RESEARCH ARTICLE

Preparation and Characterization Teeth Filling of Powder and Mixing it with Acrylic (PMMA)/N-TIO_2 to Wear Resistance and Antibacterial

Fadhil K Farhan[1], Mohammed O Kadhim[2], Mohammed H Ali[3], Awatif S Abass[4]

[1-4]AL-Karkh University of Science, University in Baghdad, Iraq; 3Ministry of Science and Technology, Baghdad, Iraq

Received: 06th July, 19; Revised: 08th August, 19, Accepted: 12th September, 25; Available Online: 22th September, 2019

ABSTRACT

In this research, the light filling with titanium oxide was formed as an anti-corrosion and antibacterial antibiotic. The white acrylic powder was used with its solvent after mixing it with different percentages of biologically active titanium oxide using the liquid mixing method and the ultrasound technique to obtain a homogeneous mixture free of aggregates and then molded into special molds for the required examination. The hard surface hardness of the samples prepared using the hardness device was examined along with the test of dry sliding wear using a Pin-on-disk method, as well as the examination of the samples to resist the bacteria of tooth decay. Structural tests were performed on X-ray diffraction techniques, scanning electron microscopy techniques, and infrared technique. The results were interpreted based on the practical density of the prepared samples.

Keywords: Titanium Oxide, Wear Resistant, Ultrasonic, Pin- on- Disc, Structural Tests.

Keywords: Body mass index, Polycystic ovarian syndrome, Vitamin D deficiency.

International Journal of Pharmaceutical Quality Assurance (2019); DOI: 10.25258/ijpqa.10.3.11

How to cite this article: Farhan, F.K., Kadhim, M.O., Ali, M.H. and Abass, A.S. (2019). Preparation and Characterization Teeth Filling of PMMA/N-TIO2 to Wear Resistance and Antibacterial. International Journal of Pharmaceutical Quality Assurance 10(3): 151-157.

Source of support: Nil

Conflict of interest: None

INTRODUCTION

Heat-curing acrylic resins are frequently used in temporary prosthetic-base materials, provisional prosthesis, and removable orthodontic appliances such as retainers and functional appliances.[1,2] Polymethyl methacrylate (PMMA) is generally used as a common component of acrylic materials due to its optical properties, biocompatibility, and aesthetics.[3] However, low mechanical properties against impact, bending, and fatigue are important issues to be addressed to improve acrylic polymers properties for removable acrylic appliances and dentures.[4] The acrylic resin materials are typically low in strength, soft and fairly flexible, as well as brittle on impact, and fairly resistant to fatigue The additives could affect the toughness, microstructure and deformation behavior under the impact and flexural tests. As the fracture resistance of a denture base resin is important, many approaches have been used to strengthen acrylic resin dentures. These approaches involve the use of metal wires or plates, fibers, metal powder, or rubber toughening agents. These reinforcing methods were done to improve the mechanical properties of the denture base resins and to overcome the problem of denture fractures.[7,8] Currently, dental composites consist of two phases: the synthetic polymer matrix and inorganic fillers. They are linked together via a coupling agent (such as silane). In addition, initiators and activators are added Inorganic fillers such as quartz, colloidal silica, and silica glass containing small amounts of zirconium, strontium, and barium are the most commonly used fillers. The inorganic fillers play an important role in improving the strength and elastic stiffness.[9] On the other hand; they reduce water absorption, polymerization shrinkage, and coefficient of thermal expansion. Bonding of organic and inorganic phases by coating the fillers with a coupling agent results in the formation of a strong covalent bond. This is a key factor in terms of the good mechanical properties of dental composite. Coupling agents have two functional groups in order to link the matrix and the fillers chemically.[10] Composites with nanofillers are so-called nanocomposites.[11] Different nanofillers have been introduced to dental composites. However, in this thesis, we are introducing a novel flowable nanocomposites reinforced with TiO_2 nanotubes. Nanofillers do not scatter visible light as they shrink to a fraction of this light wavelength (0.4–0.8 µm). So, they are invisible and they can improve the optical properties of composites.[12] Kim et al.[13] produced a new bioactive bone cement that consists of HA, PMMA. It was indicated that the compressive strength of the cement was lower than the pure PMMA. Mechanical properties of acrylic base cement reinforced with HA have been studied by Serbetci et al.,[14] It was found that the addition of HA caused to increase both compressive strength and fatigue life unlike tensile strength of the cement. Wang et al.,[15] and Roeder et al.,[16] studied the effects of particle size and shape and found that smaller particle size and larger aspect ratio increased the composite mechanical

**Author for Correspondence:* missing

properties. Because few papers study the addition of nano-hydroxyapatite in PMMA, this study tries to fill this gap. In this research, mechanical properties of nano HA-reinforced PMMA cement were investigated by using three-point bending, compressive, and wear test. Chen et al.,[17] studied the effect of SiO_2 nanoparticles on hardness and wearability of artificial teeth. For this purpose, they dispersed SiO_2 nanoparticles in MMA monomer and compared this composite resin with conventionally heat-cured resin. The result showed that nanocomposite had a better hardness and wear resistance properties than conventional heat-cured resin-based materials. In this workwear resistance properties of filling artificial teeth developed from wear-resistant polymeric composites, containing calcium titanate has been studied and compared to commercial filling artificial teeth. Zehnder et al.,[18] studied the bioactive ceramics materials of the MgO, TiO_2, SiO_2, Na_2O, CaO, ZnO systems have some antimicrobial activity when suspended in aqueous solutions via the release of their ionic compounds over time. The wear rate of the test samples was assessed from the weights of the worn-out material during the wear experiments. Wear experiments were conducted under the constant applied load of 30 N for various sliding distance in the presence of artificial saliva. The weight loss of the test tooth was recorded in all the wear experiments and from which the wear rate was estimated by using the following equations.[19]

$$Wr = \Delta m / L \quad (1)$$

$$WV = \Delta m / L\rho F \quad (2)$$

Where: Δm = weight loss (%), Wr = wear rate (g/cm) , Wv = wear volume (mm3/Nm), L = Sliding distance (m), ρ = Density and F – Force (N).

$$W_{Coeff} = \boldsymbol{W_v . H_v} / LF./ LF \quad (3)$$

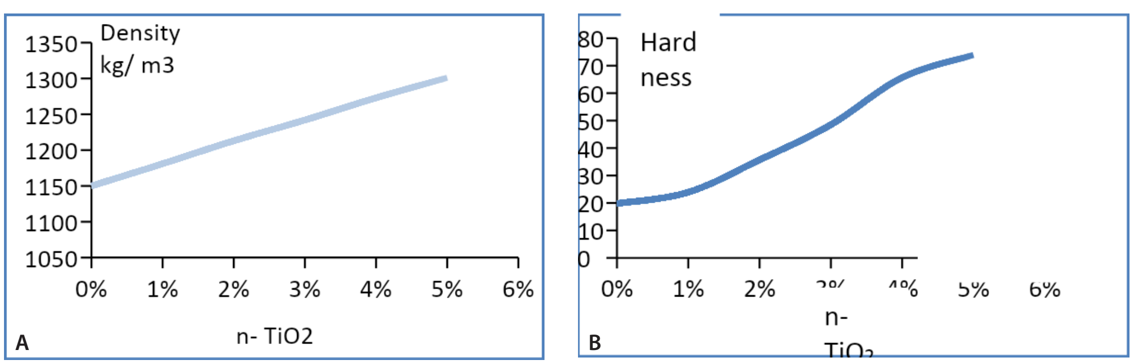
Where: Wcoeff.. – wear of coefficient and H_V – micro hardiness in MPa

EXPERIMENTAL AND MATERIALS USED

The material used in this work n- TiO_2 is a high purity powder (99%) with a granular size of 50 nm. While the polymer material is the base material in the formation of the dental fillings is (PMMA) with the material recovered its chloroform. The samples were prepared using a dry mix of titanium oxide powder and the required proportions using the effective 3-hour mechanical mixing method of 20:1 (20 small balls vs. 1 gram of mix). The solvent is then added to dissolve the polymer powder and mixed with the titanium oxide powder using the magnetic mixer for half an hour. Then the required samples are formed for examination. The Pin–on–Disc to determine the wear rate. The hardness was also checked using the Vickers hardness device, while the sample absorption of the processed, artificial saliva was tested, as shown in Table 1. Artificial saliva was prepared using different salts following the procedures described in 20, and its composition has been detailed in Table 1.

Preparation of Teeth Filling:

In this work, the toothpaste was prepared by mixing certain percentages of the prepared ceramic n-TiO_2 powder and mixing it with acrylic PMMA powder by using the grinding balls for two hours to obtain the best homogeneity between the powders and then using the chloroform solvent for the mixing process and forming the paste using the technique of ultrasonic for half an hour. The samples were then molded into standard size molds to conduct the required tests. The weight



Figures 1A and B: (A) Experimffental density as function n-TiO_2; (B) Microhardness as function n-TiO_2

Table 1(a): Chemical composition of artificial saliva

Salt	*Concentration (g.dm-3)*
NaCl	0.4
KCl	0.4
$CaCl_2.2H_2O$	0.795
$NaH_2PO_4.2H_2O$	0.78
$Na_2S.9H_2O$	0.005
$(NH4)_2CO_3$	1

Table 1(b): Experimental and surface hardness values

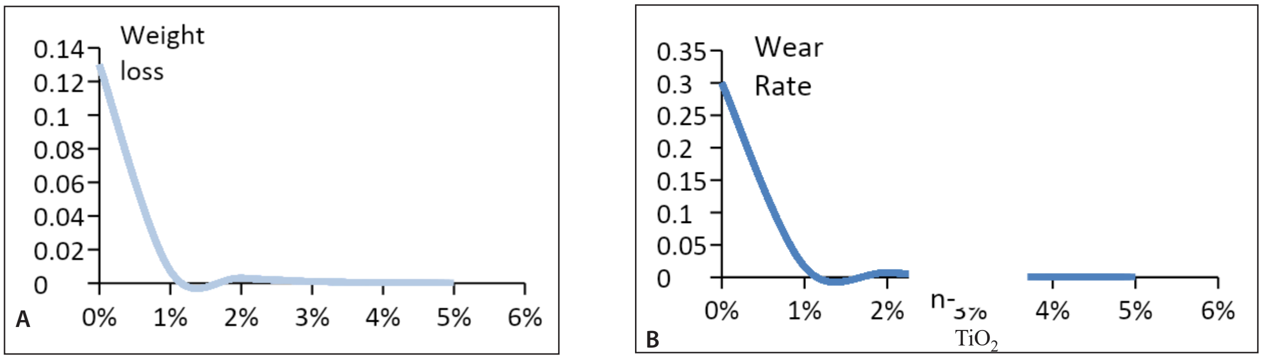
No. Samples	*Density Kg/m^3*	*Hardens MPa*
PMMA	1150	19.5
PMMA-1% (n-TiO_2)	1263	23.2
PMMA-2% (n-TiO_2)	1355	23.9
PMMA-3% (n-TiO_2)	1441	32.7
PMMA-4% (n-TiO_2)	1532	54.8
PMMA-5% (n-TiO_2)	1612	65.2

percentage ware used 1 %, 2 %, 3 % , 4 % and 5 % of the bio-ceramic powder. Five different artificial teeth filling samples were prepared, and their wear resistance was measured. For practical applications, the artificial teeth filling Require high wear resistance in saliva.

RESULT AND DISCUSSION

Surface Hardness and Density

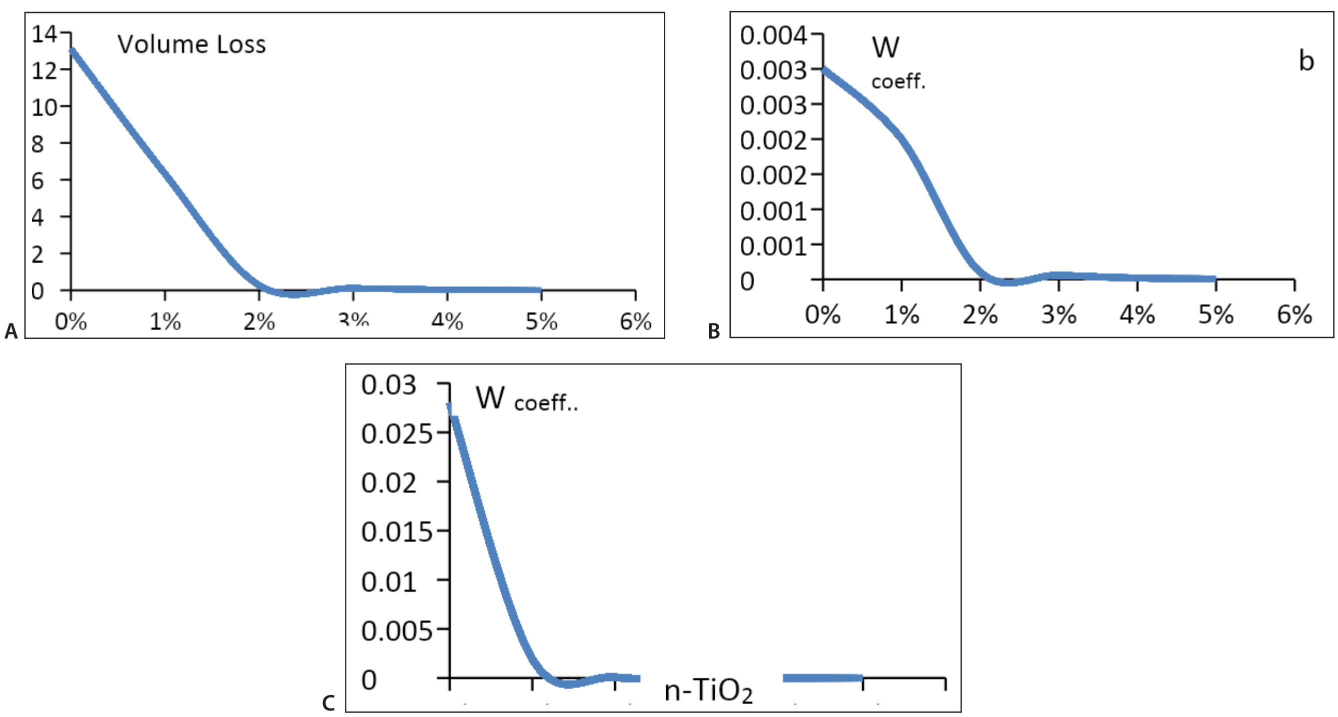
The results of the test for the prepared samples were significantly improved by increasing the weight ratio of



Figures 2A and B: (A) Weight loss a function of n-TiO$_2$; (B) Wear rate as a function of n-TiO2

Table 2: Experimental of wear rate before and after immersion

					$W_{coeff.}$		
No.	Δm	$W_r \times 10^{-6}$	$W_v \times 10^{-6}$	H_v MPa	Immersion In saliva	In air	ρ Kg/m^3
0%	0.130	0.3	13.10	19.8	0.003	0.028	1150
1%	0.0071	0.016	6.32	23.9	0.002	0.0019	1181
2%	0.0031	0.007	0.26	35.7	0.0001	0.00009	1213
3%	0.0011	0.003	0.11	48.5	0.00006	0.000057	1242
4%	0.0004	0.0009	0.03	65.7	0.00002	0.000016	1273
5%	0.00029	0.00065	0.009	73.9	0.000009	0.000011	1301



Figures 3A to C: (A) Volume loss a function of n-TiO$_2$; (B) Wear coefficient in saliva; (C) Wear coefficient in air the prepared ceramic powder. The surface hardness value improved by 70% of the non-reinforced sample by using the Vickers device. This indicates the hardness of the powder particles and bulk density improvement of the surface PMMA material. Table 1 shows the values of experimental density and surface hardness. The percentage behavior of n- TiO2 with experimental density and microhardness was shown in Figure 1. The results explained density and microhardness improvement with increases weight percentage of n- TiO2 this due to the well dispersion of nano-powder in the matrix (PMMA), decreases of porosity in all nano-composites.

WearRate (Caries)

The results of the test of the rate of wear of various types (weight loss, volume loss, and wear coefficient) showed significant improvement and high resistance to corrosion with and without industrial saliva by test the samples using the pin-on-disc technique. Was used equations (1,2 and 3) to calculator wear rate types, and this is all summarized in Table 2 and Figures 2 and 3.The wear rate measurement at applied load 20N and constant sliding distance equal 4396 m at all samples. Figures 2 and 3 show a reduction in the wear rate values of all types. This indicates the ability of the nanopowder used to strengthen friction reduction, dissipated of stored heat energy, and has high hardness while we observe that industrial fillings immersed in industrial saliva showed slight erosion compared to non-immersed filling.

Table 3: Mean values and Artificial Salvia test results mg/cm³

Sample Code	*Weight before test (M1) (mg)*	*Weight after test (M2) (mg)*	*Different In weight (mg)*	*Volume of sample cm^3*	*Sorption M2-M1/V mg / cm^3*
Neat blend	35.1	35.4	0. 30		0.07843
1%n-TiO_2	35.2	35.33	0. 10		0.02614
2%n-TiO_2	38.3	38.42	0.12	3.825	0.03137
3%n-TiO_2	36	36.3	0. 30		0.078431
4%n-TiO_2	35.6	36.0	0. 40		0.0145751
5%n-TiO_2	35.3	35.3	0		0

Artificial salvia sorption test

Table 3 shows the mean values, and sorption of artificial Salvia test results, the test showed no significant difference in water sorption mean value after addition of 5% (n-TiO_2) nanofillers. A bar chart of artificial Salvia sorption is plotted to show the difference between mean values of neat blend and experimental value shown in Figure 4. It is recommended that the rate of

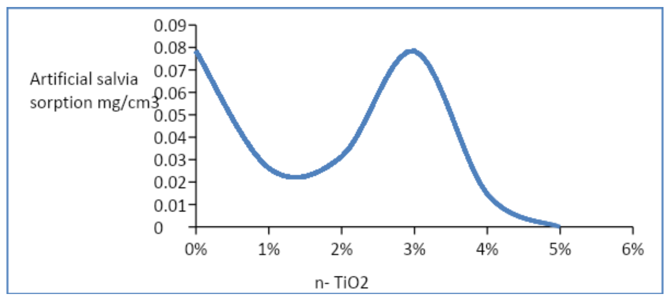
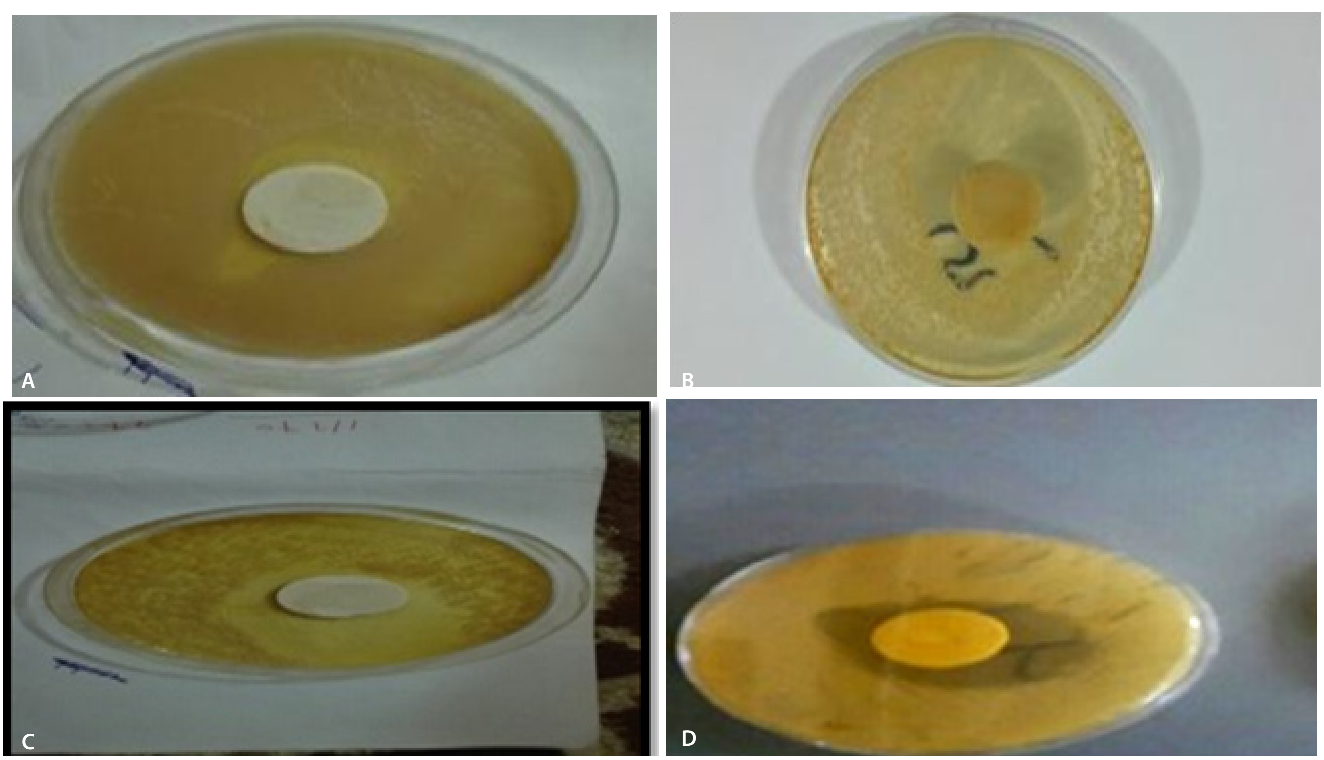


Figure 4: Artificial salvia sorption tests a function of n-TiO_2



Figures 5A to C: Images of artificial Teeth filling results of the antibacterial test. (A) PMMA; (B) 1% n-TiO_2, (C) 3% n-TiO_2; (D) 5% n-TiO_2

sorption (water or artificial Salvia) and solubility are kept minimal for dental restorative materials as the (water or artificial Salvia) molecules penetrate into dental composites and possibly generate evolution of localized stress and strain, and significantly reduce the mechanical strength of dental composites by breaking the sensitive interphase bonding between fillers and matrix (Panyayong et al., 2002).[21] The results of artificial Salvia sorption demonstrated a non-significant decrease in artificial Salvia sorption of the new polymer nanocomposite. In addition, the silanization of the nanofillers produces hydrophobic surfaces; thus, it's expected to

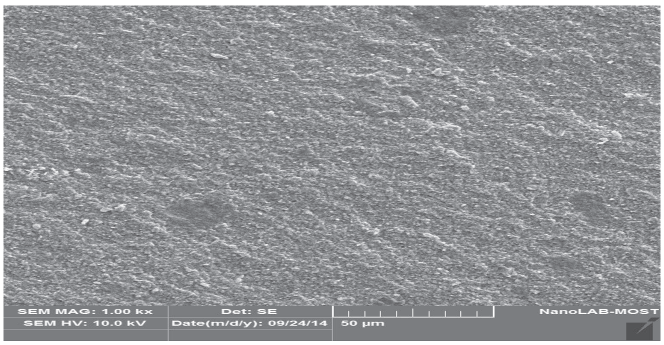


Figure 6: SEM of n-TiO_2 Powder.

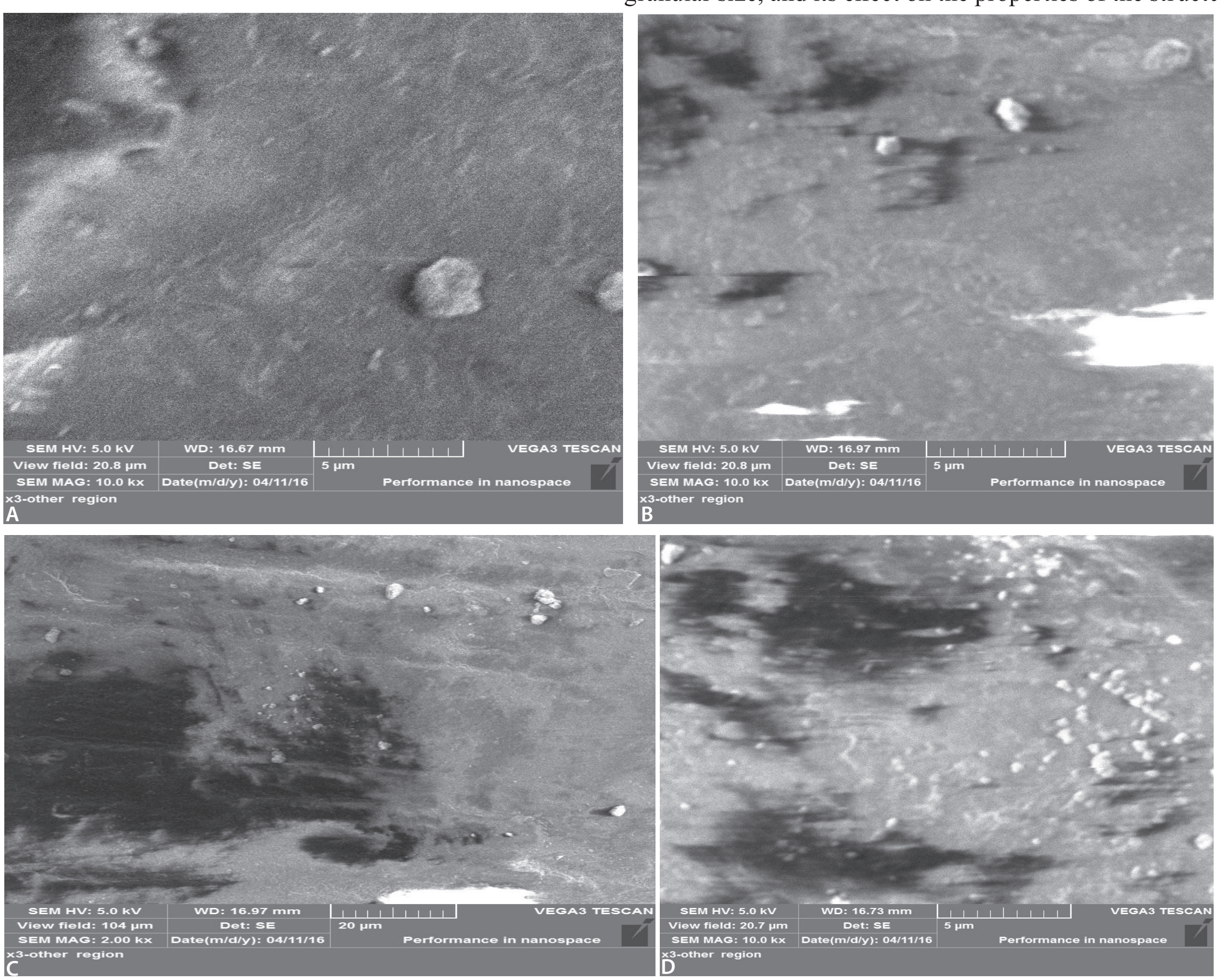
synthesize hydrophobic organic-inorganic composite materials. These results are in agreement with (Panyayong et al., 2002)[22] we found that there was a significant decrease in the values of water sorption after the addition of 5% (n-TiO_2) of nano-sized particles to PMMA in these ratios.

Antibacterial Test

An important test for artificial tooth fillings is the antibacterial. Figure 5 illustrates the method used to determine the effect of added nano-titanium oxide compared to the non-reinforced tooth fill. The identity of the types of bacteria that colonized in the mouth cavity which responsible for dental caries, gingivitis and periodontitis cases, moreover; isolating microflora which have biochemical activities to cause other oral problems this agreement with reference.[22] The large variety of bacterial genus included Streptococcus spp. which was represent the high significant ratio of 33.3 %, followed by

CHARACTERIZATION OF TEETH FILLING

In this research, several techniques were used to study the behavior of the nanoparticle within the base material to determine its distribution, the extent of its mass, the size of its granular size, and its effect on the properties of the structural



Figures 7A to D: SEM of (A) 0%n-TiO_2: (B) 1% n-TiO_2: (c) 3% n-TiO2 and 5% n-TiO2

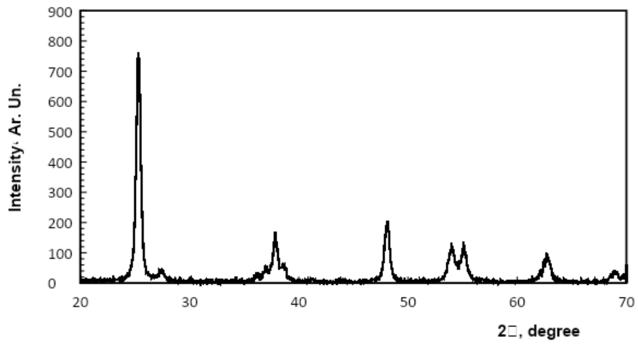


Figure 8: X-ray diffraction of n- TiO2 powder

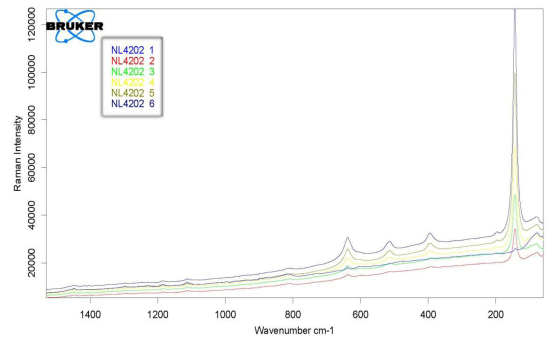


Figure 9: Raman Shift of PMMA/n- TiO2 Nanocomposites

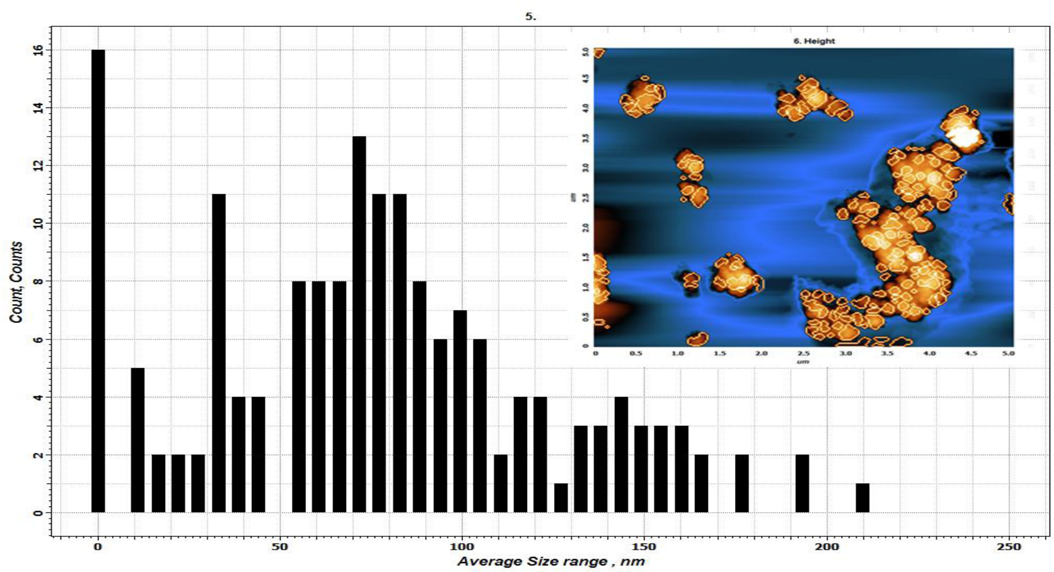


Figure 10: Atomic force microscopy (AFM) of n-TiO2

material by knowing the Raman Shift. All these changes will be known using techniques (x-ray diffraction, Scanning Electron Microscopy, Raman spectrum displacement, and atomic force microscopy). Figure 6 shows the homogeneous distribution of n- titanium oxide powder at 50 nm. Figure 7 shows different images of models prepared as industrial toothpaste according to the percentage of each model. Figure 8 shows the X-ray diffraction of the nanocrystalline powder used, and through the examination shows that the powder phase is of type (Anatase) and this type shows high purity of impurities. Figure 9 shows the Raman spectra of the samples and note from the shape peaks removed from the top of the base material by increasing the percentage of titanium oxide powder. Figure 10 shows the granular distribution of the used titanium oxide powder, which is not a single particle size value, but an average but more valuable value of the powder is within the range used.

CONCLUSIONS

We conclude from this research that:

- The wear rate of the base material (PMMA) is gradually reduced when it is reinforced by the nano- titanium dioxide powder and all the ratios used in this work.
- Dental fillings manufactured in this work shows high resistance to decay through the first conclusion in addition to the length of life.
- All the synthesis fillings reinforced are capable of adapting to industrial saliva and are not absorbable compared to the base material (PMMA).
- Using the techniques used to explain the structure characterization of manufactured dental fillings, the homogenous distribution of nano- titanium dioxide in the base material (PMMA) improved the results above.

REFERENCES

1. Price CA. A history of dental polymers. J Prosthodont 1994;8:47-54.
2. Soh MS, Sellinger A, Yap AU. J Dental Nanocomposites 2006;24: 373-381.
3. Kanie T, Arikawa H, Fujii K, Inoue K. Physical and mechanical properties of PMMA resins containing gamma methacryloxypro pyltrimethoxy-silane. J OralRehabil 2004;31:166-171.
4. Vallo CI, Abraham GA, Cuadrado TR, San Román J. Influence of cross-linked PMMA beads on the mechanical behavior of self-curing acrylic cement. J Biomed Mater Res B Appl. Biomater 2004;70:407-416

5. Mowade TK, Dange SP, Thakre MB, Kamble VD. Effect of fiber reinforcement on the impact strength of heat polymerized polymethyl methacrylate denture base resin: in vitro study and SEM analysis. Journal of Advanced Prosthodontics. 2012;4:30-36.
6. Faot H, Panza LHV, Garcia RCM, Bel Curry AAD. Impact and Flexural Strength, and Fracture Morphology of Acrylic Resins with Impact Modifiers. Open Dentistry Journal. 2009;3:137-143.
7. Narva KK, Lassila LV, Vallittu PK. The static strength and modulus of fiber reinforced denture base polymer. Dental Materials. 2005; 21:421-428.
8. Bashi TK, Al–Nema LM. Evaluation of Some Mechanical Properties of Reinforced Acrylic Resin Denture Base Material (An In Vitro Study). Al-Rafidain Dental Journal. 2009;9:57-65.
9. Chen M-. Critical reviews in oral biology & medicine: Update on dental nanocomposites. J Dent Res. 2010;89(6):549-560.
10. Ferracane JL. Current trends in dental composites. Crit Rev Oral Biol Med,1995;6(4):302-318.
11. Lindberg A. Resin composites: Sandwich restorations and curing techniques Umeå Odontology; 2005.
12. Mitra SB, Wu D, Holmes BN. An application of nanotechnology in advanced dental materials. The J of the Am Dent Ass. 2003; 134(10):1382-1390
13. K. S. Bong, K. Y. Jick, Y. T. Lim ND A. P. Su, “The Characteristics of a Hydroxyapatite-Chitosan-PMMA Bone Cement,” Biomaterials, 2004;25(26):5715-5723.
14. K. Serbetci, F. Korkusuz, and N. Hasirci, “Thermal and Mechanical Properties of Hydroxyapatite Impregnated Acrylic Bone Cements,” Polymer Testing, 2004;23(2):145-155.
15. M. Wang, R. Joseph and W. Bonfield, “Hydroxyapatite-polyethylene Composites for Bone Substitution: Effects of Ceramic Particle Size and Morphology,” Biomaterials, 1998;19(24):2357-2366.
16. R. K. Roeder, M. M. Sproul, and C. H. Turner, “Hydroxyapatite Whiskers Provide Improved Mechanical Properties in Reinforced Polymer Composites,” Journal of Biomedical Materials Research Part A, 2003;67A(3):801-812.
17. W. Chen, Y. Li-Dan, Z. Yan-ping, U. X. Lian-Lai, Tianjin Med. Univ. 94 960. (2006).
18. Zehnder, G.W., Yao, C., Murphy, J.F., Sikora, E.J., Kloepper, J.W., Induction of resistance in tomato against cucumber mosaic cucumovirus by plant growth-promoting rhizobacteria. BioControl 2000;45:127-137.
19. S. C. Ramesh, K. S. Seshadri, Wear 255 893. (2003).
20. W N. Muhammad1, S. Maitra1, I. Ul Haq2, M. Farooq[Some studies on the wear resistance of artificial teeth in presence of amorphous SiO_2 and TiO_2 fillers, Cerâmica 57 (2011) 324-328.
21. Solnit GS. "The effect of methyl methacrylate reinforcement with silane treated and untreated glass fibers". J Prosthet Dent; 1991; 66:310-314.
22. Panyayong, W., Oshida Y., Andress C.J., Borco T.M., HovijitraS. Reinforcement of acrylic resin for provisional fixed restoration. Part III:"effect of addition of titrant and zirconia mixtures on some mechanical and physical properties. Biomed. Mater. Eng.; 2002; 12(4): 353-66.
23. Sulaiman, A.W. (2000). Quantitative measurement of urea content in saliva acquired pellicle and dental plaque in relation to dental caries susceptibility in human adults. M. Sc. Thesis in preventive dentistry. College of dentistry. University of Baghdad.