



# A Study of the Physical Properties of Nanoscale Dental Fillings

Faten. D. Mirjan <sup>1\*</sup>, Mohammed A. AL Shareefi <sup>2</sup>

<sup>1</sup>College of Science, University of Babylon, [faten.mirjan.scihigh71@student.uobabylon.edu.iq](mailto:faten.mirjan.scihigh71@student.uobabylon.edu.iq), Babil, Iraq.

<sup>2</sup>College of Science, University of Babylon, [sci.mohammed.abdul@uobabylon.edu.iq](mailto:sci.mohammed.abdul@uobabylon.edu.iq), Babil, Iraq.

\*Corresponding author email: [faten.mirjan.scihigh71@student.uobabylon.edu.iq](mailto:faten.mirjan.scihigh71@student.uobabylon.edu.iq); mobile: 07830853294

## دراسة الخصائص الفيزيائية لحشوات الأسنان النانوية

فاتن ضياء مرجان\*<sup>1</sup>، محمد عبد الامير الشريفى<sup>2</sup>

<sup>1</sup>كلية العلوم، جامعة بابل، [faten.mirjan.scihigh71@student.uobabylon.edu.iq](mailto:faten.mirjan.scihigh71@student.uobabylon.edu.iq)، صندوق بريد، بابل، العراق

<sup>2</sup>كلية العلوم، جامعة بابل، [sci.mohammed.abdul@uobabylon.edu.iq](mailto:sci.mohammed.abdul@uobabylon.edu.iq)، بابل، العراق

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### ABSTRACT

#### Background:

A compound is defined as the substance that arises from the mixing of two different substances, and the new materials produced have new properties that differ from the properties of the primary materials. The material produced has a cohesive structure. A compound consists of two basic components, the base material called the "matrix" and the additive called the "filler". The matrix is a polymer, and the filler is different from the type of polymer; it is an inorganic substance like minerals. The filler within the crystalline medium (matrix) is randomly distributed; this occurs because the particle size of the two materials (polymer and filler) are nearly equal and separate when the size of the filler particles is small relative to the size of the polymeric particle.

#### Materials and Methods:

The mechanical (hardness and compressive strength) and ultrastructural properties were measured by atomic force microscopy (AFM) for different concentrations of the added nanomaterials are (0, 2.5, 5 and 7.5) wt%. With the casting method, to be used to prepare samples of structural properties, special molds have been used to examine the mechanical properties (hardness and compressive strength), through which the surface can be viewed with high precision very small (100  $\mu$ m to less than 1  $\mu$ m). Compressive strength test is important. It is very important for dental materials as these materials must have mechanical properties similar to the teeth in order to resist chewing forces and it is compressive in a force range of (450-500) newtons. The compressive strength test is very important for dental materials as these materials must have mechanical properties, for the purpose of measuring the compressive strength, cylindrical samples with a diameter of (3 mm) and a thickness of (6 mm).

#### Results:

The (AFM) images showed the surface shape of the nanopolymer (PMMA) before and after the addition of nanomaterials, where we notice in a clear picture the increase in the roughness, the mean root value (RMS) and the average particle size. After adding the oxides, and increasing the added proportions, since the roughness when adding nano-oxides is useful for tooth adhesion, and despite the difference in (roughness) values, the values are still less than (200 nm), which was reported as an initial point for the accumulation of bacterial plaques and the risk of incidence of caries and periodontitis, and it can be assumed that the surfaces evaluated in this study have a smooth surface. Which does not represent any risk of scale build-up. We also notice when studying and examining the mechanical properties of hardness, as hardness is the surface resistance to cutting and scratching, as we notice an increase in hardness with increasing concentrations of nanomaterials. The compressive strength test of the nanofilling was also carried out before and after the addition of nanomaterials, as we notice an increase in the amount of compression with the increase in the percentage of addition of nanomaterials as well. These oxides are very safe and harmless to the human body.

**Conclusion:**

Samples of novel nanocomposites (PMMA-ZnO-MgO) for dental filling applications have been synthesized. The addition of nanomaterials to the pure polymer leads to an increase in the surface roughness, as well as the roughness rate, the average root value (RMS), and the average grain size with an increase in the proportions of impurities, which leads to an increase in the cohesion and hardness of the filling, as the increase in agglomerations leads to an increase in the cohesion of the filling with the teeth and its durability. While the higher the added percentage, the roughness increased, as we found the highest roughness at (7.5)%, which reached (34.72 nm). Therefore, the filler achieved clear structural properties and a radical change in the structure of the polymer used as a result of the addition of nanomaterials, in addition to durability, quality, safety, and suitability for human tissues. The study showed the mechanical properties and proved that the material (MgO) containing the nano-filling has resistance to the compressive strength test, as it reached the highest resistance to the compressive strength of the addition ratio (7.5)%, which amounted to (150 MPa), while the hardness reached (90.7 MPa) at (7.5%), where The physical properties increase with the increase in the percentage of added nanomaterials.

**Key words:** PMMA, ZnO, MgO, Mechanical Properties, Structural Properties, Dental Fillings.

**الخلاصة****مقدمة:**

يُعرف المركب بأنه المادة التي تتشأ من خلط مادتين مختلفتين، والمواد الجديدة المنتجة لها خصائص جديدة تختلف عن خصائص المواد الأولية. المواد المنتجة لها هيكل متماسك. يتكون المركب من عنصرين أساسيين، المادة الأساسية تسمى "المصفوفة" والمواد المضافة تسمى "الحشو". المصفوفة عبارة عن بوليمر، والحشو يختلف عن نوع البوليمر؛ وهي مادة غير عضوية مثل المعادن. يتم توزيع الحشو داخل الوسط البلوري (المصفوفة) بشكل عشوائي؛ يحدث هذا لأن حجم جزيئات المادتين (البوليمر والحشو) متساويان تقريباً ومنفصلان عندما يكون حجم جزيئات التعبئة صغيراً بالنسبة إلى حجم الجسيمات البوليمرية.

**طرق العمل:**

تم قياس الخصائص الميكانيكية (الصلابة وقوة الانضغاط) وخصائص البنية التحتية بواسطة الفحص المجهرى للقوة الذرية (AFM) لتركيزات مختلفة من المواد النانوية المضافة هي (0، 2.5، 5 و 7.5%) بالوزن. مع طريقة الصب، لاستخدامها في تحضير عينات من الخصائص الإنشائية، تم استخدام قوالب خاصة لفحص الخواص الميكانيكية (الصلابة وقوة الانضغاط). في حين أن مجهر القوة الذرية (AFM) هو نوع عالي الدقة من تقنيات الفحص المجهرى، والذي من خلاله يمكن رؤية السطح بدقة عالية جداً (100 ميكرومتر إلى أقل من 1 ميكرومتر). يستخدم (AFM) لتقنية التصوير السطحي للحصول على معلومات حول التشكل السطحي للفيلم، مثل التوزيع وتوحيد الحبوب والخشونة التي تتشكل على السطح، يتم تحديد العديد من المواد بهذه التقنية، بما في ذلك البوليمرات وأشباه الموصلات والمعادن والمركبات. اختبار قوة الانضغاط مهم للغاية بالنسبة لمواد طب الأسنان حيث يجب أن يكون لهذه المواد خواص ميكانيكية مماثلة للأسنان من أجل مقاومة قوى المضغ وهي قابلة للضغط في نطاق قوة (450-500) نيوتن. اختبار مقاومة الانضغاط مهم جداً لمواد طب الأسنان حيث يجب أن يكون لهذه المواد خواص ميكانيكية مماثلة للأسنان لغرض قياس مقاومة الانضغاط، تم تحديد العينات الأسطوانية بقطر (3 مم) وسماكة (6 مم).

**الاستنتاجات:**

تم تصنيع عينات من المركبات النانوية الجديدة (PMMA-ZnO-MgO) لتطبيقات حشوات الأسنان. تؤدي إضافة المواد النانوية إلى البوليمر النقي إلى زيادة خشونة السطح وكذلك معدل الخشونة ومتوسط قيمة الجذر (RMS) ومتوسط حجم الحبوب مع زيادة نسب الشوائب مما يؤدي إلى زيادة التماسك والصلابة الحشو حيث أن زيادة التكتلات تؤدي إلى زيادة تماسك الحشوة مع الأسنان ومتانتها. كلما زادت النسبة المضافة زادت الخشونة حيث وجدنا أعلى خشونة عند (7.5)% التي بلغت (34.72nm). لذلك حقق الحشو خصائص هيكلية واضحة وتغيراً جذرياً في بنية البوليمر المستخدم نتيجة إضافة المواد النانوية بالإضافة إلى المتانة والجودة والسلامة والملاءمة للأنسجة البشرية. أظهرت الدراسة الخواص الميكانيكية وأثبتت أن مادة (MgO) المحتوية على حشوة النانو لها مقاومة لاختبار قوة الانضغاط حيث بلغت أعلى مقاومة لقوة الانضغاط لنسبة الاضافة (7.5)% التي بلغت (150 MPa) أما الصلابة فقد وصلت إلى (90.7 MPa) عند (7.5)% حيث تزداد الخصائص الفيزيائية بزيادة نسبة المواد النانوية المضافة.

**الكلمات المفتاحية:** بولي ميثاكريليت، أكسيد الزنك، أكسيد المغنيسيوم، الخصائص الميكانيكية، الخصائص التركيبية، حشوات الاسنان.



## INTRODUCTION

The most prevalent dental illness in humans is caries, sometimes known as tooth decay. It is probably the most prevalent illness in modern culture. Plaque, a yellowish film that forms on teeth and has a propensity to harbor germs, is the primary cause of tooth decay. The bacteria that inhabit plaque break down the sugar and starchy food waste into acids that erode the calcium and other minerals from the enamel and dentine of the teeth[1]. Caries typically starts on the surface enamel, notably in fissures and pits and between teeth. The decay process begins in the enamel and moves from there to the underlying dentine and, in some cases, the tooth pulp. In addition to keeping teeth clean by routine brushing and flossing, fluoridating water can significantly minimize tooth decay. Dental tissue that has deteriorated from caries is removed and replaced with filling **materials** [2]. The class of materials known as polymeric nanocomposites, which consists of organic polymers and inorganic nanoparticles, has attracted a lot of interest recently[3]. Applications for nanocomposites in microelectronic packaging, healthcare, transportation, optical integrated circuits, drug delivery, injection-molded products, sensors, membranes, aerospace, packaging materials, coatings, fire-retardants, adhesives, consumer goods, etc., are quite promising. These cutting-edge nanocomposites have a number of benefits, including reduced production costs and the ability to fabricate devices on expansive and flexible substrates. Creating nanocomposites may be a way to suitably alter the optical, electrical, thermal, and mechanical properties of individual nanomaterials[4]. The medical use of nanotechnology is known as nanomedicine, and it is a relatively young area of science and technology for illness diagnosis, treatment, monitoring, and control[5]. Nanomedicine includes anything from biological devices and nanomaterials used in medicine to nanoelectronic biosensors[6]. Both in vivo and in vitro biomedical research and applications can benefit from the usage of nanomaterials. As a result, diagnostic instruments, analytical tools, physical therapy applications, and drug administration have all been developed as a result of the combination of nanomaterials and biology[7]. In the near future, nanomedicine aims to provide a helpful collection of research instruments and clinically practical gadgets. According to the national nanotechnology plan, the pharmaceutical sector will soon see new commercial uses for enhanced drug delivery systems and in vivo imaging [8,9].

## Materials and Methods

### Method of preparation

The polymer is used in this work is poly-methyl-methacrylate (PMMA): it was obtained as powder form and could be obtained from local markets with high purity (99.8)%. Zinc oxide nanoparticles (ZnO) used as powder with particle diameter (20-30) nm from EPRUI. company and high purity (99.9)%. **Magnesium** oxide (MgO): used as powder with particle diameter (20-30) nm from EPRUI. company and high purity (99.9%). The method of work depends on the two manufactured fillings on the base material, which is a polymer (PMMA) **which** is dissolved in Chloroform or Chloromethane, which is an organic compound with chemical formula  $\text{CHCl}_3$ , which is a colorless liquid that is easy to volatilize. It is also a very good solvent for various chemicals with a density of (1.49) g/cm<sup>3</sup>. Where the polymer is dissolved in 30 ml of Chlorofoam



with continuous movement for (30) minutes. Following dissolving, the oxides (Fillers) **were** added from (ZnO, MgO) while stirring with a magnetic device for ten minutes to create a more uniform solution at ambient **temperature with** varying weight percentages of (0, 2.5, 5 and 7.5). Samples of (PMMA-ZnO, MgO) nanocomposites **were** prepared using the casting process in a petri dish with a 10 cm diameter. The thickness range of the prepared samples was (0.011-0.0105) m, and a digital micrometer was utilized to measure the thickness. The atomic force microscope (AFM) is a high-precision scanning probe microscopy technology that allows for a very accurate surface examination from (100 m to less than 1 m) [10]. The percentage of zinc oxide by weight is displayed in the table below (1). After casting, samples were analyzed by removing sections of the material.

**Table 1. Weight percentages for nanocomposites (PMMA- ZnO - MgO)**

wt. %	PMMA g	ZnO g	MgO g	Weight of Sample
0	1	0	0	1gm
2.5	0.975	0.0125	0.0125	
5	0.95	0.025	0.025	
7.5	0.925	0.0375	0.0375	

Another measurement In the present research, two different mould types constructed of stainless steel were employed [11]. Which the first model consists of a cylindrical cavity of an outside (6)mm in diameter and(10) mm in thickness. To prepare samples with a thickness of (2) mm, an iron rod has a thickness of( 6) mm and a diameter of (8) mm is put into cavity. This method is used to get samples ready for assessing their hardness. The second model, created similarly to the first, is a cylindrical cavity with a thickness of (6) mm and a diameter of (3) mm that is used to prepare samples for compressive strength. Compressive strength is generally defined as the material's maximum resistance to the applied stress before it is crushed and is affected by several factors, including the components of the composite resin, especially the filler particles, as it is greatly affected by the size of the fillers, their diameter and type [12]. The compressive strength test is crucial for dental materials because they must have mechanical properties similar to those of teeth in order to withstand chewing forces. Dental materials such as composite resin, amalgam, cement, and other brittle materials resist compressive forces more than tensile forces. This has a strength range of (450-500)N and is compressive. The compressive strength test is the best method for comparing dental composites because, as long as the chewing forces are compressive in character, it is crucial to understand how dental filling materials behave under these circumstances.



According to (ISO 9917) [13], in order to perform the compressive strength test for dental overlays, the models must be cylindrical in shape and have a length to diameter ratio of (2:1) because increasing this ratio will result in the model bending in an unfavorable manner. The compressive strength is calculated using the equation. [1].

$$\sigma = P/A \dots\dots\dots(1).$$

where :

:  $\sigma$  the compressive strength measured in (N/mm<sup>2</sup>).

: P the applied load measured in ( N).

A: Loading area in unit (mm<sup>2</sup>)

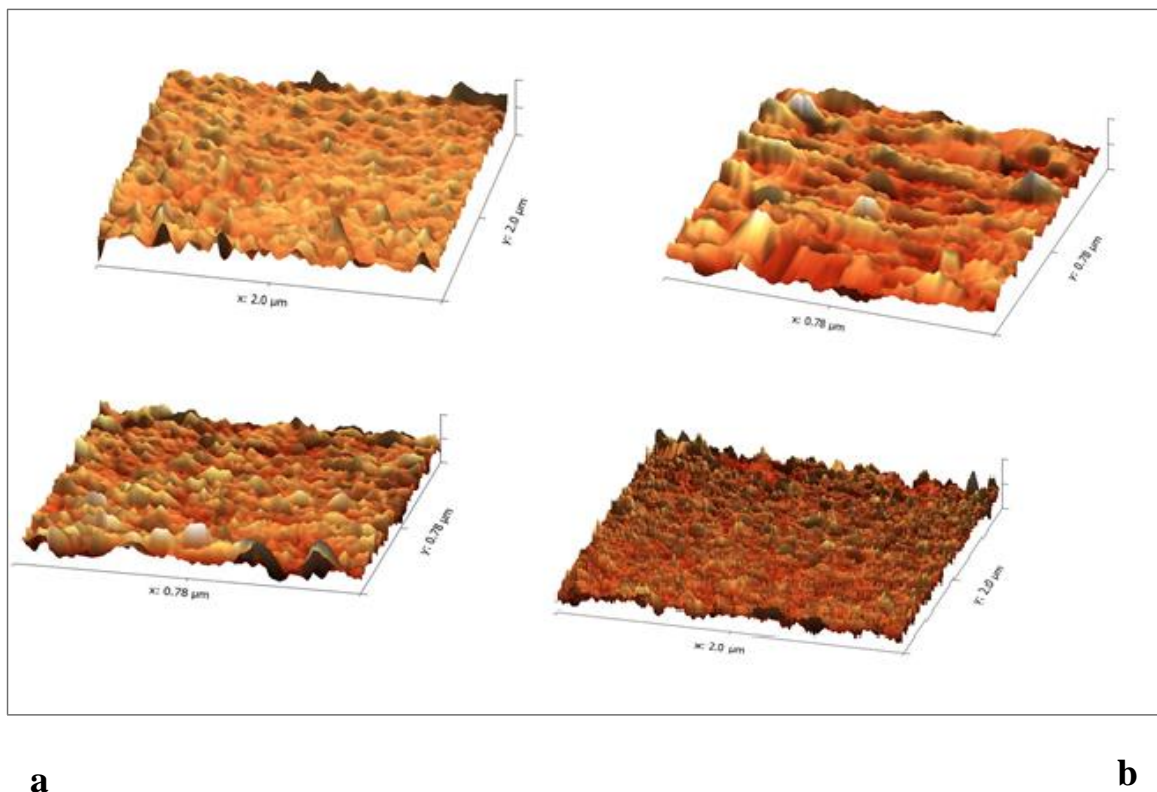
## Results and Discussion

Figure (1) shows the external surface of the proposed samples for dental nanopolymer fillings before and after adding the used concentrations. **Table (1)** shows the percentage by weight of zinc oxide by weight. After casting, samples were removed, and they were examined using atomic force microscopy (AFM), which produces three-dimensional images with a million times greater resolution. AFM uses a laser beam refractor to monitor the surface's shape and features, while the movement of the laser beam tracks the terrain. This leads to an increase in the number of molecules per unit volume, which leads to the loss of the energy of the molecules in an amount sufficient to form the molecular agglomerations that reach the surface in the form of small granules and begin to grow in the form of scars or meanders in order to ensure the strength, durability and stability of the filling in the tooth, as the increase in roughness It should be less than (200) nm, which is the initial point of bacterial plaque accumulation and the risk of caries and periodontal infections, so it can be considered that these surfaces do not represent any risk of plaque accumulation as shown in the table below (2) [16]. The higher the roughness rate, the mean root value (RMS) and the average grain size with the greater the proportions of impurities, as the appearance of agglomerates when nano-oxides are added is beneficial for dental adhesion and packing durability. Where we **observed** a homogeneous distribution of molecules and obtain improved properties and improve the quality of the polymer strength and toughness and increase the stacking and this leads to the improvement of the crystal structure by increasing the nano-oxides. These results are similar to what the researcher reached [17].



**Table 2.(AFM ) parameters for nanocomposites (PMMA- ZnO - MgO).**

Sample	wt%	RMS	Roughness	Average Diameter
PMMA Pura	0	7.08	7.33	20.98
PMMA-ZnO-MgO	2.5	18.67	17.80	26.92
PMMA-ZnO-MgO	5	28.31	20.63	32.79
PMMA-ZnO-MgO	7.5	30.55	34.72	38.85



**Figure 1. AFM 3D image of the filling before and after the addition . (a) PMMA<sub>pure</sub> ,(b) (PMMA- ZnO - MgO) 2.5 wt.% , (c) (PMMA- ZnO - MgO) 5 wt.% , (d) (PMMA- ZnO - MgO) 7.5 wt.%**



Compressive strength ( $\sigma$ ) is the most important feature of dental fillings. The compressive strength test is important in laboratory analyzes, which are usually considered good indicators for simulating the forces that fillings are exposed to during chewing in the mouth. Therefore, dental fillings must have a high value of compressive strength to withstand the resulting external forces. through chewing [18]. All samples were subject to the quality standard (ISO.9917). The length-to-diameter ratio (2:1) used in these samples and the stroke speed (0.5 cm/m) are appropriate in these standards [19]. In this study, the compressive strength was calculated and the results for fillings containing (PMMA-ZnO-MgO), the highest value of compressive strength was (150 MPa) at (7.5% g) gradually to (146 MPa) at (5% g) and (138 MPa) at (2.5% g). As the compressive strength increases with the increase of these nanomaterials, they are effective in enhancing the compressive strength of the fillings due to the large mechanical properties of each of them. It is interesting to note that the addition of (MgO) have its molecules have strength and toughness resulting through its hexagonal crystalline structure, the bond is able to form and modify bonds with compounds, which can lead to the arrangement of structures as a result of the existing strong bonding features such as covalent bonds and coordination covalent bonds that provide remarkable mechanical reinforcements to form the best structure for fillers. Table 3 shows that the type of composite material, the type of filling, and the size of the fillings all have an impact on the compressive strength. Thus, by combining nanomaterials and nanopolymers, the mechanical qualities are enhanced. [20,21].

**Table 3. Compressive strength( $\sigma$ ) parameters for (PMMA- ZnO - MgO) nanocomposites.**

Material	$\sigma$ (MPa)at 2.5% g	$\sigma$ (MPa)at 5% g	$\sigma$ (MPa)at 7.5% g	$\sigma$ (MPa) of PMMA
(PMMA- ZnO -MgO)	138	146	150	130

Table (4) shows the hardness values in units (Mpa) for the two prepared fillings. The hexagonal crystal system and strong bonds also play in enhancing the strength and durability of the filling, as by increasing the addition percentage, the surface hardness increases and the hardness of the filling is enhanced. Besides, rarely have distances between molecules. This increases the hardness of the materials, and thus the surface hardness increases. All of the experimental nanocomposites showed good hardness values with ISO standards (above 50 MPa)[22]. On the other hand, although the fillers are similar in size, they contain nanoparticles that have unique and different physico-chemical properties and these properties are not only a matter of particle size,



but also the qualities that these tiny sized particles cause. It has enhanced effects from the fillings it contains, which makes the surface highly scratch resistant when added [23,24].

**Table 4. Hardness parameters for (PMMA- ZnO - MgO).**

Material	Hardness (MPa) at 2.5% g	Hardness (MPa) at 5% g	Hardness (MPa) at 7.5% g	Hardness (MPa) of PMMA
(PMMA- ZnO -MgO)	84.3	86.6	90.7	82

### Conflict of interests.

There are non-conflicts of interest.

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