DOI: http://dx.doi.org/10.30684/etj.37.12A.5

**J.K. Oleiwi**
University of Technology
Materials Eng. Department
Baghdad, Iraq
130041@uotechnology.edu.iq

**Q.A. Hamad**
University of Technology
Materials Eng. Department
Baghdad, Iraq
qah84@yahoo.com

**N.N. Kadhim**
University of Technology
Materials Eng. Department
Baghdad, Iraq
Wnzn.love@gmail.com

Received on: 16/06/2019
Accepted on: 06/07/2019
Published online: 25/12/2019

# Study Compression, Hardness and Density properties of PMMA Reinforced by Natural Powder Used in Denture Base applications

***Abstract***- *This research had been done to investigate the effect of adding natural powder of Pistachio Shell to PMMA, which popularly used in denture applications. The powder added in different weights fraction (3%, 6%, 9%, and 12%), and different average particle size (53µm, 106 µm, 150 µm, and 212µm %), and studying Compression Strength, Surface Hardness, and Density properties. Hand Lay-Up represented the method used to prepare the specimens in this research. The results were statistically analyzed by SPSS (one-way ANOVA) to determine the mean value and showed a significant difference for each particle size. The highest value of compression strength and surface hardness of PMMA composite specimens happened at (9%wt.) of the filler particles. Also the results represented that the density values for the composite specimens are increased with increasing the weight fraction of the filler particles.*

***Keywords***- *PMMA, Pistachio Shell Powder, Compression strength, Density, Hardness, Bio composite.*

**How to cite this article**: J.K. Oleiwi, Q.A. Hamad and N.N. Kadhim, "Study Compression, Hardness and Density properties of PMMA Reinforced by Natural Powder Used in Denture Base applications," *Engineering and Technology Journal*, Vol. 37, Part A, No. 12, pp. 522-527, 2019.

## 1. Introduction

Researchers and scientists concern about petroleum depletion pushed them to think about an alternative materials, so a tendency towards the natural material such as (pistachio shell, bamboo, coir, rise husk… etc.) was increased in recent years [1]. Where the natural materials used as reinforcing material because of their advantages over the synthetic one, that characterized by their low cost, high density, renewability, biodegradability, and high degree of flexibility in the work. Moreover, the usage of natural materials provides a healthier working conditions [2]. Although the advantages of natural materials, there were disadvantages, where they have poor mechanical properties comparison with synthetic one and high affinity to moisture absorption because of hydrophilic nature which is undesirable for most properties especially dimensional stability [3], and this made the chemical treatment necessary to improve the composite properties by reducing the hydrophilic of natural material which in turn reflect on another properties such as dimensional stability and the adhesion between the matrix and reinforcing material. Hatim et al. were studied the effect of Genuine Nigella sativa and Thyme as oil to denture base resin in different percentages of (0.5%, 1%, 1.5%, and 2%) by weight and the (transverse strength, indentation hardness, color property, residual monomer, dimensional accuracy, porosity, measurement of IR spectra, and anti–microbial–sensitivity tests) were determined. The results were as the following: the additives of natural oil of Nigella sativa and Thyme with concentration of 1.5% were recommended to give proper properties, as antimicrobial after curing of the acrylic resin denture base, but Thyme oil showed no effect on the color after curing in relation to Nigella sativa [4]. Mansour et al. estimated the effectiveness of reinforcing by mica on the mechanical properties (three-point bending and micro hardness) of Poly (Methyl Methacrylate) resin (PMMA). Two micas were tested: W200 and P66 with average particle sizes of 131µm and 30µm, respectively. With volume fraction of (10% and 20%) in individually form, the following results obtained: the addition of mica resulted in decline the flexural strength, while the micro hardness increased. In addition ANOVA and Tukey tests were used for statistical analyses (Alpha = 0.05) [5]. Oleiwi et al. were evaluated the effect of Siwak and Bamboo fibers as reinforcing materials on impact and compression strength of denture base resin. The additives were in three percentage (3,6,9%) by weight, and they concluded that the impact strength of PMMA acrylic resin decreasing with increasing the weight fraction of reinforcing material while the compression strength increased. Increasing the fiber length for both types resulted in increasing the impact

strength and decreasing compression strength [6]. Okeke, et al. studied the flexural and impact strength of denture base resin reinforced by Hibiscus sabdariffa fiber with different weight fraction as (2.5, 5, 7.5, 10wt%) the results showed that the reinforcement by Hibiscus sabdariffa improved flexural strength and strength the denture base resin and the specimens reinforced by (7.5 wt%) to represent the highest value. Statistically ANOVA and Bonferroni tests showed significant differences among the groups [7]. Fahmi and, Ebrahim, have evaluated the influence of reinforcement of heat-cure poly (methyl methacrylate) resin by polypropylene fibers with percentage of 10% by weight, on Flexural Strength, Fracture Toughness and hardness properties. The results showed that by addition of polypropylene fibers lead to increase the mechanical properties of heat- polymerized acrylic resin. The results were statistically analyzed by one-way analysis of variance (ANOVA) and Tukeys test [8]. The aim of this work is to estimate the compression, hardness and density of PMMA resin after reinforcing by Pistachio shell powder in different weight fraction and different average particle size.

## 2. Materials and Method

*Pistachio Shell powder0.3line*

Figure 1, A & B shows the Pistachio Shell that was used as reinforcing material (powder) in four percentages (3%, 6%, 9%, and 12%) by weight, and four different average particle sizes of (53µm, 106 µm, 150 µm, and 212 µm) in heat cure PMMA matrix material, to prepare bio composite specimens of prosthetic denture base.

**A** **B**

**Figure 1: Illustrate Pistachio Shell A-Before and B- After grinding.**

## 3. Alkali Treatment

The natural powder was soaked in alkali solution consist of 5% (w/v) of (NaOH) in distilled water, at room temperature ($25^0$C) [9], 1:30(w/v) represent the proportion of (powder /liquor), the treated powder then washed for several times by distilled to remove the remaining (NaOH) on its surface, until reach neutral state (pH=7), after that the powder had dried at room temperature for five days, and finally put in furnace at (50-$60^0$C) to ensure that it dried completely.

## 4. Preparation of Bio Composite Specimens

An electronic balance with precision (0.0001) was used for weighting the selected percentages of pistachio shell powder as reinforcing material. Pistachio shell powder was added to PMMA powder and mixed together till reach to homogenous mixture before adding to MMA liquid to make bio composite specimens. The liquid monomer (MMA) and acrylic powder (PMMA) mixed according to standard ratio (2.25:1) as powder to liquid, 2.25 represents the weight ratio of PMMA powder, and 1 represents the weight ratio of liquid monomer. Specimens were formed by pressing suitable amount of dough in each mould cavity, then the mould covered by metallic plate fixed by 10 screws to obtain the pressure that is required to form the specimens, which is 2.5 bar, Figure 2 shows the metallic mould that is used in this research. Then the tight mould was placed in path water where the water temperature rose gradually until reach 100C˚during 2hr, the purpose of this behavior is to complete the polymerization process of composite specimens, and enhance the physical properties. After these steps, the mould was left in the path water in the open air, where it was cooled slowly in order to complete the polymerization process, and reach for the lowest level of the residual monomer. Then the specimens were removed from the mould, with very smooth upper and low surfaces, then were subjected to finish process to be ready to the subsequent tests.

## 5. Experimental Tests

*I. Compression Test*

The compression test of the composite specimens was done according to (ASTM D695) by using the same device that used in tensile test at material engineering department in across head of (5mm/min), the compressive load was applied gradually until the fracture of specimen occur [11].

*II. Hardness Test*

The hardness of pure and composite specimens where done by using the Dorumeter hardness type (Shore D) according to the standard (ASTM D2240) were be tested. The applied load was (50N) and the depressing time equal to (15sec) [12].

*III. Density Test*

The density of the composite specimens for this test was carried out based on Archimedes method according to (ASTM D 792), in this test the specimens were weighted in the air, and weighted during immersion in distilled water at room temperature, three digits balance (PS360/C/1) were used for weighting in this study [13].

## 6. Results and Discussion

*I. Compression Test*

As shown in Figure 2, the compression strength of PMMA resin increased as the weight fraction of Pistachio Shell powder is increased and reaches its maximum value at (9%wt.), the reason behind this behavior is may be due to the good compression strength of the used powder. Also it may be resulted from the ability of Pistachio Shell powder to strengthen the resin and enhance the interfacial particles –matrix bonding which in turn prohibit the propagation of crack [14], [16], also from the same figure it notice that, the compression strength of PMMA composite specimens decreased when the weight fraction of Pistachio Shell powder exceed the (9%wt.), the lack in wettability and weakly physical bonds accompanied the increasing in the weight fraction may be responsible for this behavior. Also this behavior may be resulted from that the PMMA chains will separated due to the excess of Pistachio Shell powder which in turn leads to weaken the mechanical properties of the resulted composite.

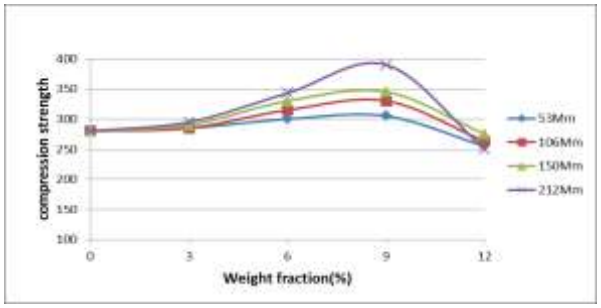


**Figure 2: The Compression Strength of PMMA Acrylic resin as a function to Pistachio Shell particles content.**

The statistically results of compression strength in Table 1 are illustrated that the mean values for all specimens are higher than that of neat PMMA, except the specimens reinforced with 12%wt. for all average particle sizes The greatest value was obtained at the specimen reinforced by 9%wt. of average particle size 212μm; all groups have been shown very high significant difference (VHS) ($p \leq 0.001$) as compared with neat PMMA..

**Table 1: Statistical Analysis of Compression Test.**

| | Groups | N | Mean | Std. Deviation | Std. Error | Minimum | Maximum | P-value |
|---|---|---|---|---|---|---|---|---|
| A53 | 0 | 3 | 280 | 4.359 | 2.517 | 275 | 283 | |
| | 3% | 3 | 285 | 3.464 | 2.000 | 281 | 287 | |
| | 6% | 3 | 300 | 9.165 | 5.292 | 292 | 310 | .000 VHS |
| | 9% | 3 | 305 | 6.245 | 3.606 | 298 | 310 | |
| | 12% | 3 | 255 | 6.083 | 3.512 | 248 | 259 | |
| A106 | 0 | 3 | 280 | 4.359 | 2.517 | 275 | 283 | |
| | 3% | 3 | 285 | 5.292 | 3.055 | 279 | 289 | |
| | 6% | 3 | 315 | 3.464 | 2.000 | 311 | 317 | .000 VHS |
| | 9% | 3 | 330 | 3.464 | 2.000 | 326 | 332 | |
| | 12% | 3 | 263 | 4.359 | 2.517 | 258 | 266 | |
| A150 | 0 | 3 | 280 | 4.359 | 2.517 | 275 | 283 | |
| | 3% | 3 | 290 | 6.083 | 3.512 | 283 | 294 | |
| | 6% | 3 | 330 | 3.464 | 2.000 | 326 | 332 | .000 VHS |
| | 9% | 3 | 345 | 3.464 | 2.000 | 341 | 347 | |
| | 12% | 3 | 275 | 5.292 | 3.055 | 271 | 281 | |
| A212 | 0 | 3 | 280 | 4.359 | 2.517 | 275 | 283 | |
| | 3% | 3 | 295 | 4.583 | 2.646 | 291 | 300 | |
| | 6% | 3 | 343 | 5.000 | 2.887 | 338 | 348 | .000 VHS |
| | 9% | 3 | 390 | 5.568 | 3.215 | 385 | 396 | |
| | 12% | 3 | 250 | 7.810 | 4.509 | 245 | 259 | |

*II. Hardness Test*

Figure 3 clarify the relationship between hardness and weight fraction of Pistachio Shell powder, an increasing is observed in the hardness values with increasing the weight fraction of Pistachio Shell powder and the highest values obtained at (9%wt.). This may be due to the using stiff and hard filler particles as compared with the matrix polymer so the stiffness and hardness of the composite material will increase [17]. Also the increase in hardness may be related to the alkali treatment of particles which is resulting in enlarge the wettability and the strength of bond between the matrix and the Pistachio Shell powder which in turn increase the hardness of the surface by obstacle the motion of the PMMA chains [18 and19]. But the hardness results at the weight fraction of Pistachio Shell powder above the (9% wt.) had been decreased, the reason behind this behavior, may be related to the lack in wettability and weakly physical bonds accompanied the increasing in the weight fraction. Also this behavior may be resulted from that the PMMA

chains will separated due to the surplus(excess) of Pistachio Shell powder which in turn leads to weakens the mechanical properties of the resulted composite.

Table 2 illustrated the results of hardness test statistically, as shown from this table the mean values for all specimens are higher than that of neat PMMA, except the specimens reinforced with 12% wt. for all average particle sizes. The greatest value was obtained at the specimen reinforced by 9%wt. of average particle size 212µm; all groups have been shown non-significant difference (NHS) ($p>0.05$) as compared with neat PMMA.

**Table 2: The Results of Hardness test statistically.**

| Groups | | N | Mean | Std. Deviation | Std. Error | Minimum | Maximum | P-value |
|---|---|---|---|---|---|---|---|---|
| **A53** | 0 | 3 | 84 | 3.605 | 2.081 | 80.00 | 87 | .874 |
| | 3% | 3 | 84 | 1.417 | .8185 | 82.60 | 85.3 | **(NS)** |
| | 6% | 3 | 84.8 | 2.685 | 1.550 | 81.70 | 86.4 | |
| | 9% | 3 | 86 | 2.662 | 1.537 | 83.50 | 88.8 | |
| | 12% | 3 | 84.5 | 1.473 | .8504 | 82.80 | 85.4 | |
| **A106** | 0 | 3 | 84 | 3.605 | 2.081 | 80.00 | 87 | .680 |
| | 3% | 3 | 84.35 | 1.353 | .781 | 82.80 | 85.3 | **(NS)** |
| | 6% | 3 | 85.2 | 1.442 | .832 | 83.60 | 86.4 | |
| | 9% | 3 | 86.5 | 1.907 | 1.101 | 84.50 | 88.3 | |
| | 12% | 3 | 84.8 | 1.852 | 1.069 | 82.70 | 86.2 | |
| **A150** | 0 | 3 | 84 | 3.605 | 2.081 | 80.00 | 87 | .741 |
| | 3% | 3 | 84.7 | 1.802 | 1.040 | 82.70 | 86.2 | **(NS)** |
| | 6% | 3 | 85.6 | 2.498 | 1.442 | 82.80 | 87.6 | |
| | 9% | 3 | 86.8 | 2.291 | 1.322 | 84.30 | 88.8 | |
| | 12% | 3 | 85.6 | 2.498 | 1.442 | 82.80 | 87.6 | |
| **A212** | 0 | 3 | 84 | 3.605 | 2.081 | 80.00 | 87 | .505 |
| | 3% | 3 | 85 | 2.424 | 1.400 | 82.80 | 87.6 | **(NS)** |
| | 6% | 3 | 86.3 | 1.732 | 1.000 | 84.30 | 87.3 | |
| | 9% | 3 | 87.2 | 1.417 | .8185 | 85.60 | 88.3 | |
| | 12% | 3 | 86.4 | 1.824 | 1.053 | 84.30 | 87.6 | |

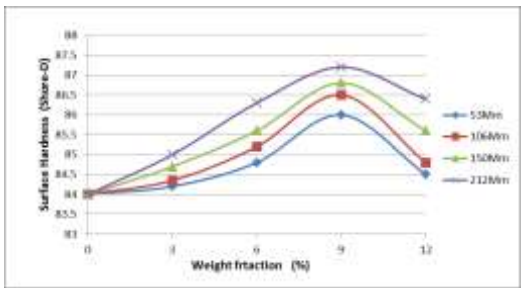


**Figure 3: Represents the Relationship between the Hardness and Weigh Fraction of Pistachio Shell powder**

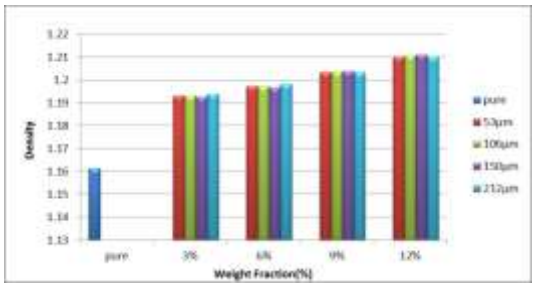


**Figure 4: Illustrate the Relationship between the Density and the Weight Fraction of Pistachio shell powder**.

*III. Density Test*

The relationship between the density of the specimens and the weight fraction of Pistachio Shell powder is illustrated in the Figure 4, it can be seen that the density values for the composite specimens are increased with increasing the weight fraction of Pistachio Shell powder. This behavior may be attributed to that. These particles minimize and filled the PMMA internal porosity, so we obtained higher density composite for the same volume [20], or it may be resulted from that, the density of reinforcing powder is higher than that of PMMA matrix material [21], [22].

Table 3 shows density results statistically, this table illustrated that the mean values for all specimens are higher than that of neat PMMA, and the specimen reinforced with 12% wt. of all average particle sizes showed the highest values. All the groups that is reinforced with pistachio shell particles have been showed non-significant difference ($p>0.05$) as compared with neat PMMA.

DOI: http://dx.doi.org/10.30684/etj.37.12A.5

**Table 3: Represent the Statistical Analysis of Density Test.**

| Groups | N | Mean | Std. Deviation | Std. Error | Minimum | Maximum | P-value |
|---|---|---|---|---|---|---|---|
| **A530** | 3 | 1.161 | .0397 | .0229 | 1.115 | 1.185 | |
| 3% | 3 | 1.193 | .0178 | .0103 | 1.172 | 1.204 | .384 **(NS)** |
| 6% | 3 | 1.193 | .0148 | .0085 | 1.184 | 1.210 | |
| 9% | 3 | 1.192 | .0198 | .0114 | 1.177 | 1.215 | |
| 12% | 3 | 1.194 | .0096 | .0055 | 1.183 | 1.202 | |
| **A1060** | 3 | 1.161 | .0397 | .0229 | 1.115 | 1.185 | |
| 3% | 3 | 1.197 | .0163 | .0094 | 1.178 | 1.207 | .344 **(NS)** |
| 6% | 3 | 1.197 | .0137 | .0079 | 1.186 | 1.212 | |
| 9% | 3 | 1.196 | .0306 | .0176 | 1.168 | 1.229 | |
| 12% | 3 | 1.198 | .0087 | .0050 | 1.188 | 1.205 | |
| **A1500** | 3 | 1.161 | .0397 | .0229 | 1.115 | 1.185 | |
| 3% | 3 | 1.203 | .0137 | .0079 | 1.188 | 1.215 | .153 **(NS)** |
| 6% | 3 | 1.203 | .0223 | .0129 | 1.185 | 1.228 | |
| 9% | 3 | 1.204 | .0102 | .0059 | 1.192 | 1.213 | |
| 12% | 3 | 1.203 | .0114 | .0066 | 1.195 | 1.216 | |
| **A2120** | 3 | 1.161 | .0397 | .0229 | 1.115 | 1.185 | |
| 3% | 3 | 1.210 | .0154 | .0089 | 1.196 | 1.226 | .076 **(NS)** |
| 6% | 3 | 1.210 | .0133 | .0076 | 1.201 | 1.226 | |
| 9% | 3 | 1.211 | .0144 | .0083 | 1.196 | 1.225 | |
| 12% | 3 | 1.210 | .0158 | .0091 | 1.196 | 1.227 | |

## 7. Conclusions

According to the experimental results of bio Composite materials which were prepared in this research, it can be concluded that:

1. The compression test and surface hardness results showed an increasing, with the increasing the weight fraction of Pistachio Shell filler particles until reach the weight percentage of 9% for all average particle sizes, and above this percent the compression strength and surface hardness results will be decreased.
2. With regard to density test the results showed an increasing, with the increasing the weight fraction of Pistachio Shell filler particles.
3. The statistical analysis of compression test results illustrated that there were very high significant difference (VHS) ($p \leq 0.001$) as compared with neat PMMA. The greatest value was obtained at the specimen reinforced by 9%wt. of average particle size 212μm.
4. The statistical analysis of surface hardness results had shown that: all groups have been shown non-significant difference (NHS) ($p > 0.05$) as compared with neat PMMA. The greatest value was obtained at the specimen reinforced by 9%wt. of average particle size 212μm;
5. The statistical analysis of density test results showed non-significant difference ($p > 0.05$) as compared with neat PMMA, and the specimen reinforced with 12% wt. of all average particle sizes showed the highest value.

## References

[1] K. Natarajan, and P.C. Balasubramanya, "Mechanical and Morphological Study of Coir Fiber Reinforced Modified Epoxy Matrix Composites," International Journal of Emerging Technology and Advanced Engineering, Vol. 3, pp.583-587, 2013.

[2] S.C. Venkateshappa, B. Bennehalli, M.G.Kenchappa, and R. P. G. Ranganagowda, "Flexural Behaviour of Areca Fibers Composites," Bio Resources, Vol. 5, No. 3, pp. 1846-1858, 2010.

[3] U.S. Bongarde and V.D. Shinde, "Review on Natural Fiber Reinforcement Polymer Composites," International Journal of Engineering Science and Innovative Technology, Vol.3, No.2, 2014.

[4] N.A. Hatim, A.A. Taqa, A. Abbas and M.A. Shuker., "The Effect of Thyme and Nigella Oil on Some Properties of Acrylic Resin Denture Base," Al-Rafidain Dent Journal, Vol. 10, No. 2, 2010.

[5] M. M. Mansour, W. C. Wagner, and T. M. G. Chu, "Effect of Mica Reinforcement on the Flexural Strength and Microhardness of Poly Methyl Methacrylate Denture resin," Journal of Prosthodontics: Implant, Esthetic and Reconstructive Dentistry, Vol. 22, No.3, pp. 179-183, 2013.

[6] J. K. Oleiwi, S. I. Salih, and H. S. Fadhil, "Study Compression and Impact Properties of PMMA Reinforced by Natural Fibers Used in Denture," Engineering and Technology Journal, Vol. 36, No.6, Part (A) Engineering, pp. 652-655, 2018.

[7] K. N. Okeke, A. Vahed, and S. Singh, "Improving the Strength Properties of Denture Base Acrylic Resins Using Hibiscus Sabdariffa Natural Fiber," Journal of International Dental and Medical Research, Vol. 11, No.1, pp. 248-254, 2018.

[8] M.K. Fahmi and M. I. Ebrahim, "Effect of Polypropylene Fiber Addition on the Flexural Strength, Fracture Toughness, and Hardness of Heat-Polymerized Acrylic Resin," EC Dental Science, Vol. 8.3, pp. 101-109, 2017.

[9] H. C. Obasi, N.C Iheaturu, F. N. Onuoha, C. O. Chike-Onyegbula, M. N. Akanbi and V.O. Ezeh, "Influence of Alkali Treatment and Fiber Content on the Properties of Oil Palm Press Fiber Reinforced Epoxy Biocomposites," American Journal of Engineering Research, Vol.3, No.2, pp. 117-123, 2014.

[10] Annual Book of ASTM Standard, "Standard Test Method for Compressive Properties of Rigid Plastics," D 695-02a, pp. 1-8, 2002.

[11] Annual Book of ASTM Standard, "Standard Test Method for Plastics Properties-Durometer Hardness," D 2240-03, PP. 1-12, 2003.

[12] E. Charles. Carraher and Jr. Seymour Carraher's, "Polymer Chemistry," Seventh Edition by Taylor & Francis Group, LLC, 2008.

[13] P. K. Mallick, "Fiber-Reinforced Composite: Materials, Manufacturing, and Design," International Standard Book, 3rd edition, 2007.

[14] S. Majid, M.K. Nabi, and R.Abbas, "An Experimental Investigation of $HA/AL_2O_3$ Nano Particles on Mechanical Properties of Restoration Materials," Engineering Solid Mechanics, Vol. 2, No. 3, pp. 173-182, 2014.

[15] B. Suryasarathi, and P. A. Mahanwar, , "Effect of Fly Ash on the Mechanical, Thermal, Dielectric, Rheological and Morphological Properties of Filled Nylon 6," Journal of Minerals & Materials Characterization & Engineering, Vol. 3, No. 2, pp. 65-72, 2004.

[16] S.I. Salih, J.K. Oleiwi and H. S. Fadhil, "Preparation and Investigation of some Properties of Acrylic Resin Reinforced with Siwak Fiber Used for Denture Base Applications," Kurdistan Journal of Applied Research, Vol. 2, No. 3: pp. 309-314, 2017.

[17] J. K. Oleiwi, and Q. A. Hamad, "Studying the Mechanical Properties of Denture Base Materials Fabricated from Polymer Composite Materials," Al-Khwarizmi Engineering Journal, Vol.14, No.3, pp. 100-111, 2018.

[18] S. C. George, T. Jose, M. S. sreekala, and M. G. Maya, "Mechanical Properties of Short Sisal Fiber Reinforced Pheno Formaldehyde Eco-Friendly Composites," Polymers from Renewable Resources, Vol. 8, No.1, PP. 27, 2017.

[19] Q. A. Hamad, "Fabrication and Characterization of Denture Base Material by Hybrid Composite PMMA Resin," PH.D. Thesis University of Technology, Baghdad, Iraq, 2015.

[20] H. J. Abdul Rahman, "The Effect of Some Natural Powders on the Properties of Acrylic Resin Denture Base Material," M.Sc. Thesis, University of Technology, Baghdad, Iraq, 2018.

[21] S. Hashemian, and J. Shayegan, "A Comparative Study of Cellulose Agricultural wastes (Almond Shell, Pistachio Shell, Walnut Shell, Tea Waste and Orange Peel) for Adsorption of Violet B dye from Aqueous Solutions," Oriental Journal of Chemistry, Vol. 30, No.4, pp. 2091-2098, 2014.