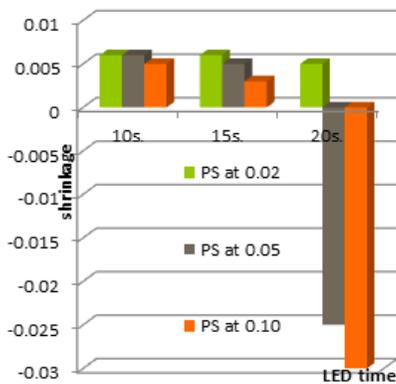


Figure 4: Polymerization shrinkage values of ZnO filler-containing composite resin at ZnO ratios 0.02, 0.05, 0.10.

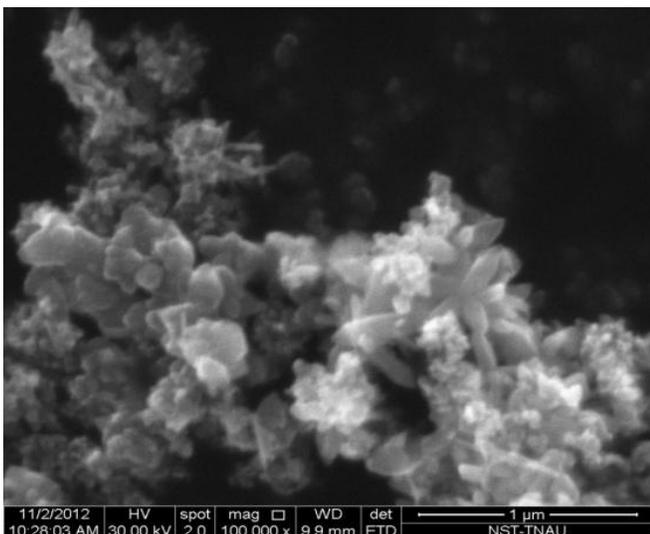


Figure 5: SEM images of ZnO

Results of Polymerization Shrinkage (PS) Test

Polymerization shrinkage (PS) is defined as a reduction in the size of a sample after it is cured. Polymerization shrinkage was measured by the equation 1:

$$PS = (D_1 - D_2) / D_1 \quad (1)$$

Where

PS is polymerization shrinkage.

D_1 is diameter of sample before curing.

D_2 is diameter of sample after curing¹⁴

The minus sign means that sample expands, while the positive sign means sample contract. From Figure 4, it could be concluded that increasing light exposure duration reduces polymerization shrinkage because more monomers will convert to polymers; thus, polymerization degree enhances.¹⁵

It could also be seen from these figures that increasing filler load reduces PS; that agrees with^{11,16-18} and means that these fillers at these ratios enhance polymerization process thus improve restorative dental filling.

The best ratio made the dental filling expanded with silver filler is at 0.0015.

From the figures above, it can be concluded that there is no polymerization shrinkage with Nano silver filler at most of its ratios; besides that, the mechanical properties are much better than other fillers.

Lanceolated nanoscaled rods measuring 50–80 nm diameter; appeared to be radiating from a central core

Rod-shaped fused at the centre to form a radiating structure as observed in SEM

Characterization of Nanoparticles (ZnO)

The surface morphology of Zinc Oxide (ZnO), nanoparticles was examined under SEM, TEM, Particle Size Analyzer, and Raman Spectroscopy. The morphology of different



Figure 6: TEM images of ZnO

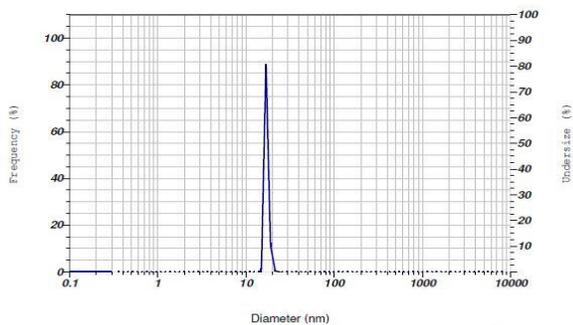


Figure 7: Particle analyzer average size and intensity distribution of ZnO nanoparticles

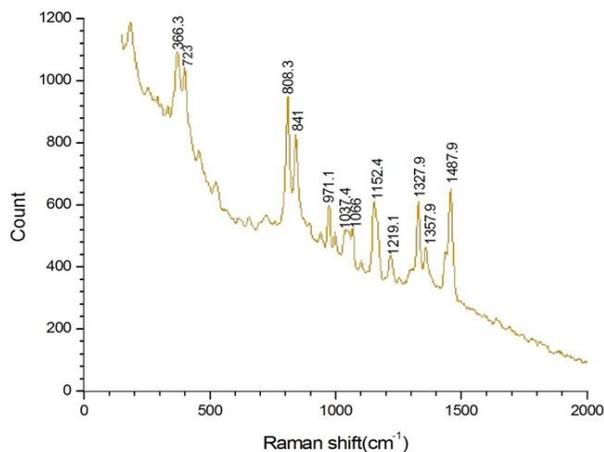


Figure 8: Raman spectra of ZnO

nanoparticles observed is presented below. The particle size analyzer was used to analyze the size of the particle using laser scattering principle for estimating the average particle size and distribution pattern for synthesized ZnO nanoparticles. The particle size distribution of ZnO was found to be 16 nm (Figure 6).

Raman spectroscopy was employed to identify the chemical composition and confirm the four nanoparticles synthesized by observing the peaks. The peaks were observed at 366, 723, 1066, and 1219 cm^{-1} for ZnO nanoparticles, confirming the respective chemical compounds Figure 8.

In this study, the ZnO nanoparticles were successfully synthesized by the direct precipitation method using zinc nitrate as zinc source and KOH as a precipitating agent in an aqueous solution. The size range of the generated ZnO powder was approximately 20–40 nm. In summary, we have successfully designed a facile and fast synthesis route to produce ZnO nanoparticles, and finally, ZnO nanoparticles were characterized by UV-visible, TEM, and DLS analysis.

CONCLUSIONS

The most important conclusion in this work is that Nanosilver solves polymerization shrinkage problem of polymeric (light-cured) dental filling and cancels it, and polymeric or light-cured dental filling will not fall.

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