SYNTHESIS OF ZINC OXIDE (ZnO) AND STUDY ON MECHANICAL PROPERTIES OF POLYMERIC DENTAL FILLING

Sanaa A. Alsahib[1], Khalida A. Thejeel [2 *]

[1]Department of Chemistry, College of Science for women, Baghdad University, Iraq.
[2]Department of Geophysics, College of Remote Sensing and Geophysics, Alkarkh University for Science, Iraq.

*E-mail: khalidathejeel@gmail.com

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 ZnO, by sol-gel, laser ablation, chemical, and sol-gel methods according to papers published earlier. These nanoparticles were then added to 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, relation between depth of cure and degree of cure, diameter of samples, and polymerization shrinkage. The examinations and results showed that polymeric dental filling containing silver nanoparticles has maximum values of compression strength and hardness and no polymerization shrinkage. The most important result was that **ZnO** nanoparticles filler cancelled polymerization

Key words: laser, nanoparticles, light-cured, polymer, and dental filling.

INTRODUCTION

Using resin composite in treating teeth began when Joseph Redenbacher discovered acrylic acid and used acrylic resin as a dental restoration filling[1] Afterwards, poly methyl 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 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 fluid state, but when it is cured by a light, the monomers convert to polymer where polymer is in solid state. Cure process is transformation of fluid material to solid material.

Light types used in 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.

Polymerization process is 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.

Aim of this work

The aim of this work is to improve polymeric dental filling by nanoparticles.

MATERIALS AND METHODS

The base material that was used in this work is the polymeric dental filling "COMPOSAN LCM" which has 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 min 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 $ZnSO_4.5H_2O$ for reduction of ZnO NPs at 50 C for 2 h. Obtained mixture was centrifuged at 10,000 RPM for 15 min to separate agglomerated, broad sized particles as well as plant admixtures. X-Ray Diffractometric (XRD). The morphology was monitored by 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 organic phase and inorganic phase is called composite. It is 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 depth of cure, compression strength, upper surface hardness, lower surface hardness, degree of cure, 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 6mm 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).

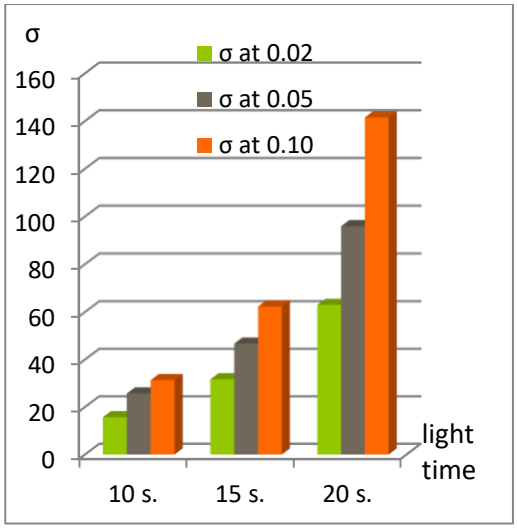

Figure 1: compression strength (σ) in MPa values of ZnO filler-containing resin composite Samples in three exposure times of light at the filler ratios 0.02, 0.05, 0.10 of ZnO.

From the 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 filler is inorganic solid material [12].

Results of upper surface hardness test:

To do this test, composite samples were template in cylindrical template with 6mm diameter and 2mm height. 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.

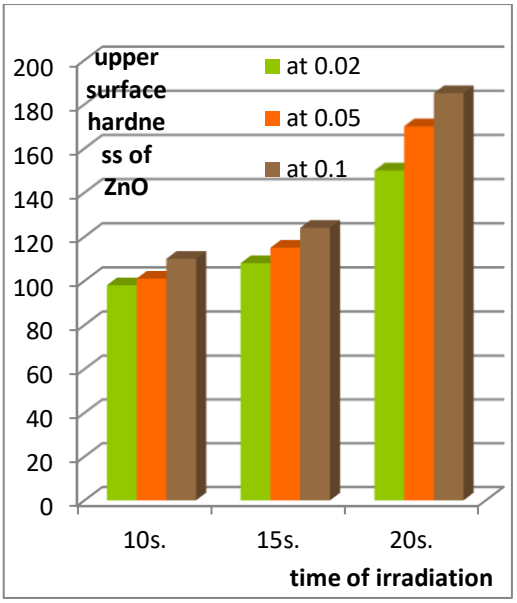

Figure 2: values of upper surface hardness in MPa of ZnO filler-composite samples at the ratios 0.02, 0.05, and 0.01 versus LED exposure time.

From the figures above, it can be concluded that upper surface hardness increases with increasing filler ratio. The reason of that is the solidity of the fillers[11]. 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:

To calculate polymerization shrinkage, diameter (D) in mm of samples was calculated. The template that was used to do this test is 6x8mm cylinder. It was assumed that diameter of sample before curing is D_1. D_1 was equal to 6mm for all samples. Diameter of sample after curing is D_2. The results of diameter after curing (D_2) are displayed in the figures below:

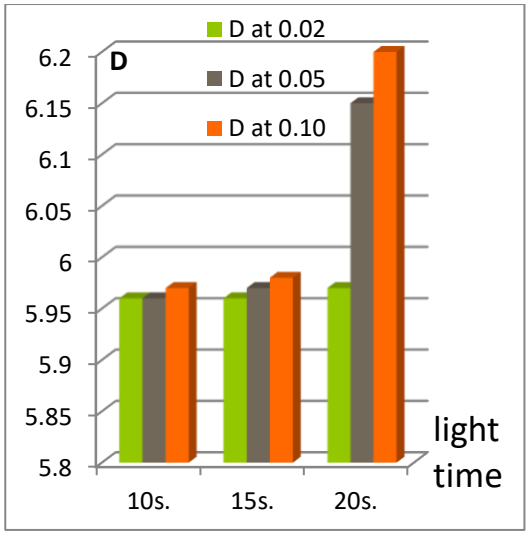

Figure 3: diameter of the samples containing ZnO filler at the ratios 0.02, 0.05, and 0.1 versus LED period time.

From the figure 3 it could be seen that 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 increasing filler load increases diameter of the samples, i.e., increasing filler ratio improves polymerization process.

Results of polymerization shrinkage (PS) test:

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

$$PS = \frac{D1-D2}{D1} \text{ ---------- (1)}$$

Where

PS is polymerization shrinkage.

D_1 is diameter of sample before curing.
D_2 is diameter of sample after curing [14]

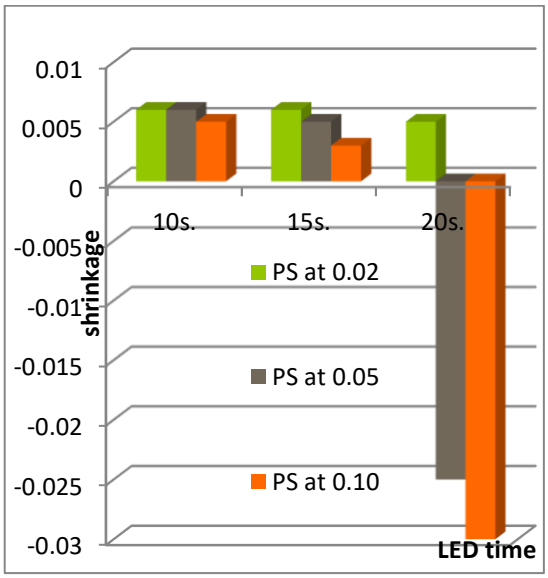

Figure 4: Polymerization shrinkage values of ZnO filler–containing composite resin at ZnO ratios 0.02, 0.05, 0.10.

The minus sign means that sample expand, while the positive sign means sample contract. From the figures 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 [16,11,17,18] and means that these fillers at these ratios enhance polymerization process thus improve dental restorative 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; beside that, the mechanical properties are much better than other fillers.

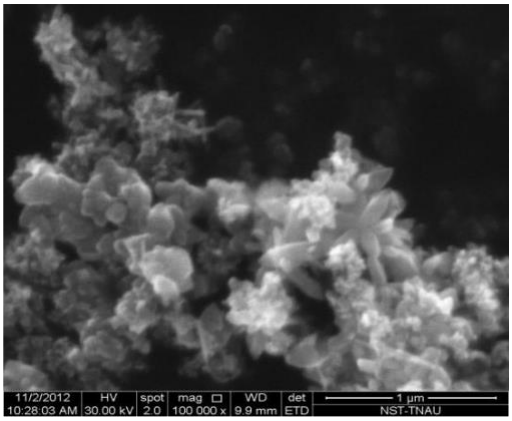

Figure 5*:* SEM images of ZnO
Lanceolated nanoscaled rods measuring 50-80 nm diameter; appeared to be radiating from a central core

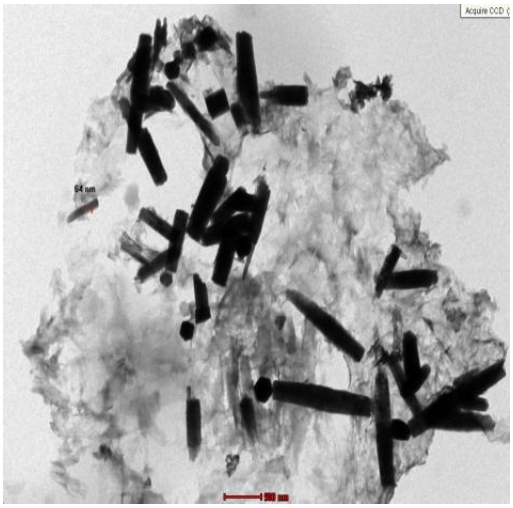

Figure 6*:* TEM images of ZnO
Rod shaped fused at centre to form a radiating structure as observed in SEM

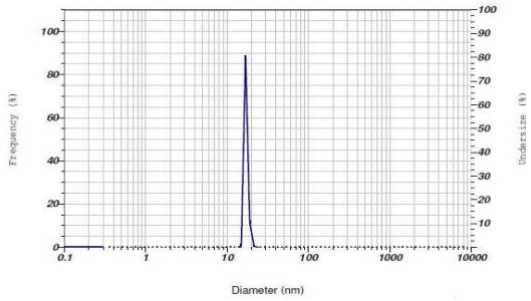

Figure 7*:* Particle analyzer average size and intensity distribution of ZnO nanoparticles

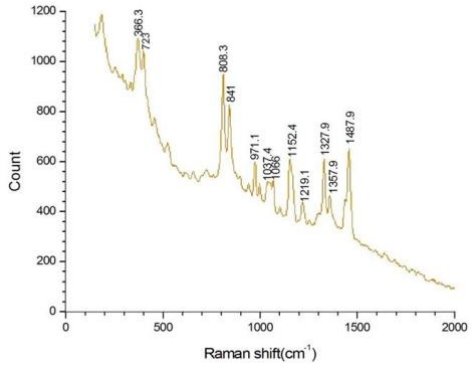

Figure 8: Raman spectra of ZnO Characterization of Nanoparticles (ZnO)

The surface morphology of Zinc Oxide (ZnO), nanoparticles were examined under SEM, TEM, Particle Size Analyzer and Raman Spectroscopy. The morphology of different nanoparticles observed are 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 to confirm the four different nanoparticles synthesized by observing the peaks. The peaks were observed at 366, 723, 1066 and 1219 cm-1 for ZnO nanoparticle confirming the respective chemical compounds Figure 8.

In this study, the ZnO nanoparticles were successfully synthesized by direct precipitation method using zinc nitrate as zinc source and KOH as precipitating agent in 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 Nano silver 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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