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Water Temperature Effect on Hardness and Flexural Strength of (PMMA/TiO_2 NPs) for Dental Applications

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Abstract:

PMMA (Poly methyl methacrylate) is considered one of the most commonly used materials in denture base fabrication due to its ideal properties. Although, a major problem with this resin is the frequent fractures due to heavy chewing forces which lead to early crack and fracture in clinical use. The addition of nanoparticles as filler performed in this study to enhance its selected mechanical properties. The Nano-additive effect investigated in normal circumstances and under a different temperature during water exposure. First, tests applied on the prepared samples at room temperature and then after exposure to water bath at (20, 40, 60) C° respectively. SEM, PSD, EDX were utilized for samples evaluation in this study. Flexural strength, shear stress, hardness value were evaluated for all samples, the tests applied periodically at (15, 30, 45) min. Interestingly, it is found that at the normal condition (NC) the cooperation of TiO_2 NPs significantly improves the selected properties. The maximum increase is located at 4 wt % of TiO_2 NPs addition. The results also showed that temperature increase accelerate the water permeability within all samples thus decrease the mechanical properties. Obviously, a better hardness value determined for the prepared nanocomposite comparing to the control group at the same test condition.

Keywords: Flexural strength, PMMA for dental use, Shear stress, Shore (D) hardness, TiO_2 NPs.

Introduction:

For several years, PMMA is considered as a major substance for denture base fabrication process[1]. However, this polymer dose not possess enough strength to resist fracture in case of a sudden falls on a tough surface or breaks after submitting to over forces of mastication [2-4]. Moreover, during the time frequent biting endues deformation effect, which represents another kind of failure[5]. Since it is used in this application, there were many researches who attempt to enhance this acrylic resin mechanical properties such as flexural strength, impact resistance, hardness and other important feature[4,6-9]. In flexural strength, the weakness was due to brittle nature at glass transition temperature, which is about 110 C^{o}, in addition to cyclic loading ability.

Yet, the addition of a special material as reinforcement to PMMA could help to enhance these weakness points in the mechanical properties. The detailed mechanism of such composite materials mechanical properties and the proper procedure for fabrication are considered as highly important requirement in dental world[10]. Several studies were introduced to improve the properties of PMMA like flexural strength or hardness, shear strength and tensile strength for dental use by the addition of different fillers like glass fibres [4,11], whiskers[12] nylon [13] or even using blend polymers mixture [14].

Nanotechnology produced a new era for reinforcement additive to material composite because of their specific physical, chemical and biological characteristic. The use of Nanoparticles (Nps) such as alumina Nps, TiO_2 Nps, silver Nps, ceramic Nps and ZrO Nps are utilised to improve denture base resins mechanical properties[15,16]. Moreover, nanoparticles materials is one of the most used additive on fabricating PMMA nano-composite, this is due to the characteristic of high surface area-to-volume ratio. This characteristic is considered as an advantage so a great enhancement in material performance was finally obtained. The size, the type and the distribution of nanoparticles represent the most essential features that control the

benefit of improving the mechanical properties of nano-composite[17,18]. The nano additive integrates with the polymeric matrix to modify most of the mechanical properties such as the rigidity, fracture toughness, hardness and other functional properties of the new prepared nano-composite[19].

Many researches utilised synthetic nanoparticles like ZrO_2, Al_2O_3 and the results showed enhancement for some tested mechanical properties[20,21]. Also, using mixture of ZrO_2/ TiO_2 [22] introduced higher property values. Al_2O_3, Zr_2O_3, and SiO_2 were used as nano-filler for nano-composite denture base[23,24]. Titanium dioxide is one of the best nanoparticles used by researchers in the medical field due to its unique characteristic. Pleasing color, excellent mechanical properties, high biocompatibility, high stability and most importantly the antimicrobial effectiveness that make these particles a favourite addition to biomaterials [25-27]. Many improving results reported previously by different researchers suggesting that the incorporation of TiO_2 nanoparticles in PMMA resin matrix increases properties like surface hardness, impact and transverse strength of the resin [28,29].

Over all, pure PMMA possess insufficient value to maintain the longevity of use under the frequent stress [30]. Generally, a deficiency in the mechanical properties like flexural strength and hardness are considered as an essential issue in the denture base fabrication and need to be deeply studied. Hence, the main goal of this research is to experimentally study the advantage of manufacturing a reinforced PMMA using nanosize particles, evaluate the property of flexural strength, shear strength in addition to hardness under a specific circumstance. This paper presents an experimental study on Flexural strength and (Shore-D) hardness of PMMA before and after reinforcement with TiO_2 NPs in the range of (2 and 4) wt. %. Flexural strength and shear strength of denture base were examined in this study because their deficiency in the materials leads to a major cause of clinical failure. All selected tests were applied before and after immersion into special water bath. Different water temperatures were considered in this evaluation due to the gab in the previous researches, which neglect that factor involved nano-composites materials. These additives were investigated to give more explanation on the benefit of reinforcing PMMA with the TiO_2NPs, also to make a comparison between the control PMMA and test group prepared. Finally, to compare the effect of TiO_2NPs different concentration as additive to PMMA at these specific mechanical properties.

Materials and Methods:

In this study PMMA was utilized as pure polymer and reinforced through the addition of TiO_2 nanoparticles to prepare our nano-composites

Preparation of PMMA

In this study, PMMA was utilized as pure polymer powder. Pure PMMA was made from Stryker® How medica to prepare the all test specimens. In this work the mixing ratio between PMMA powder to MMA liquid was of 2:1. PMMA is considered as moldable, the liquid of (MMA) was poured in the prepared beaker containing the specific amount of polymer powder. Using hand lay-out technique the mixture must be stirred until the dough stage at room temperature. Therefore, a slow and continually mix before the dough stage must be kept to avoid the formation of bubbles inside the mixture. Finally, the mixed dough was poured to the prepared mould. A glass mould already was prepared for curing and formation, the exact dimensions were (10x 5x 0.5) cm. Also, the mould has glass cover at the same dimension to provide a fix pressure on the prepared dough. For solidification purposes, this mixture was left at room temperature for about (1hour). For post cure, the cast sheet was released out of the mould and placed in an oven at 55 C° for another 1hour.

Preparation of Nano Composite

Nano-composite specimens were made from PMMA and TiO_2 nanoparticles at room temperature 23 C°. The used TiO_2 NPs in this study are manufactured by company (Reinste Nano Ventures Pvt. Ltd). The selected weight fraction for this work was (2% and 4%*wt*). A total number of samples (36) was prepared for this research. Flexural strength and Hardness (Shore D) were applied for this research. The preparation process was made by hand lay- up technique at room temperature. It was mixed until the uniformly distribution of all nanoparticles in PMMA powder. Table 1 indicates the mixture composition prepared for this research with full details. Figure.1 shows the tested samples (pure PMMA and nano-composite of PMMA / TiO_2). It is clear that the observed color of the samples after full curing depends on the additive percentage, as the weight fraction increase of added nanoparticles the samples color tends to change from transparent pink to light pink. The addition of the specified amount of additive was firstly mixed with the correct amount of PMMA powder for each sample according to Table. 1, which indicates the group name according to TiO_2NPs. Then after mixing both (polymer and additive) powder, the MMA liquid was poured slowly in to the baker of the mixture with continuous mixing. Lastly, the whole prepared mixture was poured to the prepared

mould and let to dry. As the process with pure PMMA, these samples should be released from the mould and placed in an oven at (60C°) for another 1hour. The prepared nano-composites plates as indicate in Figure.1 were cut into the slandered samples dimension (ANIS/ASTMD790).

Table1. Ratio of mixing of the prepared composite

Group name	Concentration of TiO_2 (%)	Weight percentage of additive(g)	Weight percentage of PMMA+MMA liquid(g)
A	0	0.00	53.46
B	2	1.07	52.39
C	4	2.14	51.32

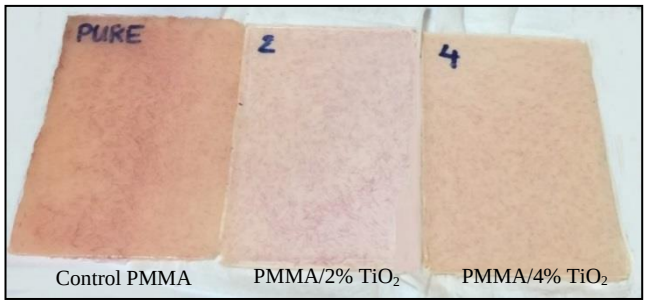

Figure 1. Prepared PMMA pure and nano-composite reinforced by (2%, 4%) TiO_2 NPs

Experimental Part

Flexural Strength Test

This test was performed using the universal tensile machine manufacture by (Laryee Company in china), type is (WDW-50) at room temperature. These tests were performed at constant cross-speed of 1.7 mm/min. The prepared samples were fixed on the two support points horizontally. A specific load then gradually was applied on the samples until breaking point. The required value for each sample was detected by the computer screen which connected with the same device. Flexural strength (F.S.) value is calculated by eq.1 [31]:

$$F.S. = \frac{3PL}{2bd^2} \qquad 1$$

Where (P) is fracture energy in *N*, (L) support span in mm, (b) sample width in mm (d) sample thickness in *mm*. At the same time maximum shear strength value were detected for the prepared samples. This value determined using the eq.2:

$$\tau_{max} = \frac{3P}{4bd} \qquad 2$$

Hardness (Shore D) Test

Shore (D) Hardness were applied according to ASTM D 2240, ISO 7619. The PCE-HT200 (Shore D) hardness device was utilized in this work to evaluate the indentation depth. This value should be change according to the sample material composition. With the digital screen of the device, the final value of the hardness is detected. Three different points were tested then an average value finally is considered for each sample.

Result and Discussion:

SEM, PSD, EDX Evaluation

Scanning electron microscope (SEM) for each sample was performed to correlate the determined mechanical evaluation for each sample state. Fig. 2 (A, B, C) presents the micrograph of the pure PMMA, PMMA/TiO_2NPs at (2 wt% and 4 wt%) respectively. Fig. 2A displays the homogeneous morphology of the pure sample, while in Figure B and C for 2% and 4% reinforcement respectively there was a clear change in the distribution of sample surface due to the presence of TiO_2 NPs. In these figures, it noticeably indicated that as the percentage of the nanoparticles increase as the surface shows more roughness and became semi-continuous morphology due to some particles aggregation. Furthermore, it was observed that the dispersion of the additive used was relatively good and uniformly dispersed throughout the entire matrix. Also, a better distribution could be found in Figure 2B compared to Figure 2C due to the increase of additive inside the acrylic matrix. However, it was hard to clarify the size and TiO_2 NPs embedded in the polymer matrix, therefor Particle Size Distribution (PSD) and Energy Dispersive X-Ray (EDX) analysis observations were performed on the samples. Additionally, the particle size distribution of used fillers powders was carried out by (90 pulse particles sizing device). Fig. 3 shows the value of PSD of TiO_2NPs used in preparing the nano-composite. As in the figure, the mean diameter of nano-filler was around 38.6 nm.

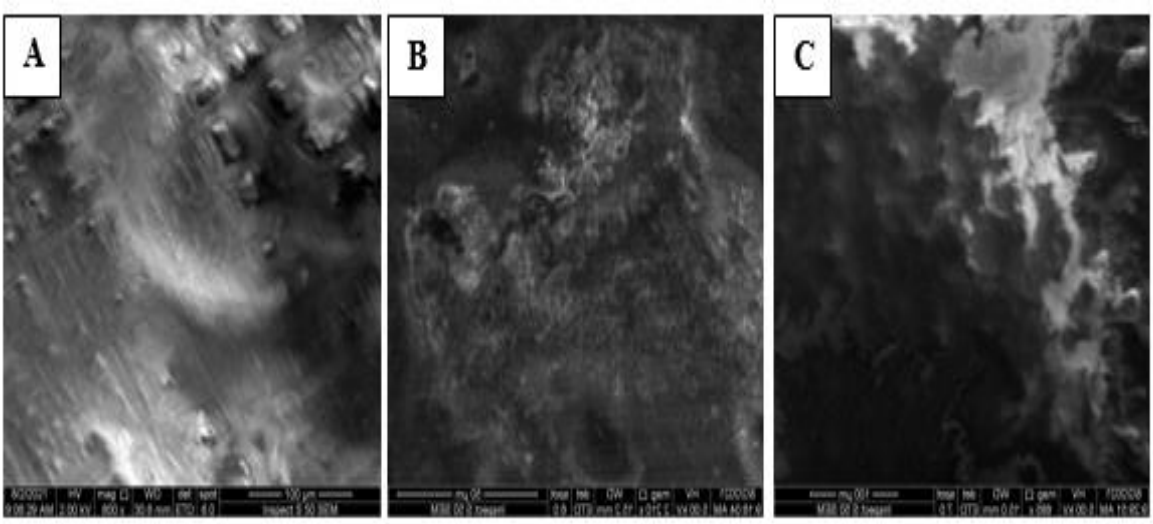

Figure 2. SEM micrographs PMMA samples: (A) Pure PMMA (B) PMMA/TiO_2NPs at 2wt% (C) PMMA/TiO_2NPs at 4wt%. scale (50 µm)

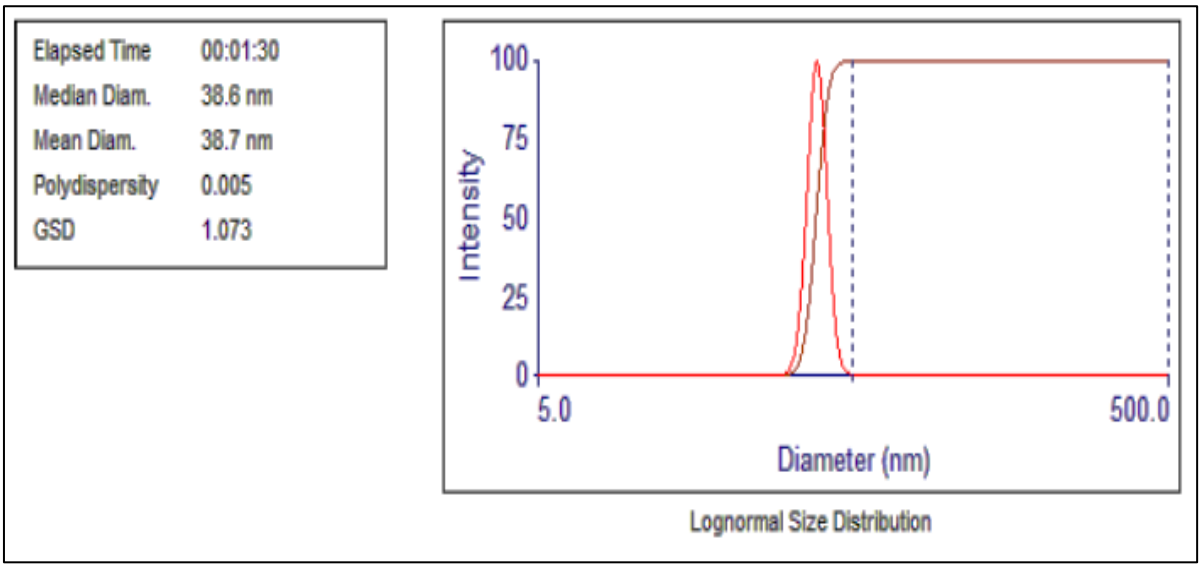

Figure 3. Particles size distribution of TiO_2NPs.

Energy Dispersive X-Ray Analysis is an analytical technique used to determine elemental or chemical characterization of the sample. Fig. 4 (A, B, C) presents the EDX measurement of the samples before and after reinforcement. The figure showed a result related to the ratio presence of TiO_2NPs for (A; pure PMMA sample, B;PMMA/TiO_2 2wt%, C;PMMA/TiO_2 4wt%) respectively.

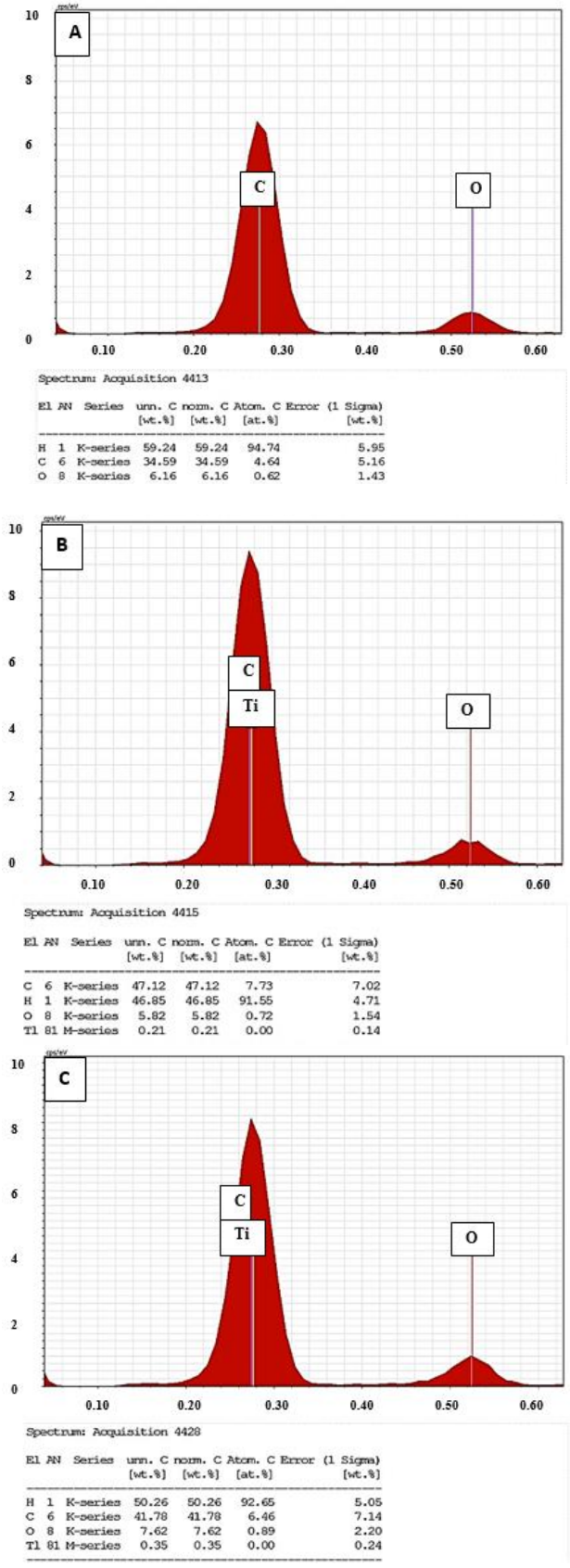

Figure4.(A, B, C) (A; pure PMMA, B;PMMA/TiO_2 2wt %, C; PMMA/TiO_2 4wt %) respectively.

Flexural Strength and Shear Strength Evaluation

Figs. 5 and 6 indicates the results obtained for the flexural strength and shear stress evaluation respectively, at the normal condition for all prepared samples (control case) Pure PMMA and Nanocomposite reinforced with titanium dioxide. Clearly, both results present better behavior for the nano-composite than the control PMMA. Both determined values show a noticeable increase as the additive percentage increase. The shape and size of TiO_2 spherical particles are used in this work to improve these properties. Also, these nanofillers could work as a stress transfer effect, which leads to enhance the polymer matrix properties[32]. These nanofillers become as an obstacle or barrier for rapid fracture and clinical failure[33]. Nanoparticles addition work on increasing the stiffness of the new nano-composite by restricting the matrix chain mobility. Also, the high interfacial shear strength between the PMMA matrix and nanoparticles could be another reason for such behaviour, due to formation of cross-links bonding that shield or cover the nano-additive so this shield works to prevents the propagation of the cracks inside the material. A good bonding between the pure PMMA and nano-additive[25,26] can also change the crack propagation. Furthermore, this improvement is related to the fact that both of flexural strength and shear stress value for TiO_2 NPs are much higher than PMMA matrix.

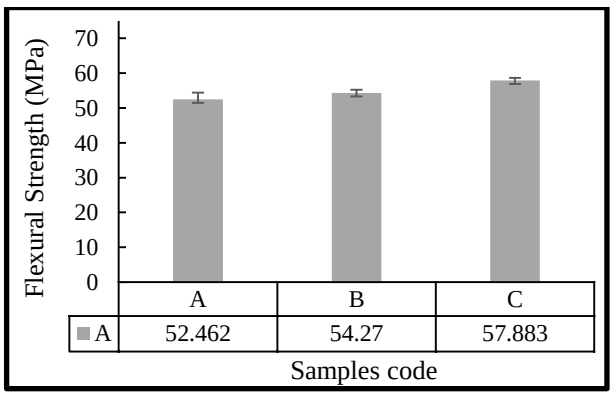

Figure 5. Flexural strength for Pure PMMA, PMMA with (2%, and 4%) of TiO_2 respectively

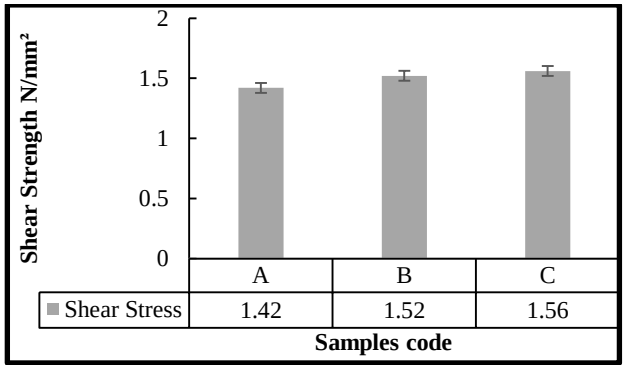

Figure 6. Shear stress for Pure PMMA, PMMA with (2%, and 4%) of TiO_2 respectively.

Three different groups according to additive percentage TiO_2 were prepared (0, 2%, 4%), pure PMMA sample and prepared nanocomposite samples were all exposed to water at three different temperature 20, 40, and 60 C°, and tested at three specific times 15, 30, and 45 min to find the effect of water temperature on some mechanical properties as stated before. A special water bath was used for each selected temperature to make sure the exact circumstances for the required period of each test which is 45 min. At 20 C° the results of flexural strength test are shown in Figures.7- 9. The value of F.S. of the prepared samples (control PMMA, 2% TiO_2 nano-composite and 4% TiO_2 nano-composite (test group) gradually decrease as the exposure time inside water bath increases until it reaches about 55.77, 52.7and 46.8MPa respectively after only45 min. This behavior could be explain due to poor workability, when water inters the material component that leads to decrease matrix - additive bonding which finally leads to increase porosity and increases water absorption. Increasing porosity results in strength reduction i.e. (increase materials plasticity)[34]. Also, the reduction of the measured F.S. value with incorporation nano fillers is due to poor interfacial adhesion with the matrix. Adding a suitable coupling agent may enhance surface interaction between both fillers and polymer matrix. Therefore, many micro cracks formed because of this and the stresses concentration happens on the ends of these micro cracks and spread gradually until the final failure occurs [35]. Same behavior was found in the case of immersion in to water bath at 40 C° and 60 C° as illustrated in Figures.8 and 9. It is noted that increasing the water temperature plays an important role in regression rate of the F.S. value determined. Generally, temperature is define as a measurement of kinetic energy so increasing the temperature works on increase polymer molecule energy which reflect on molecules movement and thus increase the water diffusion rate required for sample failure.

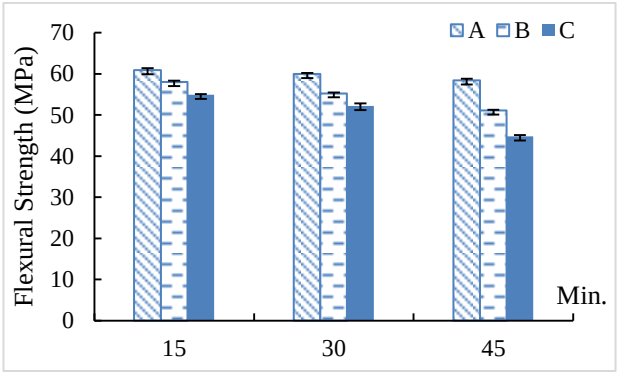

Figure 7. Flexural strength value as a function of time exposure to water at 20 C° for Pure PMMA, PMMA/ TiO_2 (2%, and 4%) respectively.

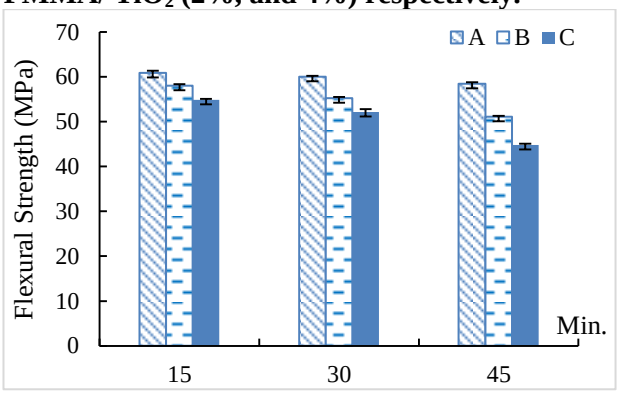

Figure 8. Flexural strength value as a function of time exposure to water at 40 C° for Pure PMMA, PMMA/ TiO_2 (2%, and 4%) respectively.

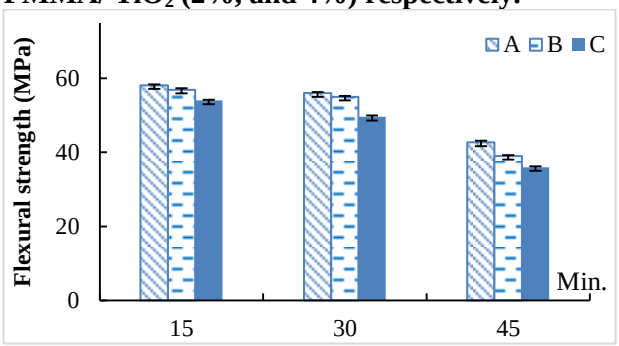

Figure 9. Flexural strength value as a function of time exposure to water at 60 C°for Pure PMMA, PMMA/ TiO_2 (2%, and 4%)respectively.

For shear strength measurement, Figures 10-12 illustrate a gradual decrease in the magnitude as the time of water bath increase for the three different temperatures (20, 40, and 60) C° respectively. The obtained results showed the lower value reached by the samples were at 60C°, the higher the water bath temperature is, the lower shear strength is measured. Water diffusion effects the samples as it works on material dissolution under test, which is attributed to final failure. Spreading the liquid inside the components of the polymer alternatively leads to breaking the bonding and emergence of bubbles that cause sample deformation easily and then final failure. There was

an obvious trend on shear strength decrease along with the increase of additive percentage of titanium dioxide.

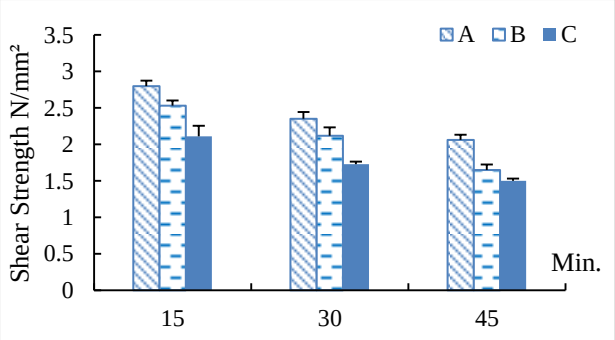

Figure 10. Shear strength value as a function of time exposure to water at 20 C° for Pure PMMA, PMMA with (2%, and 4%) of TiO_2 respectively.

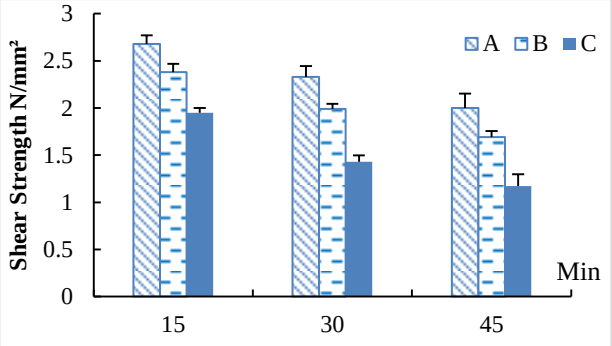

Figure 11.Shear strength value as a function of time exposure to water at 40 C°for Pure PMMA, PMMA with (2%, and 4%) of TiO_2 respectively.

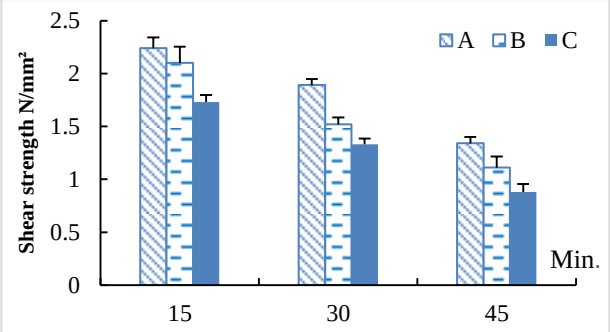

Figure 12. Shear strength value as a function of time exposure to water at 60 C° for Pure PMMA, PMMA with (2%, and 4%) of TiO_2 respectively.

Hardness (Shore D) Evaluation

A significant difference observed clearly between the determined value of pure polymer (control groups) hardness and the reinforced polymer (test groups) hardness, Figures 13 shows that the used additive enhanced this parameter in a good manner. It is clear that as the additive percentage increases the hardness value increase respectively. 73.2 was the value of the hardness in the control case, yet an obvious improvement was observed for the study case of 2% and 4% TiO_2 percentage (80.2 and 83.9) respectively. TiO_2 particles size utilized in this study with size is as tiny as possible for perfect manipulation. Subsequently, the oxide of metal fillers spreads through the polymer matrix in interstitial spaces of matrix particles and that leads to the heterogeneous mixing and prevent the segment chain displacement of polymers[36]. Interestingly, the nano additive improved this parameter slightly for the prepared nano-composite, good wet ability and bonding strength between these nanoparticles and the PMMA polymer increase the surface hardness by restricting the matrix chain motion along the stress direction [28,29].

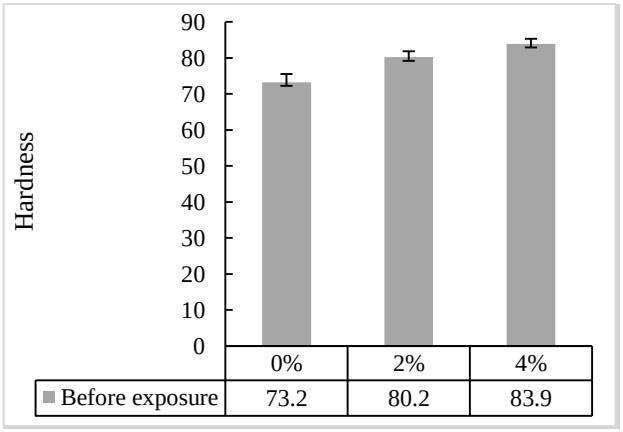

Figure 13. Shore (D) hardness value as a function of PMMA/TiO_2 NPs additive percentage.

Same test procedure was performed for the prepared nano-composite after exposure to water in three different temperatures. Table 2 presents the results of hardness value determined after (15, 30, 45) min of water exposure. Generally, it is obvious that for the three different temperatures (20, 40, and 60) C° as the temperature increases the value of hardness decreases. Raising the temperature affects samples hardness values, as hardness magnitude definition is the permanent deformation evaluation of the sample undergo an outside stress load.

As a result, for raising the temperature, material flexibility increases respectively due to the primary units motion and bound loosen between them which causes material resistance reduction and permanent scratching[37]. In addition, it is noted that as the time of water exposure increases the hardness value decreases. This could be as the effect of water diffusion inside the nano-composite materials, which works on formation of thin layer between the nanoparticles and the polymer matrix. Finally, the failure happens due to micro cracks formation inside the polymer matrix because of swelling of the tested sample. However, the hardness value of the prepared nano-composite with 4% then 2% of

titanium dioxide respectively still possess the higher amplitude than the control group at the same period of exposure due to the NPs used nature which is responsible for the improvement in material resistance for penetration.

Table 2 Hardness value for the samples under different temperature measured periodically.

Samples code	20C^0			40 C^0			60 C^0		
	15 min	30 min	45 min	15 min	30 min	45 min	15 min	30 min	45 min
A	78.1	77.1	76.5	79.9	68.2	65.0	66.7	66.0	61.6
B	81.8	81.1	78.1	83.8	76.8	75.2	80.0	75.6	74.4
C	84.8	81.8	79.5	80.7	77.5	77.0	80.6	76.7	74.1

Conclusion:

This paper research is carried out to investigate the effect of TiO_2 NPs in PMMA composite for denture base production, many achieved results could summarised as: the incorporation of TiO_2 NPs in PMMA composite is approved to improve both flexural strength and shear stress, significantly enhance the hardness value at the NC. In addition, there was a noticeable increase in the tested properties as the NPs ratio increase until 4wt.% the maximum value in the study yet, the incorporation of TiO_2 NPs to PMMA polymer presents a better hardness value after immersion at different temperature comparing to control PMMA. So, generally there was a significant effect of raising the temperature on accelerating water permeability within all the samples. Interestingly, the samples found to be crock free even after the exposure to 60 C° water bath, which referred to a good adhesion between the nano-composite components (matrix-additive). Finally, the results showed no sign for sample swelling or dimension changing.

Author's declaration:

- Conflicts of Interest: None.
- I hereby confirm that all the Figures and Tables in the manuscript are mine. Besides, the Figures and images, which are not mine, have been given the permission for re-publication attached with the manuscript.
- Author sign on ethical consideration's approval
- Ethical Clearance: The project was approved by the local ethical committee in University of Technology.

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تأثير درجة حرارة الماء على الصلادة وقوة الانحناء لـ(PMMA/TiO2 NPs) المستخدم في تطبيقات الأسنان

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الخلاصة:

تعتبر مادة PMMA (بولي ميثيل ميثاكريلات) من المواد الأكثر استخدامًا في تصنيع قاعدة طقم الأسنان نظرًا لخصائصها المثالية. على الرغم من أن المشكلة الرئيسية لهذا الراتنج هي التكسر المتكررة بسبب قوى المضغ الثقيلة التي تؤدي إلى حدوث تشقق مبكر وكسر في الاستخدام السريري. تم إضافة الجسيمات النانوية كمواد مالئة في هذه الدراسة لتعزيز خصائصها الميكانيكية المختارة. يقدم هذا البحث دراسة عملية على قوة الانحناء والصلابة (Shore-D) لمادة PMMA قبل وبعد استخدام مواد التقوية (TiO_2 NPs) وبمدى (2 و 4%) . تم فحص تأثير المادة النانوية المضافة في الظروف العادية وتحت درجات حرارة مختلفة أثناء التعرض للماء. أولا، قد تم إجراء الاختبارات على العينات المحضرة عند درجة حرارة الغرفة ثم بعد التعرض لحمام مائي عند درجة حرارة (20، 40، 60) م. تم اجراء فحوصات EDX, PSD, SEM للعينات في هذه الدراسة. على التوالي تم تقييم قوة الانحناء، إجهاد القص، قيمة الصلادة لجميع العينات، طبقت الاختبارات بشكل دوري عند (15، 30، 45) دقيقة. من المثير للاهتمام، قد وجد أنه في الحالة الطبيعية(NC) ، يؤدي تعاون (TiO_2 NPs) إلى تحسين الخصائص المحددة بشكل كبير. تقع الزيادة القصوى عند 4٪ بالوزن من إضافة (TiO_2 NPs) كما أظهرت النتائج أن زيادة درجة الحرارة تسرع من نفاذية الماء داخل جميع العينات وبالتالي تقلل من الخواص الميكانيكية. من الواضح، قياس قيمة صلادة أفضل للمتراكب النانوي المحضر مقارنة بالمجموعة الاساسية النقية عند نفس ظروف الاختبار.

الكلمات الافتتاحية: متانة الانحناء، PMMA لتطبيقات الاسنان، اجهاد القص، صلادة شور (d).