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Study the Structural and Mechanical Properties of HAP-MgO NPs-PMMA Nano-Composite

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ABSTRACT

Polymer composite of heat cure acrylic (PMMA) matrix reinforced with different weight of nanoparticles Hydroxyapatite and Magnesium Oxide (1, 5 and 10% wt.) were prepared by hand molding technique. The Magnesium oxide nanoparticles were synthesized by Simple Chemical Method (Co-precipitation). The structure and morphology properties of MgO examined by X-ray diffraction (XRD), Field Emission Scanning electron microscopy (FESEM) and EDX. Lattice constant is a= 4.19**A**◦ and crystallite size is 17.233nm, and average particle sizes of Magnesium oxide is(110) nm. While, the Atomic Force Microscopy showed that the roughness insufficient affected by NPs adding, the fluctuating is also happened in average grain size. The composite mechanical properties were positively affected through the addition of nanoparticles, where the young modulus for pure PMMA was 429.2566 N/m^2 which increased for the (1, 5 and 10% wt.) (HAP/MgO) addition 908.345, 1697.482and 564.175 MPa respectively, and PMMA Vickers hardness test was 314.31HV and the values after enhancement with the nanoparticles were 442.786HV, 306.959HV, and 318.727HV. Thermal behavior decreased in the glass transition temperature when the nano material load increased, also the composite showed an amorphous structure. These results indicated that nano-composite possesses good structural, mechanical and thermal properties for dental applications.

KEYWORDS: Bio composite; dental applications; DSC; nano ceramics powder; PMMA

الخلاصة

تم تحضير متراكب بوليمري من بولي مثيل ميثاكريلايت (PMMA) الحراري المدعم بنسب وزنية مختلفة(١٪، ٥٪، و ١٠٪) من (Hap / MgO) بتقنية القولبة اليدوية.حيث تم تصنيع الجسيمات النانوية من أكسيد المغنيسيوم بالطريقة الكيميائية البسيطة, و فحص الخصائص التركيبية لـ MgO بواسطة حيود الأشعة السينية (XRD) والمجهر الإلكتروني لمسح الانبعاث الميداني (FESEM) والاشعة السينيةالمشتتة للطاقة EDX. وكان ثابت الشبيكة = ٤,١٩انكستروم والحجم البلوري ١٧,٢٣٣ نانومتر، ومعدل الحجم الحبيبي لأوكسيد المغنيسيوم (١١٠,٨) نانومتر. أظهر الفحص المجهري للقوة الذرية تأثر كل من الخشونة ومعدل الحجم الحبيبي بشكل طفيف عند إضافة الجسيمات النانوية ،.كما تأثرت الخواص الميكانيكية المركبة لفحص الانضغاطية بشكل إيجابي عند إضافة الجسيمات النانوية، حيث كان معامل يونك لـ البوليمر النقي ٤٢٩,٢٥٦٦ والذي زاد عند أضافة (HAP / MgO) بالنسب الوزنية (١٪ ، ٥٪ و ١٠٪)(٥٦٤,١٧٥ ، ٩٠٨,٣٤٥ ، ١٦٩٤,٤٨٢) ميجا باسكال على التوالي ، وكان اختبار صلادة البوليمر النقي ٣١٤,٣١ وقيم الصلادة لعينات المتراكب النانوي هي ٤٤٢,٧٨٦،٣٠٦,٩٥٩ و ٣١٨,٧٢٧ على التوالي، بينما انخفض السلوك الحراري في درجة حرارة التزجج عندما زاد تركيز مادة النانو، كما أظهر المركب بنية غير متبلورة. أشارت هذه النتائج إلى أن مركب النانو يمتلك خصائص هيكلية وميكانيكية وحرارية جيدة لتطبيقات طب الأسنان.

INTRODUCTION

Each Polymer nano composite developed quickly as a diverse research field with the potential to enhance polymer applications to the benefits of industry, polymers mixed with nanoparticles NPs obtaining unique properties due to their properties. Polymer-NPs bio- composites widely attraction attention in medical applications such as in dentistry as a bone cement [1] and prosthetics [2]. Due to aesthetic urgent needs for dental restorations also polymer materials have to simulate oral tissues so they must be mixed with nanoparticles because of their unique characteristics like the high volume-to-surfaces area ratio. To improve the mechanical properties, increase elasticity without losing strength, scratch endurance and improve thermal properties also reinforce the binding agent such as nano-adhesives for polymers [2-4]. The poly methyl

methacrylate is one of the most common polymers that is used for dental applications as a dental prosthetic material in nano composites because of their mechanical and physical properties along with the low cost, ease to manufacture, good compatibility with the human tissue, low density [2, 5] and non-toxic[6]. PMMA polymer exhibits better impact strength and mechanical and physical properties than other polymer materials, were 5.27 (kJ/m^2) [7]. However, the material possesses poor mechanical and physical properties when used alone [8-10]. It is found that the incorporation of additive particles like magnesium oxide (MgO)[11], hydroxyapatite (HAP) [12] and other minerals [10,13] added into polymeric material can be improved at least one benefit such as biological, thermal, and mechanical properties of PMMA. Polymeric-based composites of PMMA/HAP/MgO operate as materials designed to sustain numerous mechanical stresses and perform in adverse oral climatic conditions [14]. Recently Ali Abd A. Mansour *et al.* created a ceramic-polymer mixture utilized as a replacement for injured bones or teeth. Composites were made using a manual mixing procedure, and then ultrasonic technology was used to assure equal powder dispersion within the polymer. The polymer (PMMA) is supported by a ceramic powder (HA/MgO) that was prepared using an effective mechanical mixing method and contains hydroxyapatite (HAP) and metal nanoparticle magnesium oxide (MgO), which is an additive to the polymer (PMMA). the proportion utilized for reinforcement (1%-4%**)** the findings of the particle hardness analysis revealed a gradual rise in percentages. The findings of compressibility and Young's modulus durability increased in direct proportion to the percentages of ceramic powder applied [15]. The study aims are preparing a bio composite for dental applications from PMMA/HAP/MgO. The structural, mechanical and thermal properties were studied in term of XRD, FESEM, EDS, compressing, bending, impact, and DSC.

MATERIALS AND METHODS

Synthesis MgO NPs: Magnesium oxide nanostructure were prepared by simple chemical method through preparing two solutions A and B, where solution A prepared by dissolving 0.2 mole of $MgCl_2.6H_2O$ in 50mL distilled water solution A and solution B prepared by dissolving 0.4 mole sodium hydroxide NaOH with urea as a catalyst dissolved in 25 ml distilled water, each solution magnetic stirred for 15 min. then solution A was added to solution B drop by drop using syringe and stirred for 30 min. at room temperature. White precipitate was formed then isolated by centrifuge at 3000rpm for10min then double washed by distill water and ethanol, the precipitate dried at 100 °C. Finally, the calcinations were carried out at 700°C for 6 hours, to obtain MgO powder.

Synthesis of PMMA/HAP/MgO composite mold sample: Nano composite mold hybrid samples were through mixing heat cure acrylic PMMA (Heat Cure Acrylic; Integra, Turkey) with (1, 5 and 10% wt.) of the nano hydroxyapatite ($HCa_5O_{13}P_3$) (Shanghai Hualian Chemical, China) and prepared MgO NP, using a magnetic stirrer (FourE's /china) for 15 minutes, then grinned with the usual mill. The powders is then placed in a Vortex device to confirm homogenous distribution, the molds were made due to the company instructions sheet for using heat-PMMA polymer, with a 2:1 weight ratio of powders to liquid in order to make a paste, the paste was left to rest until the dough stage is reached, the nanoparticles mix ratio added to the matrix was (1, 5 and 10% wt.) (1%HAP 0%MgO), (4.7% HAP, 0.3%MgO), and (9.4%HAP-0.6%MgO) respectively. To remove any air bubbles in the dough, the dough-like pate is placed in a pre-prepared corrosion-resistant steel mold; the material casts in the mold middle plate two rectangular hollows (150x50 mm). Then put in 80˚ furnace for 3hours. After that the molds cut according to the required measurements. The mold used for preparing the samples for mechanical and thermal testing and experiments.

RESULTS AND DISCUSSION

Structural, morphological analysis (XRD/EDS/FESEM for Nano MgO and AFM for Nano composite PMMA/HAP/MgO): Figure 1 (a) represented the relationship between intensity and (2θ) in range 27˚ and 80˚ for the MgO NPs. The NPs has a polycrystalline structure with face center cube shaped. It obviously exhibits the peaks were absorbed 27.51˚, 31.83˚, 37.09˚, 43.07˚, 45.57˚, 56.5 6˚, 75.08˚, 78.77˚, and 84.03˚ along with Miller's indices (300), (220), (111), (200), (400), (422), (311), (222), and (444) respectively according to (JCPD No. 45.0946). The average

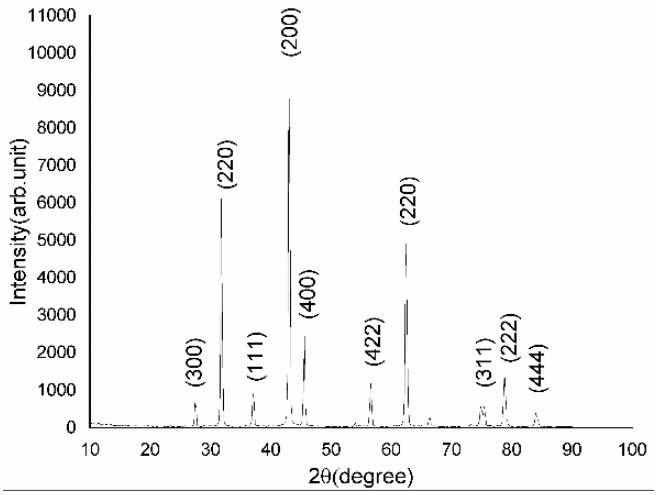
Crystallite size was calculated using Scherer equation:

$$\text{Crystallite size} = 0.9\lambda/(\beta \cos\theta) \quad (1)$$

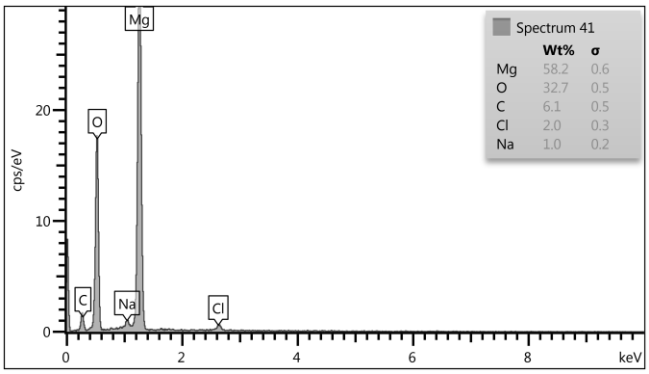
Where λ=1.54A° is the wavelength of X-ray, Full Half Width Maximum (β) is the breading of the diffracted peak at half maximum and θ is the Bragg angle. The crystallite size calculated via Scherer Eq. (1) for the strongest peak MgO was 17.233 nm compared to the research [16, 17]. Lattice constant was a = 4.19 Å confirms that MgO NPs was pure and free of impurities.

Figure 1 (b) displayed the EDX, the quantitative analysis of MgO NPs clear strong showed peaks are belongs to Mg and O and presence small other peak does not effect on MgO high purity [9]. Figure 1(c) includes FESEM images of MgO calcined at 700 ◦C. It is clear from these images that the morphologies and surface features (particle size and shape) of Magnesium oxide nanoparticles prepared via simple chemical method showed nano-spherical shapes with different diameter of well-uniform were crystallized with agglomeration appearing in magnesium oxide nano-powder [18] aggregations and large grain size was because of the calcination preformed at high temperature due to the internal particles that generated by electrostatic forces [17]. The FESEM illustrative histogram in Figure 1 (c) obtained using (Image J program) of at least 100 particles shows the particles have an average diameter of 110.8 nm and a standard deviation of 30.75 nm. The minimum diameter of MgO particles was 62.7 nm, and the maximum was 181.14 nm.

The Atomic Force Microscopy test of the nano composite PMMA/HAP/MgO shown in Figure 2: a, b, c and d elucidate the average particle size was measured, which is indicated average diameter for each sample displayed for 2D and 3D in Table 1. In it, elucidate Roughness average, Root Mean Square (RMS)/nm and average grain size/nm of particle size distribution for these nano composites of PMMA samples of different surface roughness that the composite with 5% nanoparticles filler exhibits an increasing in the value of pure PMMA in both roughness and root mean square, meaning this filler ratio acquired the composite distribution of surface height and particles shape more than the pure polymer that led to an increase in roughness. These behaviors attributed to the average grain size variation according to filler content.



(a)



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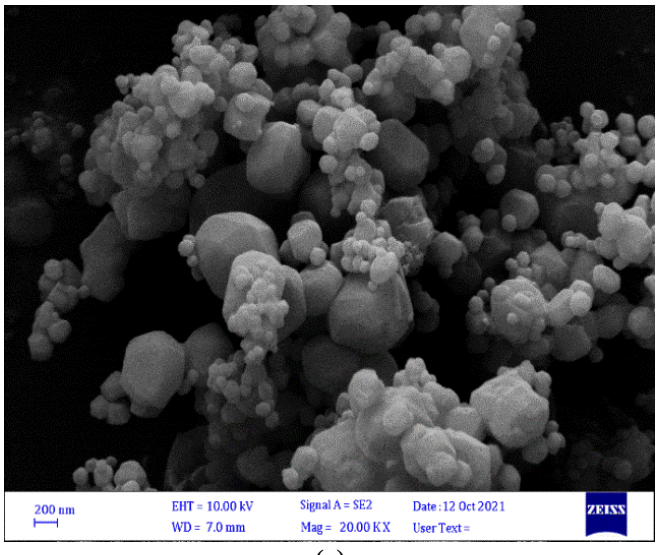

(c)

Figure 1. The shows morphological and structural tests for MgO NPs (a) XRD (b) EDX spectrum (c) FESEM image.

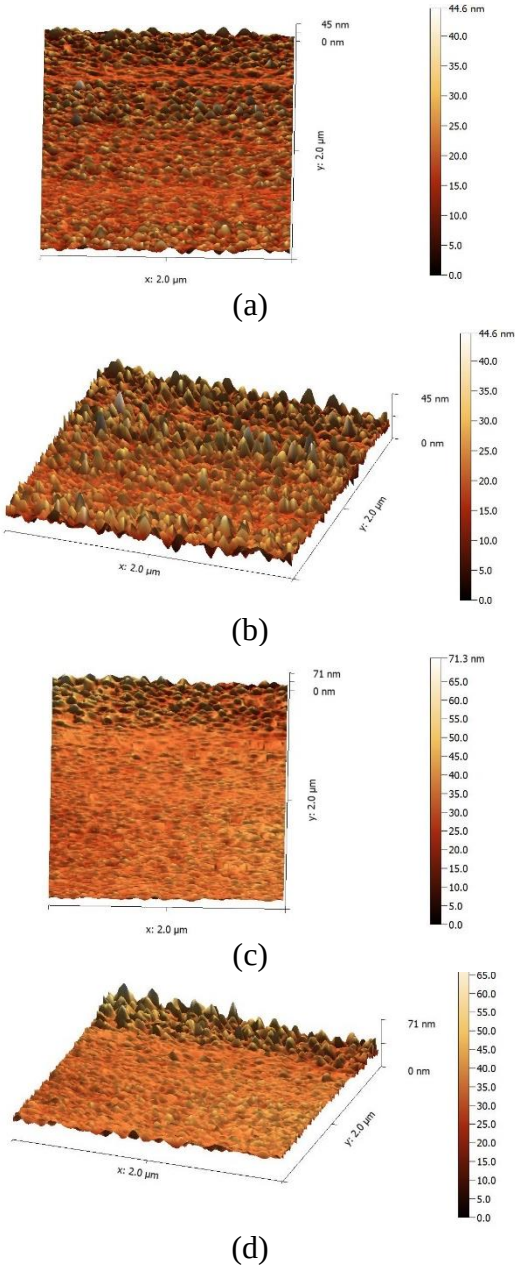

Figure 2. shows AFM surface morphology of PMMA/HAP/MgO nano composite in 2D and 3D (a) Pure PMMA, (b) 99% PMMA, 1%HAP, (c) 95%PMMA, 4.7%HAP, 0.3%MgO, (d) 90%PMMA, 9.4%HAP, 0.6%MgO.

Table 1. AFM results of PMMA/HAP/MgO.

Composite	Pure PMMA	1%	5%	10%
Roughness average (Ra)/nm	3.04	2.01	3.1	2.00
Root Mean Square (RMS)/nm	4.05	2.72	4.33	2.85
Average grain/nm size/nm	19.07	16.15	33.91	18.15

Mechanical properties

Compression test: The compression test is an important tool for determining the mechanical properties of the polymeric composites, in a compression test a specimen responds to forces that push or compress until the material flattens. Mechanical properties of tough polymeric materials were determined by calculating using testing procedures for compressive properties of tough polymeric materials, uniaxial compression analyses at room temperature, by using a universal Hydraulic press supplied by the company (Ley bold Harris no.36110). The test technique is obeying ASTM D695 [19], specimen's dimensions (5x5x10) mm were cut. The Young modulus and ultimate stress as a function of sum of nano-particles filler weight ratio are given in Table 2. This behavior is the result of a relationship of stress-strain curve as shown in Figure 3.

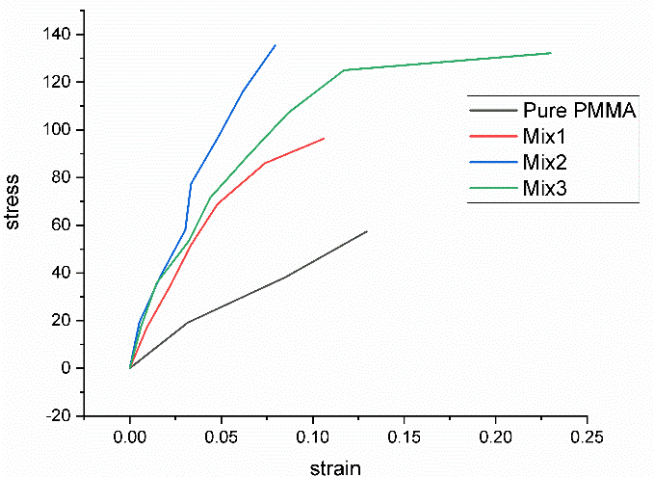

Figure 3. Stress-strain curve.

The mechanical tests from Table 2 noticed young modulus have doubled and tripled at MIX2 and MIX3 respectively, and then, drawn at MIX3. The low interatomic bonding in polymers they have low young elasticity, adding nanoparticles showed strong effects on young elasticity because the nanoparticles feature of size dependence the mechanical properties of NPs, like elastic modulus and interfacial adhesion are significantly related to the type and the number of nanoparticles are used. With an extra number of nanoparticles, the composite became brittle and lost a large part of its elasticity. Figure 3 illustrated the behavior change from low yield ductile polymer to high yield brittle composites. The slight difference in the polymer PMMA behavior after being loaded with HAP and MgO nanoparticles for each weight

ratio percentage is due to them that the elastic modulus and ultimate stress are increased when additives are being used since the nanoparticles fill the pores and close the interfaces between the composite, causing the material to change from a polymer-like behavior to a stronger material by increasing density, and thus the young modulus is significantly increased.

Table 2. Compression test results of different weight ratio filling.

PMMA (Wt%)	MIX (Hap/MgO) (%)	HAP (Wt%)	MgO (Wt%)	Young modulus (MPa)	Ultimate stress (MPa)
100	0	0	0	429.2566	57.416
99	1	1	0	908.345	96.386
95	5	4.7	0.3	1697.482	135.396
90	10	9.4	0.6	564.175	132.175

Bending strength test: The bending test was performed in order to evaluation of the bending strength properties. The main objective to do this type of test is to identify the material linear behavior for all the prepared bio composites called (Hook's behavior) which it is the behavior of a material when it is subjected to a load on the material's surface and in its perpendicular direction[20] The three-point bend tests on the samples without notch were conducted at room temperature and at a loading speed 5 mm/Min. using tensile test device (Instron 1195, England). The effect of the addition the nanoparticles powders HAP/MgO as reinforcement particle to PMMA on the flexural strength is shown in figure (4) when compared with pure PMMA samples bending strength increased first and then decreased with the constant addition of HAP/MgO. It may be considered that the addition of HAP/MgO nano powders played an important role to endow good adhesion for the nano composite and mechanical properties can be improved greatly by the incorporation of HAP/MgO.

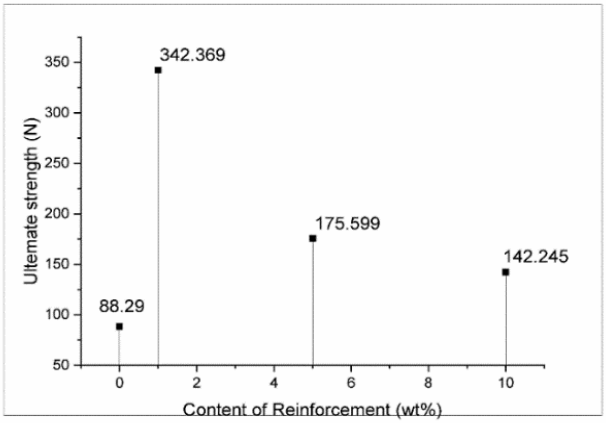

Figure 4. Ultimate stress dependence on filler ratio.

Vickers's hardness test: Vickers's hardness was performed to determine the VH according to specification of instrument, 0.49 kg load for 30 seconds. Calculated the final number was considered as the average hardness among 2 points of the indentation. VH average values of the samples pure PMMA and samples composites displayed in Table 3 the value increases when adding Nano HAP and Nano MgO Table 3. Enamel tissue is the hardest crown of the dental above the gum tissue in the human body also contains almost no water, By comparing Vickers hardness value, enamel protects the dentin by covering the entire anatomy and coming into contact with food during mastication [21, 22] Hardness measurements were in the range from 270 to 360 VHN for enamel and 50 to 60 VHN for dentin. So, it is possible to be good samples as a nano composite material used for the manufacture or filling of the enamel layer of prosthetic teeth. The table revealed that hardness was enhanced with a slight increase in particles filler content and decreasing in sample Mix2 this is due to an increase in the hard and brittle nano content of HAP and MgO stiffened the elasticity of the matrix material and improved the material resistance to indentations on the surface of the materials. More hardness of the nanoparticles filled composite compared is related to a good binding of. It may mention the fact that a smaller filler size will enhance the dispersion of the filler particles in the polymer, which led to an improvement in interfacial bonding between the filler and polymer. These results confirmed that the incorporation of nanoparticle additives strongly affects the mechanical properties of polymer composites.

Table 3. Micro hardness of PMMA-HAP-MgO.

Samples and samples weight ratio (%)				Average Hardness (HV)
Sample	MgO	HAP	PMMA	
Pure	0	0	100	314.31
1%	0	1	99	442.786
5%	0.3	4.7	95	306.959
10%	0.6	9.4	90	318.727

Impact strength test: Specimens for impact strength test were prepared into bar-shaped blocks with dimensions of (4mm×10mm×55mm) according to ASTM-D256. A pendulum Charpy-type impact test machine (LARYEE, China) was used to achieve the impact test at room temperature and relative humidity 80%. A drop weight of (7.5) J was released at the specimen; impact strength was recorded for each specimen introduced in Figure 5. The effect of stress from the impact strength test causes cracking in the polymer surface due to weak Van der Waals bonds. Therefore, the addition of nanoparticles reinforced the polymer for shock resistance. the nanoparticles enforcement also increased the absorbed energy and therefore impact strength increased [23] the addition of Nano HAP/MgO increased the impact strength of the pure specimen of PMMA Nano-HAP effect on fracture energy The best fracture energy value is obtained at Mix3 were nano HAP powder formed 9.4% of sample increased nano HAP leads to reducing the fracture energy. This behavior proved that the material becomes brittle at high filler content of HAP.

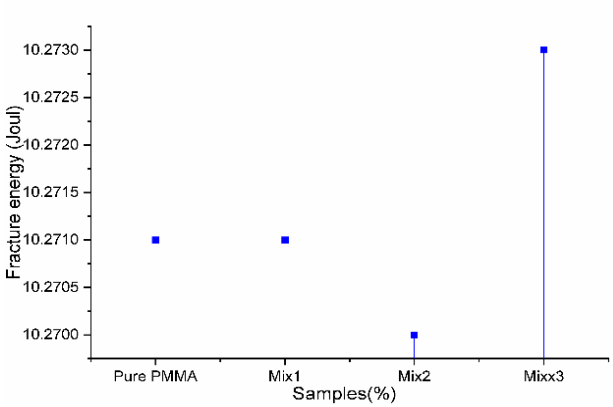

Figure 5. Fracture energy for Pure PMMA, MIX1 99% PMMA, 1%HAP, MIX2 95%PMMA, 4.7%HAP, 0.3%MgO, MIX3 90%PMMA, 9.4%HAP, 0.6%MgO.

Thermal analysis Differential scanning calorimetric (DSC) analysis was performed by (SETARAM, type:131 EVO, France) device to evaluate the glass transition temperature (Tg) and melting temperature (Tm) of the (PMMA/HAP/MgO) nano-composite. Samples of 15 g weight were heated from 20 to 350 °C at a rate of 10 °C min^{-1} in pan sized 100. Figure 6 showed the DSC analysis and Table 4 illustrated the DSC analysis data. Gradual increases in the curve as the heat flux increases, increases the heat capacity of the PMMA polymer, which is known as the glass transition. At the glass transition point, the polymers have a greater heat capacity than they do at lower temperatures. The small amount of Hap (1%) increased the glass transition temperature by 14.5 degree. On the other hand, this improvement in Tg is regressing when the mixture HAP-MgO NPs was used.

The composites' glass transition behavior depends on: polymer host-filler particles interaction, changes in molecular weight, and retained monomer. but if the particle surfaces are coupled to the matrix, the decrease in Tg will completely disappear. This is because the interface difference is quite dynamic between the polymer and the particle as the Tg depends on the distance between the interfaces and the molecular weight. It assumed that the decrease in the glass transition temperature (at 5% and 10% wt. nanoparticles content) was expected for particles showing little or no interfacial interaction with the host polymer [24]. The polymer dynamics allowed more degrees of freedom for chain relaxation. Therefore, the high chain mobility decreased the Tg , this behavior is also occurred in [24, 25]. The polymer has a great deal of flexibility over the glass transition. At a certain temperature, the polymers get enough energy to transition into highly organized crystal formations, and in this case, the polymers release heat. The decrease in heat flux that occurs when this heat is cooled results in a significant decrease in heat flux against the temperature chart of the furnace. The polymer's crystallization temperature, since PMMA is amorphous, it in not get curve dips.

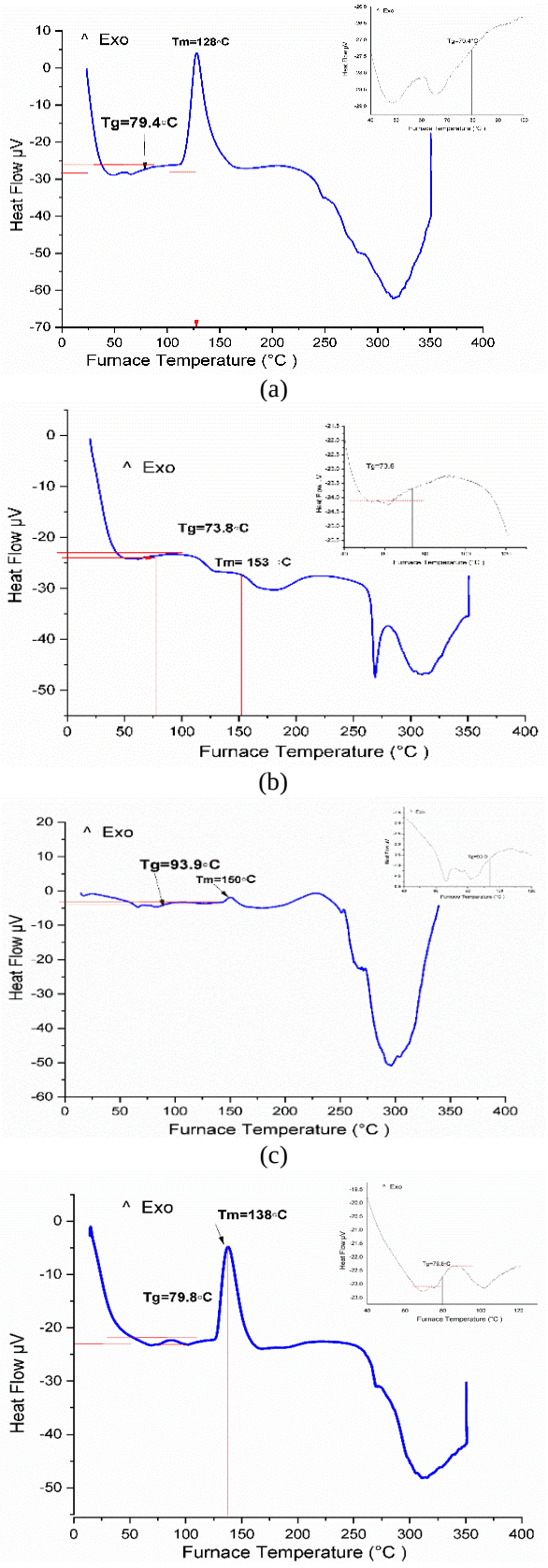

Figure 6. DSC for samples: (a) Pure PMMA, (b) PMMA-1%(HAP-MgO), (c) PMMA-5% (HAP-MgO), (d) PMMA-10%(HAP-MgO).

Table 4. DSC of PMMA/HAP/MgO.

NP ratio	PMMA wt %	HAP wt%	MgO wt%	Tg ◦C	Tc ◦C	Tm ◦C
0	100	0	0	79.4	amorphous	128
1%	99	1	0	93.9	amorphous	150
5%	95	4.7	0.3	73.8	amorphous	153
10%	90	9.4	0.6	79.8	amorphous	138

CONCLUSIONS

In this paper, we concluded that the addition of (hydroxyapatite and magnesium to oxide) mix by weight ratios (1, 5 and 10% wt.) as a reinforcement nano powders to poly methyl methacrylate polymer significantly improved mechanical and thermal properties of the polymer and showed good result to use as dental material such as manufacturing filler for enamel layer or teeth prosthetic.

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Disclosure and Conflict of Interest: The authors declare that they have no conflicts of interest.

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