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# Nano-reinforcement effect on PMMA denture base mechanical properties after immersing in different solution

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## Abstract

Poly methyl methacrylate PMMA polymer has been used continually in dental application during the recent years. Yet, it is commonly known for its poor strength properties for long periods under pressure. The aim of this research was to improve the performance of PMMA denture base through the addition of different nanoparticles selected from artificial and natural sources. For comparison, Nano -particles from $Al_2O_3$ and crushed pistachio shell were utilised. (*1%, 2% and 3%*) were the weight fraction used in this study for both reinforcement types. In this work, a study and evaluation in of Compression Strength (C.S.) as well as Young's Modulus (*Y*) was done before and after exposure for special liquids. The new prepared composites were immersed in two type of liquids (vinegar and olive oil). Both physical values were determined periodically for three specific times (*10, 20 and 30 min*).All tests were carried out at room temperature. As a result a clear increase was obtained in the evaluated values of (C.S.) and Young's Modulus for the composite samples after the addition of both type of particles compared to the pure case. As the weight fraction was increased, these values also increased. It was found that the pure polymer (PMMA) lost almost half of the value of C.S. after 30 min of exposure in both liquid used. When immersing the prepared composites inside both liquids While the obtained results for pistachio shells particles showed slight increase after 10 min of exposure then a slight decrease through the time. Overall, the prepared composites for both type particles showed a better behavior after immersion in these liquids than the pure PMMA polymer. Also, a significant enhancement was clear with the determined value of Young's Modulus between the prepared composites compared to the non-reinforced PMMA in all situations.

**Keywords**: PMMA, Compression strength, Young's Modulus, Al2O3nanoparticles, pistachio shell particles, immersion, Nano composite material.

# تأثير التدعيم النانوي على الخواص الميكانيكية لبوليمر(PMMA) بعد الغمربمحاليل مختلفة

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## الخلاصة

في السنوات الاخيرةتم استخدام بوليمرالبولي مثيل ميثااكرليت كمادة اساس في صناعة قوالب الاسنان. ومن المتعارف علي هذا البوليمر ضعف خصائصه الميكانيكية عند استخدامه تحت الضغط لفترات طويلة. أن الهدف من هذا البحث هو تحسين اداء هذا البوليمرمن خلال اضافة دقائق نانوية مختلفة مستخرجة من مصادر صناعية وطبيعية ولغرض المقارنة استخدمنا دقائق الالومينا $Al_2O_3$ ومسحوق قشور الفستق وبثلاث كسور وزنية (1%,2%,3%) . وقد تم في هذا البحث دراسة متانة الانضغاط بالاضافة الى حساب معامل يونك قبل وبعد التعرض للغمر حيث تم غمرالمواد المتراكبة المحضرة في نوعين من السوائل (الخل ،زيت

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الزيتون)وبثلاث ازمان لكلا النوعين (10,20,30) دقيقة و بدرجة حرارة الغرفة . من نتائج البحث هناك زيادة ملحوظة بقيمة متانة الانضغاط ومعامل يونك للموادالمتراكبة المدعمة بكلا نوعي الدقائق مقارنة مع البوليمرالنقي,حيث ان زيادة نسبة التدعيم أدت الى زيادة القيم. ولقد وجد ان قيم متانة الانضغاط لبوليمر PMMAقد انخفض للنصف بعد مرور 30دقيقة من الغمر بكلا نوعي السوائل المستعملة.بينماالمادة المتراكبة المدعمة بدقائق الالومينا $Al_2O_3$ قد انخفضت بصورة ملحوظة مع الزيادة بزمن الغمر ، بينما في حالة مسحوق قشور الفستق كان هناك زيادة طفيفة يليها نزول بسيط بزيادة الزمن.بصورة عامة، المواد المتراكبة المحضرة بكلا نوعي الدقائق قد ابدت سلوك افضل بعد تعرضها للغمر في المحاليل مقارنة بسلوك بوليمرPMMA النقي. وهناك ايضا تحسن فعلي في قيم معامل يونك بين القيم المحسوبة للمواد المتراكبة والبوليمر النقي غير المدعم في اغلب الحالات.

## Introduction

Poly methyl methacrylate (PMMA) was selected as the most popular material utilized in synthetic prosthetic base material since it is introduction in 1937. It remains the most used of all polymeric denture bases[1].This polymer wield is used for fabrication of denture base which represent a removable replacement for lost teeth and the surrounding tissue[2]. It usually consists of poly (methyl methacrylate) (PMMA) as powder form and methyl methacrylate(MMA) as liquid form made by a heat cured polymerization technique[3]. The popularity of selecting PMMA material is because of its simple fabrication process, easy laboratory and clinical manipulation, low cost and light weight, colour matching ability, ease in finishing and polishing, excellent biocompatible material and good aesthetic appearance [1,4,5].

However, PMMA has limitation concerning the mechanical properties like low resistance to fatigue, low elasticity coefficient and low impact strength which lead to early crock and fracture in clinical use [6, 7, 8]. Generally, heavy occlusal forces induce ease fracture in denture base.Fractures normally happen in many cases like when the user applies high mastication force between upper jaw and mandible jaw [9]. Moreover, deformation effect could occur throughout time due to biting and mastication forces [10, 11].

Moreover, in order to get better efficiency for these prepared dentures it must have good mechanical properties so it can withstand heavy chewing forces inside the mouth [2, 5]. Using special materials as reinforcement to PMMA could overcome these disadvantages [4, 12, 13]. Hence, more understanding and definition of the required mechanical properties for such material and the proper procedures to improve such application performances are highly essential in dental world [14].

Recently, numerous studies were presented to improve the general properties of denture base materials through the addition of different fillers into PMMA [15, 16, 17, 18].These various additives include nanoparticles. the improvement of the Nano composite mechanical properties critically depends on the type of nanoparticles additive for preparing the Nano composite, especially the size, the type and even the distribution. The concentration and the interaction of these specific additives with the resin matrix are also essential for better properties. Nano particles integrate with the polymeric matrix to enhance most of the mechanical properties such as the rigidity, fracture toughness and other functional properties of the new Nano composite. The high surface area-to-volume is considered as an advantage, so it could be used to improve the mechanical properties of PMMA. Moreover, a better interfacial interaction is the result of this ratio and then a better enhancement in material performance is expected [19, 20, 21].

To enhance the mechanical properties of denture base some researches adopted the use of fillers and rubber particles [22]. Others used $ZrO_2$, $Al_2O_3$, and $TiO_2$ and found a better result with some properties [23]. Same results were introduced through using matrix of $ZrO_2/TiO_2$ [23], and$ZrO_2/ Al_2O_3$ [24]. Studies involve using $Al_2O_3$, $Zr_2O_3$, and SiO2 as Nano fillers with PMMA were presented [25, 26]. In denture base fabrication and interfacial silane, reformulation nanoparticles were greatly used [26].Mechanical strength like compression has a huge importance in the denture base fabrication. As mentioned before, these values for the pure polymer are not sufficient to maintain the longevity of the synthetic dentures. Most fractures occur due to many factors like poor fit of the dentures, poorly balance occlusion, and the frequent stress on the denture base for long period of use [27, 28, 29].

However, there is an essential need to experimentally study the advantage of using nanoparticles reinforcement with PMMA for denture base fabrication especially after exposure to different condition similar to what exposed inside the mouth. Hence, the main aim in this research is to fabricate PMMA based bio- composite reinforced with artificial and natural Nano sized particles, to study the behaviour of the prepared new Nano composites on compression strength property and for determining Young's Modulus values before and after immersion in special liquids. These additives were investigated to find out whether or not they improve mechanical properties compared to the pure PMMA.

## Experimental part

### Technique of samples preparation

In this study PMMA was utilized as a pure polymer and as reinforced through adding two types of nanoparticles to prepare our Nano-composites:

1-($AL_2O_3$) Nano particles
2-Pistachio shell micro particles**.**

### A- Preparation of $AL_2O_3$ Nano powder.

In this study PMMA utilized as pure powder polymer as indicate in Fig.1a and reinforced through adding two type of particles to prepare our composites:

### B-Preparation of pistachio shell powder

Pistachio shells were cleaned first with water and dried at room temperature. The shells then were grounded to smaller particles by manual hammering. The obtained powder was transferred to a mechanical Nano-grinding ball(Zirconia ball) which was used to crush the pistachio shell to a Nano sized powder, the duration of ball crushing was 2 hours.Figure-1(b, c, d) shows the preparation of pistachio shell powder from pistachio shell. The obtained micoparticles were then transferred to a special particle size (90-plus) analyzer to determine the average size of the final pistachio shell powder. Fig.2 presents the average particle size of the prepared powder, the average effective diameter for pistachio shell micro particles was (1.576) µm.

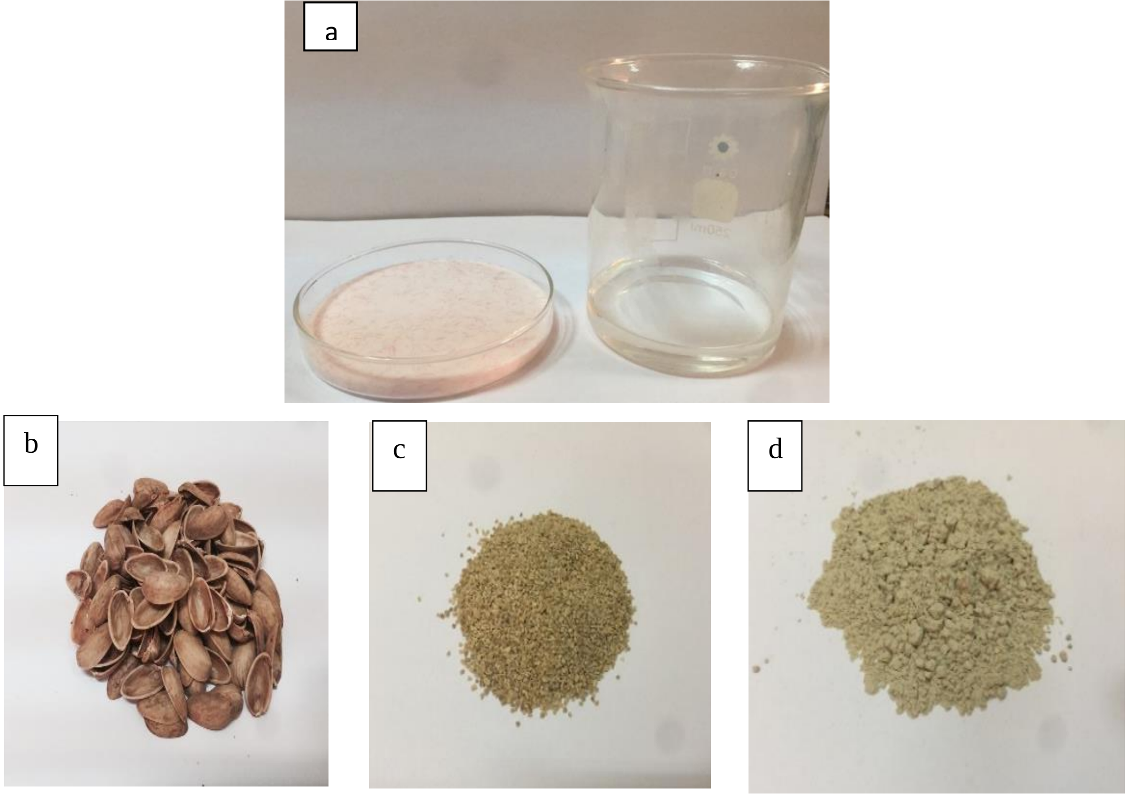

Figure 1-(a) pure PMMA powder and its monomer liquids (b) natural Pistachio shell, (c) Pistachio shell pieces after manual hammering, (d) Pistachio shell powder.

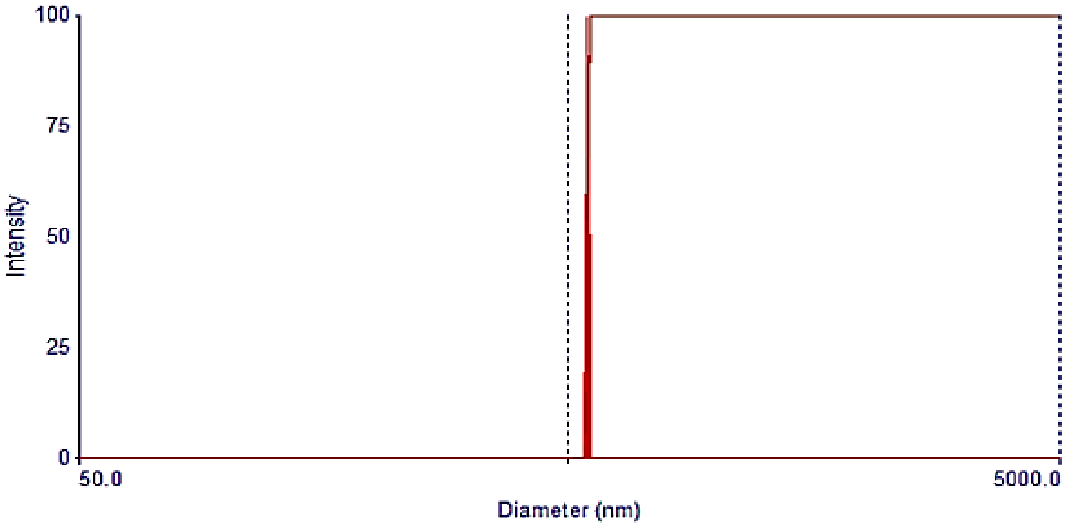

**Figure 2-**The lognormal size distribution obtained for Pistachio shell particles.

## C-Mould and Nano composite sample preparation

The required pure PMMA and reinforced PMMM with selected additives were mixed with weight percentages as shown in Table 1. Pure PMMA was made from Vertex™ Castavaria to prepare the all test specimens. The pure PMMA proportion for mixing weight ratios is usually about (50% to 50%) from polymer powder and monomer liquids (MMA). PMMA is moldable for a long period of time in cold curing, where the liquid of (MMA) was poured in clean and dry container (glass beaker). The prepared mixture must stirred using hand lay-up technique until the dough stage, the whole process is done at room temperature. Slow and continues mixing must be applied before the dough stage to avoid the formation of bubbles inside the mixture . The mixed powder was then poured from the mixing beaker to the prepared mould.The used mould made of glass with fixed dimensions of (15cm × 10cm × 0.4cm) and must have a glass plate to provide smooth sample surface by covering the mould top.For solidification purposes this mixture was left at room temperature for about (1hour). For post cure the cast sheet released from the mould and placed in an oven at (55°C) for another (1hour).

In this study, the required Nano-composite specimens were made from PMMA polymer with particles additive from Nano $Al_2O_3$ and micro particles from crushed pistachio shells. Using selected weight fraction (1%, 2% and 3%) the reinforcement process was made by hand lay- up technique, all the samples prepared at room temperature. It was mixed until all particles were uniformly distributed in the PMMA powder. Table 2 explains the 6 different mixtures prepared for this research with full details.

In Figure-3 the prepared samples were presented (Pure PMMA), (PMMA + $Al_2O_3$) and (PMMA +pistachio shell) with the selected weight fraction. It is clear from the figure that the color of the samples depend on the additive percentage, the increase of weight fraction of both particles change the color from transparent pink to light pink and light beige .Finally, the prepared composites plates were cut into the mentioned dimension above based on ASTM standard for the compression strength.

**Table1-**Composition of different particles additive reinforced composite

| Percentage of Additive (%) | weight percentage of additive(g) | weight percentage of PMMA+MMA liquid(g) |
|---|---|---|
| 0 | 0.00 | 53.46 |
| 1 | 0.53 | 52.93 |
| 2 | 1.07 | 52.30 |
| 3 | 1.60 | 51.86 |

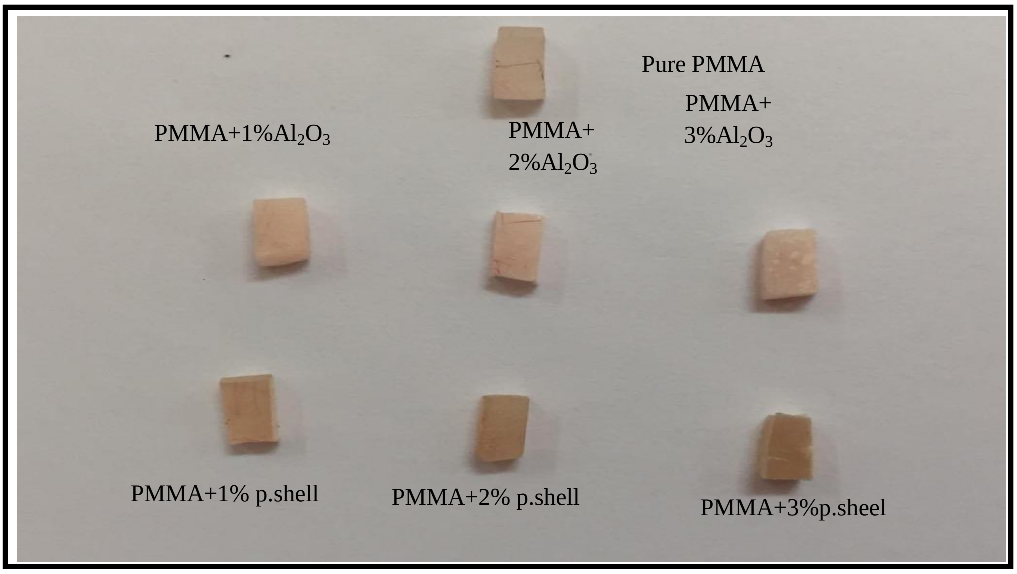

**Figure 3-**Compression samples of the prepared PMMA pure and composite reinforced by both ($Al_2O_3$) and (pistachio shells) particles respectively after cutting to the required dimension.

## Compressive strength test

This test was carried out using a hydraulic piston device ( ley Bold Harris, No. 36110) as showed in Figure-4. Compression strength (C.S.) is the value determined for the prepared samples which represent the maximum stress that the material possesses before the final failure; In another way it is the maximum stress for a rigid material under longitudinal compression. The prepared samples must be cut according to (ASTM-D695) standard and the C.S. can be determined mathematically as:

C.S. = Maximum Stress ($\Delta\partial$) = $\frac{\mathrm{Fmax}}{\mathrm{A}}$

Where ($F_{max}$) represent the maximum load (N) until the failure point of the sample.

(A) is the cross section area ($mm^2$)

To evaluate the Young's Modulus:

Young's Modulus E=$(slope)\frac{\Delta\partial}{\Delta\varepsilon}$, where Strain (ε) =$\frac{\Delta L}{L}$ .

ΔL is the elongation of the sample, L is the original length for the sample (mm).

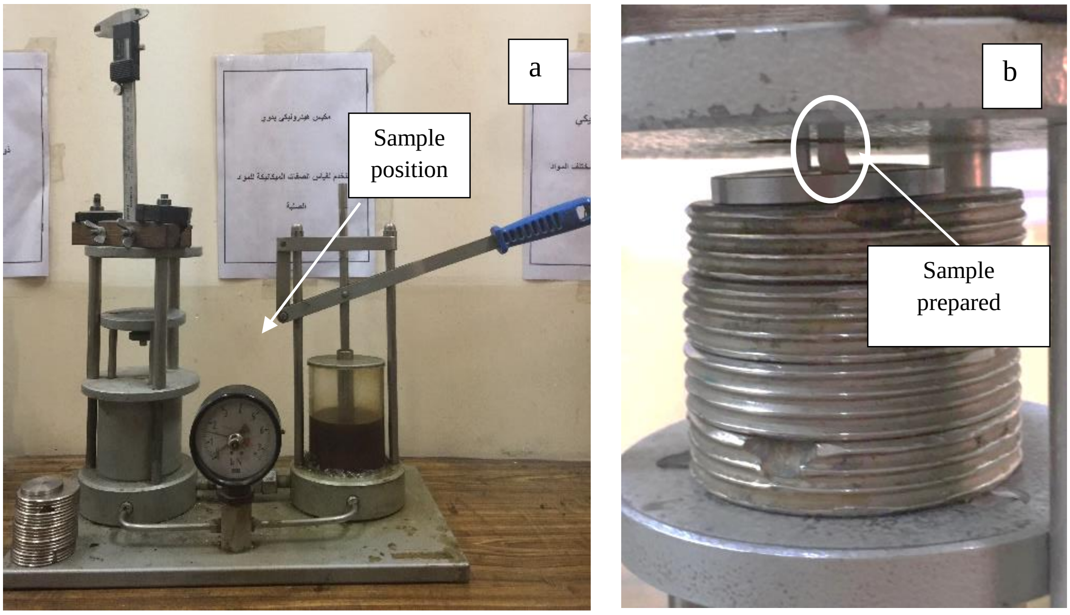

**Figure-4**(a) Photograph of Compression device (b) sample under compression test after failure

## Results and discussion:

### I. Compression strength evaluation

Figure-5 shows the results obtained for evaluating C.S. for all prepared samples (pure PMMA, composite reinforced with both type of particles before immersion process). Clearly both types of additive present better results for the composite than that forthe pure PMMA, As the particles weight fraction increases the value of C.S. increases. This could be explained that these fillers become as an obstacle or barrier for rapid failure [30]. Also, the reason of such behavior is the high interfacial shear strength between the PMMA matrix and the particles because of the formation of cross-links bonding which shield or cover the particles in a way that it prevents the propagation of the cracks inside the material. in addition, the propagation of the crack can be changed by good bonding between the PMMA matrix and particles [31, 32]. These additions of particles work on increasing the stiffness of the prepared composite by restricting the matrix chain mobility [33].

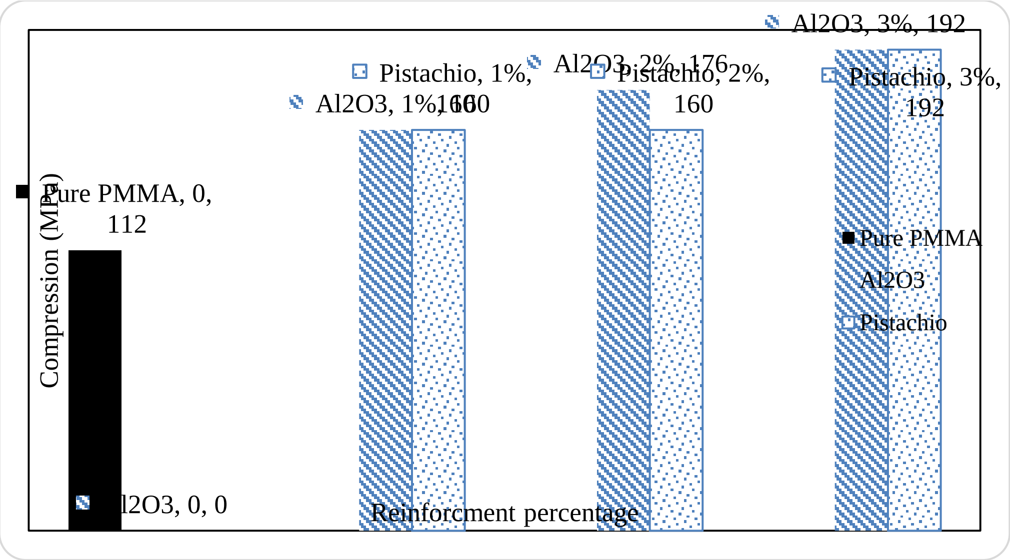

**Figure 5-**Comparison of Compression strength for Pure PMMA, PMMA with (1%, 2%, and 3%) of $Al_2O_3$ and pistachio shells micro particles, respectively.

After immersion, the samples with the different weigh fractions were examined using vinegar. The results are shown in Figures-6(a and b), the C.S. values of the pure PMMA changes gradually with the immersion time inside the liquid reaching between (64-96 MPa) after 30 min. This behavior could be overcomed through the addition of the nanoparticles $Al_2O_3$ and micro particles pistachio shells (Figure-6a); it is obvious that the prepared Nano composite using $Al_2O_3$ has higher C.S. values than that of the pure one in the same situation . C.S. values increased as the reinforcement increased. Yet, the overall results showed also gradual decrease of C.S. as the time of increased for all samples. This could be due to the effect of liquids on the pure PMMA and other Nano composite Liquids molecules enter the samples in a way that it works on dissoluting the polymeric material under test which attributes to final failure. Spreading of the liquid through the components of the polymer leads to breaking the bonds and formation of bubbles that deform the sample easily and the lead to the final failure [34].Moreover, the increase of immersion time leads to the decrease of the C.S.values due to the increase of the material plasticity. Obviously, PMMA nano-composite possess higher C.S. than pure PMMA at the same situation after immersionThis behaviour is caused by the effect of the liquid that leads to the release and removal of any residual monomer, as well as residual stresses which adversely affect the compressive strength of the pure PMMA and PMMA Nano-composites[35]. It is clear from Fig.6b that Pistachio shells micro particles composite presents the same behavior by increasing the determined value of C.S. compare to the value of the pure PMMA at the same situation. Interestingly, these samples showed slight decrease or even remained unchanged as the time of exposure increases due to the additive nature.

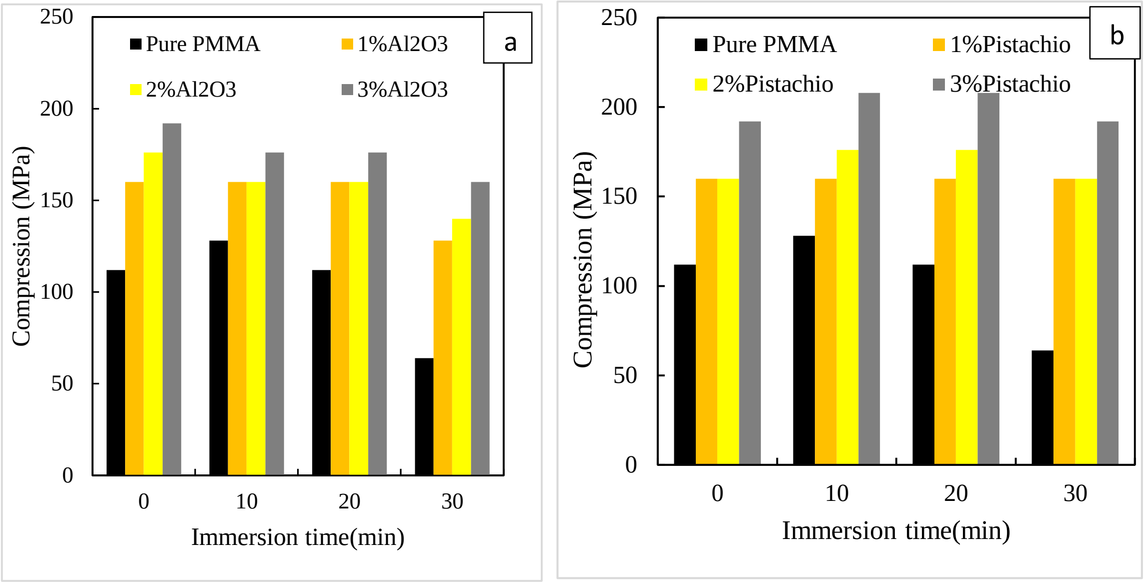

**Figure-6**(a and b).Compression strength values as a function of immersion time using vinegar

The determined value of the C.S. after immersion in olive oil for all prepared samples is illustrated in Figure-(7a and b), Figure-7a clearly shows slight decrease or the same value of the C.S.value when using $Al_2O_3$ as additive.The same trend was obvious in the case of using the natural shell powder as shown in Fig.7b. This slight decrease is related to the increase of the exposure time .As mentioned in the previous part many reasons are behind the better performance of composite material in the determined value of C.S. from the pure PMMA at the same situation. The increase of the adhesion bond is also an important factor of strengthing, which happens because of the absorbed liquids between the pure polymer and the particles additive which increases the interfacial shear strength between the matrix and additive. So, strong physical bonding occurs in the composite that requires high compressive strength to break it [36].

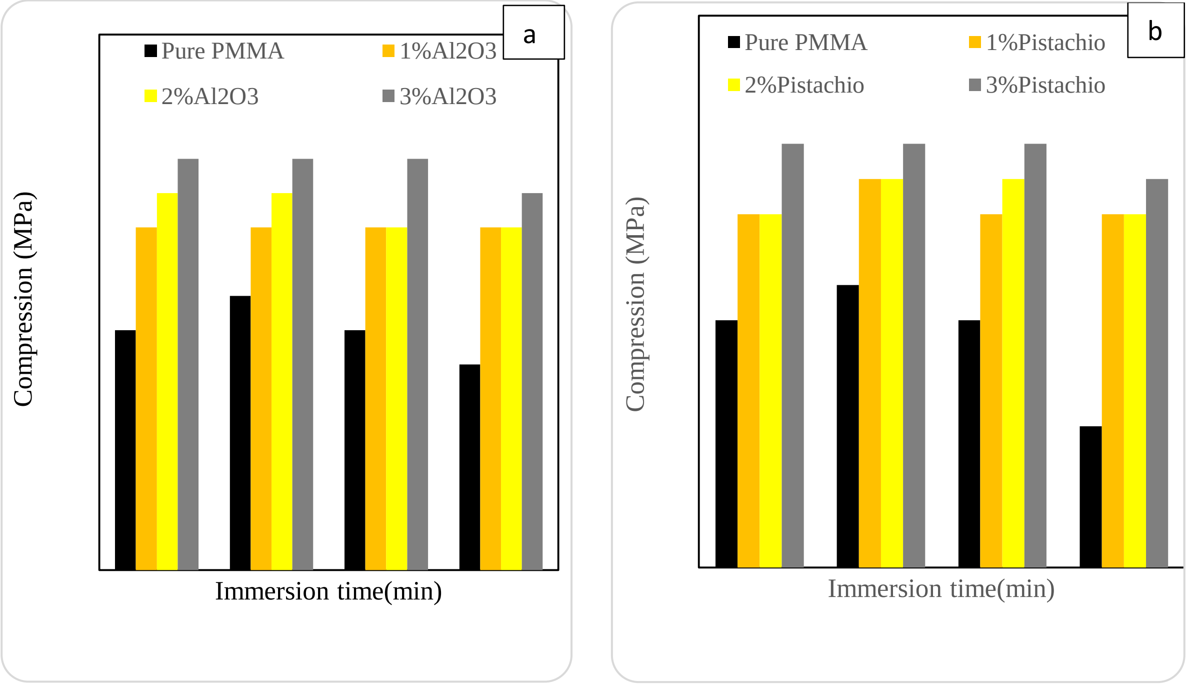

**Figure7-**(a and b): Compression strength values as a function of immersion time using olive oil.

## II. Young Modules evaluation

Interestingly, it was observed that after the addition of the prepared particles, the value of Young'sModulus increased gradually with the increase of the weight fraction. As shown with Figure-8 , both composites prepared presents almost similar trend. The maximum values of Young'sModulus measured for the 3% of pistachio shells micro particles of $Al_2O_3$ nanoparticles were (620.32MPa) and (600.23 MPa). respectively. This increase of (*Y*) could be explained as due to the particles additive nature (tough). This characteristic leads to the decrease of the elongation before final failure(break) and thus increase the determined value of Young'sModulus. The composite with pistachio shells particles showed higher value than the other composite due to the stiffness that these particles possess. The higher the stiffness of the particles used in the prepared composite the higher the Young's Modulus for the same polymer matrix [37].

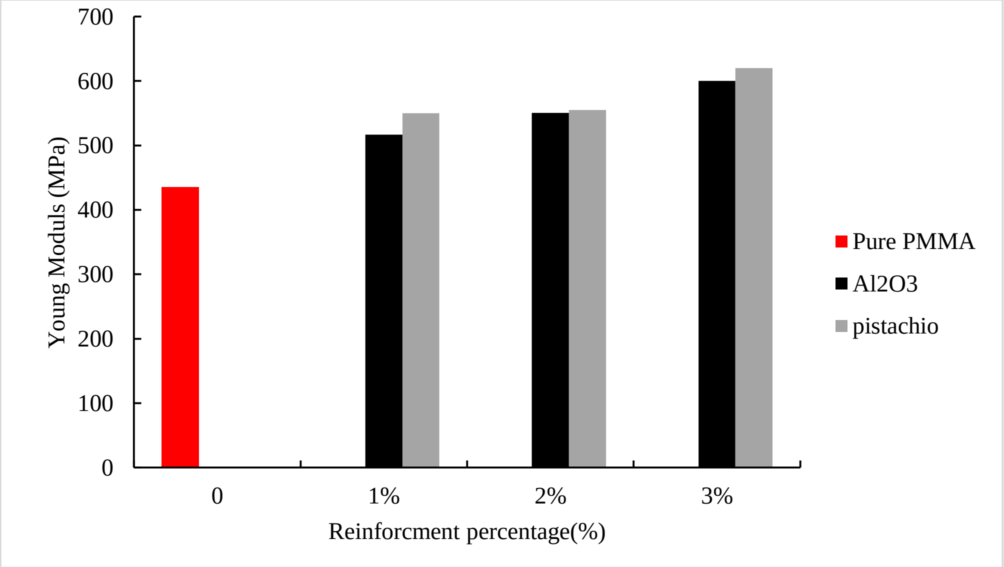

**Figure 8-**Young's Modulus values as a function of fraction weight of both type of additive.

This value was also determined for all samples after immersion in both liquids (vinegar and olive oil).Figure-9 and Figure-10) present Young's Modulus as a function of immersion time. Both prepared composite (PMMA/$Al_2O_3$, PMMA/ pistachio shell) with the same percentage mentioned before where tested against the pure PMMA polymer. Both Figures showed a noticeable decrease in (Y) value for all samples after immersion in vinegar and as immersion time increases. Same trend was obvious in both liquids used; this reduction in Young's Modulus could be related to the absorption and diffusion characteristics of liquid molecules inside the tested samples and the formation of a thin layer between the additive and matrix [38]. Figures-(11, 12) clearly present the same decrease behavior after immersion inside Olive oil for both prepared composites. The reason behind this decrease is due to the liquids diffusion inside the composite materials which is governed by three mechanisms: First, diffusion of liquid molecules inside the gaps of the polymer chains. Second, the capillary transport into the gaps and its spread which creates an interface between the matrix and the additive, The last mechanism involves the transporting of micro cracks in the polymer matrix as a result sample swelling that leads to final failure [39].

Good enhancement in Young's Modulus value was obvious between the pure case and the reinforced case of PMMA at the same situation (same immersion time). The higher the additive weight faction the higher the determined value detected. Fig.13 presents Young's Modulus as a function of the additive weight fraction after maximum immersion time (30 min) in both used liquids. The values obtained were (519.22 MPa) and (575.2 MPa) for $Al_2O_3$ and pistachio shells particles, respectively in the case of using the vinegar compared to the value determined of the pure PMMA at the same period which was (300.4 MPa). While Young's Modulus values in the case of using olive oil where (519.4 MPa) and (580.9 MPa) for $Al_2O_3$ and pistachio shells particles, respectively, while the value of the pure PMMA decreased significantly to (355MPa).

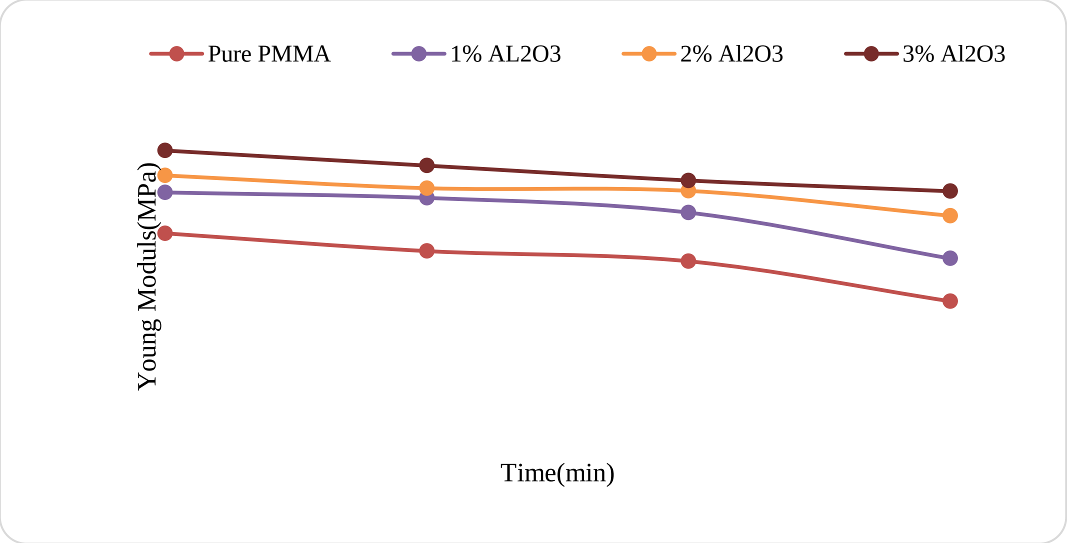

**Figure 9-**Young's Modulus values as function of immersion time in vinegar for Nano composite base $Al_2O_3$.

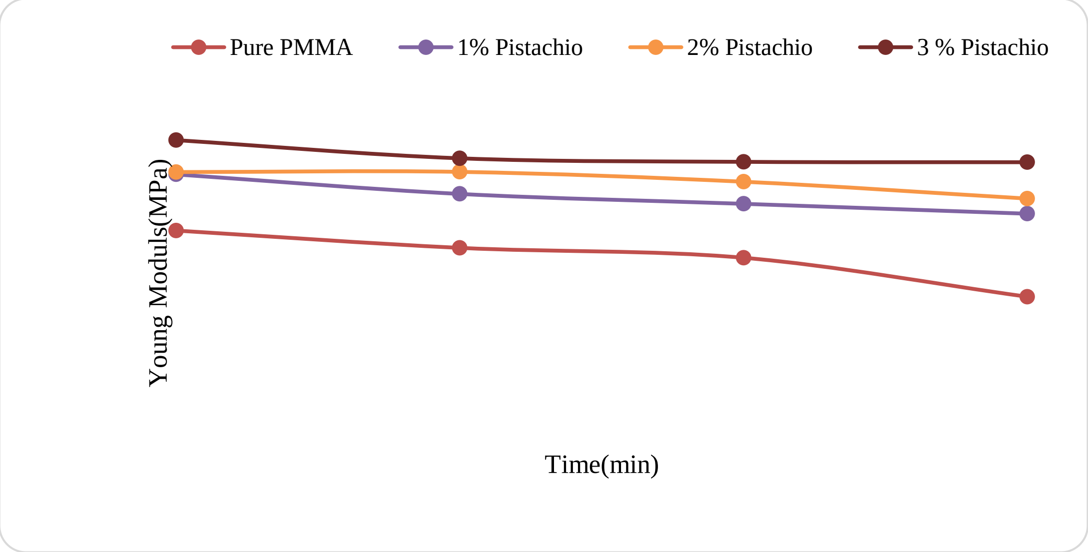

**Figure 10-**Young's Modulus values as a function of immersion time in vinegar for composite base Pistachio shells powder.

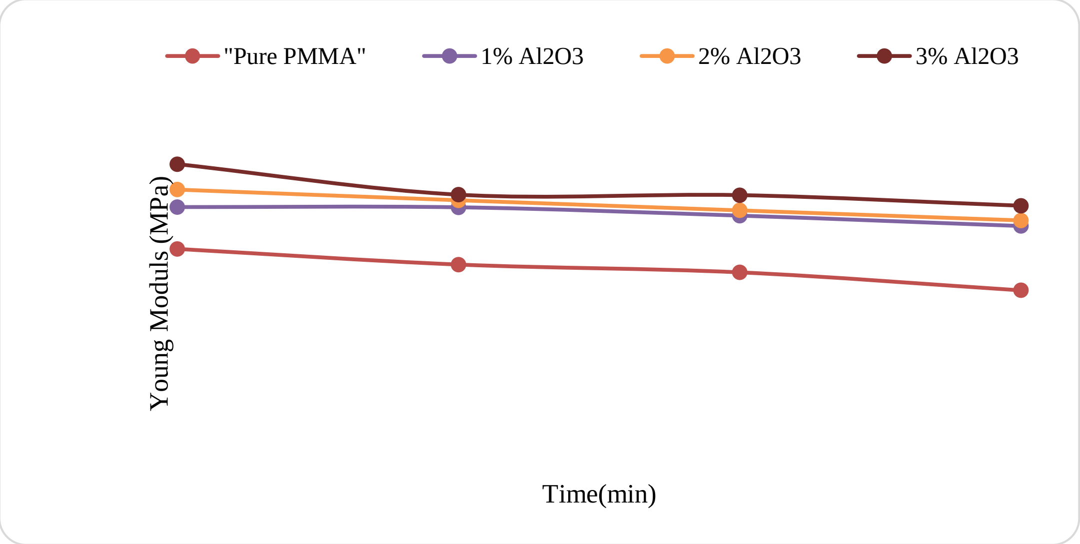

**Figure11-**Young's Modulus values as a function of immersion time in olive oil for Nano composite base $Al_2O_3$.

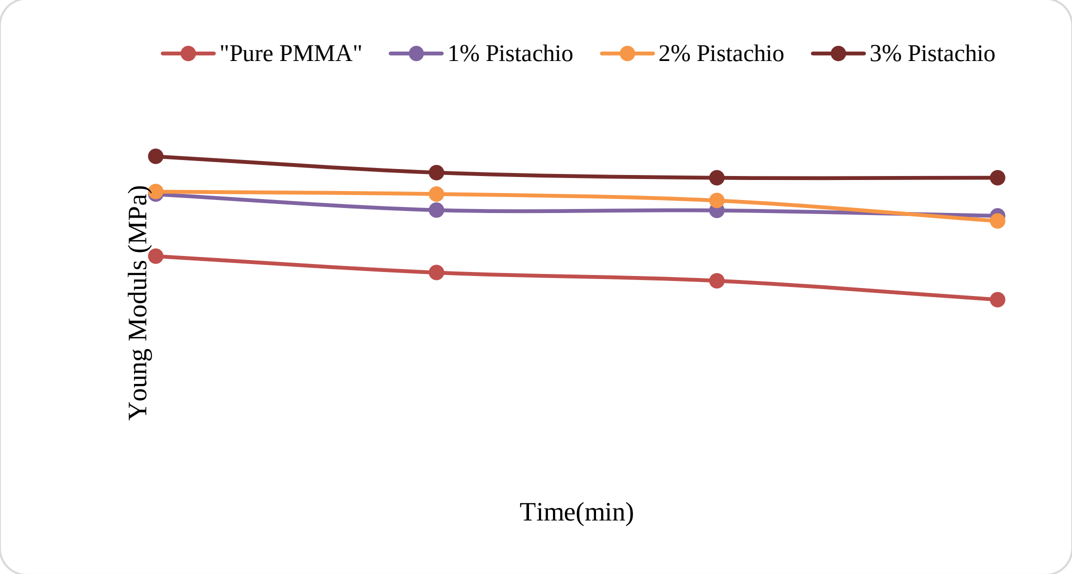

**Figure 12-**Young’s Modulus values as a function of immersion time in olive oil for composite base Pistachio shells powder.

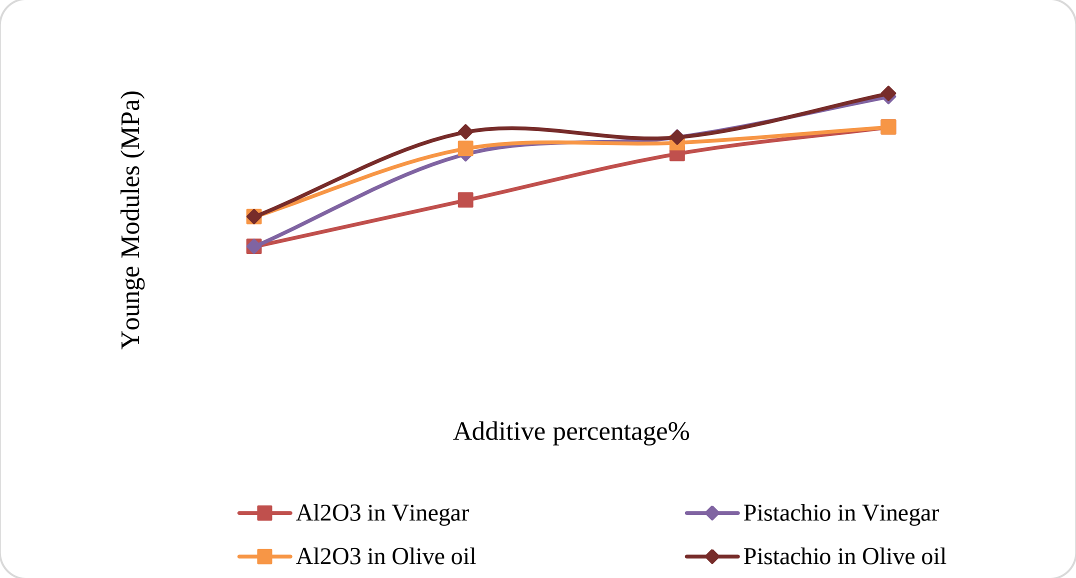

**Figure13-**Young’s Modulus values as a function of additive percentage at maximum immersion time in both liquids.

## Conclusion

This study was conducted to evaluate both values of Compression strength and Young’s Modulus , before and after immersion in selected liquids , for some prepared samples (pure PMMA polymer, prepared composite of PMMA and both $Al_2O_3$ and crushed pistachio shells ds. In this research several points were concluded:

- All prepared composites with both additive types showed better results of both C.S. and Young’s Modulus.
- The improvement in the value of C.S. and Young’s Modulus increased gradually with the increase in weight fraction of additive. This research suggest that the case of 3% weight fraction of both additives can give very good values compared to all other samples, (192 MPa) for C.S. and (600.23-620.32 MPa) for Young’s Modulus .
- After immersion the value of C.S. of prepared composite showed higher result than the pure case (160-176 MPa) for $Al_2O_3$ sample and(176-192MPa) for crushed pistachio shell samples.

- After immersion, the value of Young's Modulus for all composite samples presented better results than the pure PMMA case, (519MPa) and (575-580MPa) for both $Al_2O_3$ and crushed pistachio shells powder ,respectively after maximum exposure time.
- All samples suffered a gradual reduction of the determined values. Interestingly, the declined trend for the pure case was higher than that of the prepared composite due to the additives nature.

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