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Effect of Natural Fillers on the Tensile and Flexural Properties of PMMA used in Denture Manufacture

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Abstract. The mechanical properties of the dental are very important in order to make the proper selection of the right material to be used by the dentist. The effect of natural filler on the mechanical properties of PMMA used for denture manufacture has been investigated. Three natural materials, which are Siwak, Pomegranate, and Olive, are selected. The filler is prepared by grinding the material and sieving it to get the filler with a grain size of 50, 75, and 100 μm for each. The filler is added with three weight fractions in which become four parameters with three levels. Using the standard Taguchi's array L9 (4^3). S/N ratio and ANOVA are used to determine the optimum process parameters with their significant factor. The results show that the optimal parameters combination with their levels for higher modulus of elasticity appear by using grain size of 75 μm , with (0.6% Olive, 0.6% Pomegranate, and 0.9% Siwak) filler weight fraction. Whereas for the tensile strength and bending strength at (100 μm , with (0.6% Olive, 1.2% Pomegranate and 0.9% Siwak)) and 75 μm , with (0.9% Olive, 0.6% Pomegranate and 0.9% Siwak) respectively. The Siwak filler has the most significant influence on the mechanical properties with contribution ranged from (25-52) %, followed by pomegranate filler. The grain size affects tensile strength more than the other mechanical properties. The experimental and predicted results are very close, with errors not exceed 4%.

Keywords. Natural Fillers, Siwak, PMMA.

1. Introduction

Biomaterials can be defined as artificial material or natural origin. It was used as supplements, treatments, and to substitute any part of the body tissues where it does this function in tight contact with all living tissues. The artificial bones and teeth region's developments seem to solve the most challenging tissue problems [1]. The teeth' composition and structure are perfectly adapted to the mouth's functional demands and are far superior compared to any artificial material. Dental Materials are the fabricated materials that are designed to be used in dental material manufacturing. They affect the physical, chemical, and mechanical properties of the dental with their heat treatments. All these properties are very important to properly select the right material to be used by the dentist. Also, studying all the properties of these materials gives a full understanding and ability to explain the behavior of the materials by the dentist. The suitable selection to be made for the right material for the patient and knowing how to use it to achieve the best results. Prosthetic dentistry can be defined as the surrogate of the lost tooth, which may have been missed because of different reasons, either accident, fall, or any emergency case by either fixed or removable denture [2]. A denture is defined as the



removal and replacement of the surrounding tissues and missed tooth. It can be further categorized as two types of dentures: the complete and the partial denture bases.

Due to the denture's importance in our life, many researchers try to enhance the base material used in denture manufactured. Hanan [3] studied the mechanical properties of adding Siwak powder as the reinforcement material with an average particle size of (75 μ m) in three varying contents (3%, 5%, and 7%) weight fraction on the heat-cured acrylic resin PMMA. The results show a considerable reduction in compressive strength, tensile strength, and impact strength at a concentration of (7 %) from the powder of Siwak to the acrylic resin. Qahtan [4] improved PMMA acrylic resin properties by adding two types of particles (micro-zirconia and nano-hydroxyapatite) as reinforcing materials with varying volume fractions. The results show that adding the micro-zirconia and nano-hydroxyapatite will improve the physical and mechanical characteristics. Hwazen et al. [5] demonstrated the mechanical characterizations of polymethyl methacrylate (PMMA) resin as a denture base material strengthen with Siwak fibers as a natural material. The output falls on the improvement in hardness, tensile strength, Young modulus, fracture toughness with increasing in the length and content ratios of the fiber of Siwak. Jawad et al. [6] investigated the effect of adding natural material such as Siwak fibers and bamboo fibers on polymethyl methacrylate's mechanical properties (PMMA). The result shows that the compression strength increased by increasing the weight fraction of both fibers. Moreover, the compression strength of both fibers decreased by increasing the fiber length.

The fracture of an acrylic resin denture is the main problem that happens in prosthodontic serving, which is the primary failure of the prosthodontics denture base materials, yet it remains an unsolved issue till this moment. According to the survey, it was found that 63% to 68% of the complete denture prostheses were broken within a few years after fabrication by where the failure occurs. Therefore, this work's main objective is an experimental investigation to enhance the denture material's mechanical properties by adding natural filler using a robust design.

2. Experimental work

2.1. Material selection

The materials selected to prepare test specimens used in denture manufacturing are (PMMA) polymethyl methacrylate acrylic resin manufactured by Spofa Dental Company, with three reinforced natural fillers pomegranate peels powder, Olive kernel powder, and Siwak powder. The three types of reinforcement powders were prepared with a grain size of (50, 75, and 100) μ m.

2.2. Experiment design

Four parameters with three levels for each that:

The powder grain size of (50, 75, and 100) μ m, Pomegranate peels powder weight fraction of (0.6, 1.2, and 1.8) %, Olive kernel powder weight fraction of (0.3, 0.6, and 0.9) %, Siwak powder weight fraction of (0.3, 0.6, and 0.9) %.

These were considered to optimize the composite denture mechanical properties using the Taguchi technique. The selection of control factors is the most crucial stage in the experiment design. Therefore, choose a suitable orthogonal array and control parameters with their levels to get a combination of optimum levels [7, 8]. Table 1 shows these parameters with their levels. To study the impact of parameters in this work, L_9 (4^3) orthogonal design has been used, as shown in Table 2.

2.3. Specimens preparation

The specimens were manufactured based on the dry hand layup technique. The standard mixing ratio for PMMA acrylic resin is mixed with a volumetric ratio of 2:1 (PMMA: MMA) [9]. The powders filler was added with known amounts to resin until mixed well. Using metallic mold made from iron, low carbon steel type with dimensions of (200mm \times 200mm \times 10mm), with two separate plates, the lower plate of the die has a smooth surface while the die's upper plate has four cavities, as shown in Figure 1. The mold was designed according to the specimen's standard. The tensile test samples were manufactured according to the international standard (ASTM D638M-87b) with a length of 150 mm

and a thickness of 4 mm. The bending test specimens were manufactured according to ISO 178 international standard with a length of 120 mm, width 10 mm, and 4 mm thickness.

The mixture was then poured as a thin straight line in a distributed way inside the metallic mold's cavities. The plate's interface on the mold, which is in contact with the dough, was covered by a thin layer and must behave like a smooth surface to ensure no adhesion. After pouring the mixture into the die cavities, the die must be vibrated very gently, and then fixed was done from the metallic side to the other side to remove any residual air or gas bubbles. The mixture was poured into a mold (die) hole until the metallic mold holes were filled. The mold's upper surface was covered by a smooth thermal layer and pressed by using a metal plate with a size similar to the size of the mold obtained by the cavities within the samples with a smooth upper surface. After completing the cooling of specimens, the acrylic resin specimens were all removed from the metal mold and using a specialized manual grinder to remove any cracks or bubbles found in the edges of the surfaces of specimens. The specimens of different shapes with smooth surfaces were obtained, as shown in Figure 2. All the specimens are immersed in the distilled water for two days at temperature ($37 \pm 1^\circ\text{C}$), to ensure that the denture base stays in the semi oral cavity environment and release the residual stress and bring out the residual monomer.

Table 1. Parameters with their levels.

Parameters	Symbol	Levels of Parameter		
		1	2	3
Grain size (μm)	A	50	75	100
Olive Kernel filler	B	0.3%	0.6%	0.9%
Pomegranate filler	C	0.6%	1.2%	1.8%
Siwak filler	D	0.3%	0.6%	0.9%

Table 2. Experimental factors and levels using L9 orthogonal array.

Parameters and level									
No.	Grain size (μm)-A		Olive filler-B		Pomegranate filler-C		Siwak filler-D		PMMA (wt%)
	level	value	Level	Value	level	value	level	value	
1	1	50	1	0.3%	1	0.6%	1	0.3%	98.8
2	1	50	2	0.6%	2	1.2%	2	0.6%	97.6
3	1	50	3	0.9%	3	1.8%	3	0.9%	96.4
4	2	75	1	0.3%	2	1.2%	3	0.9%	97.6
5	2	75	2	0.6%	3	1.8%	1	0.3%	97.3
6	2	75	3	0.9%	1	0.6%	2	0.6%	97.9
7	3	100	1	0.3%	3	1.8%	2	0.6%	97.3
8	3	100	2	0.6%	1	0.6%	3	0.9%	97.9
9	3	100	3	0.9%	2	1.2%	1	0.3%	97.6

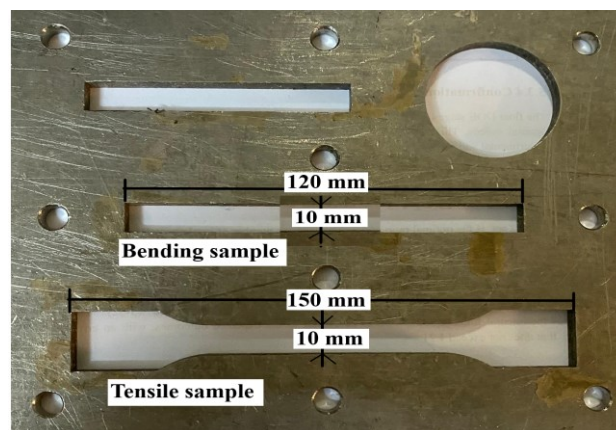


Figure 1. The metallic mold.

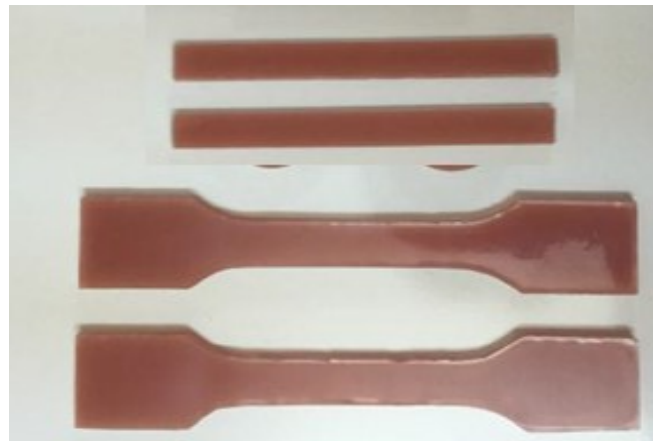


Figure 2. The specimens.

2.4. Experimental tests

The experimental tests were carried out at room temperature in the Material Engineering Department Laboratories/University of Technology- Iraq. The results represent the average data for three tested specimens. To achieve the research objective, the following tests were accomplished:

1. The tensile test was used to estimate a stress-strain relationship for the prepared specimens. The tensile test is performed according to the (ASTM D638-87b) by using the universal tensile instrument manufactured by (The Laryea Company in China country). Type (WDW-50) at a crosshead speed feed (5mm/min) and the applied load of (5000 N) until specimen break occurred.
2. The bending test method uses three-point. In this method, a vertical force was applied at the middle of the composite specimens to obtain the relation between the force in (Newton) and the displacement in (millimeter) for all the prepared specimens. The sample was prepared according to the standard (ISO 178) [10]. Figure 3 illustrates the experimental flexural test instrument.

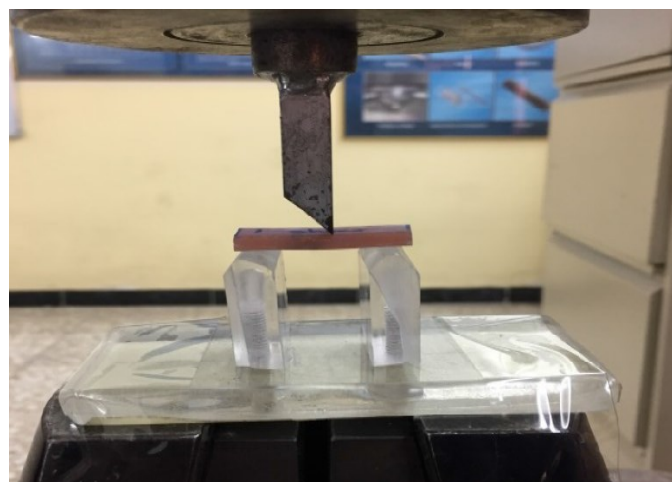


Figure 3. Inspections flexural test.

3. Results and discussion

The tensile and bending tests were done using the tensile test and the flexural instruments. The mechanical properties investigated here are the modulus of elasticity, tensile strength, and bending strength was estimated, and their results are listed in Table 3.

Table 3. Experimental results and their signal-to-noise ratio.

No.	Modulus of Elasticity		Tensile strength		Bending strength	
	GPa	S/N dB	MPa	S/N dB	MPa	S/N dB
1	1.063	0.531	28	28.943	92	39.28
2	0.722	-2.829	40	32.041	62	35.85
3	1.045	0.382	38	31.596	100	40.00
4	1.127	1.038	44	32.869	104	40.34
5	1	0.000	32	30.103	83	38.38
6	1.069	0.580	36	31.126	117	41.36
7	0.572	-4.852	45	33.064	88	38.89
8	1.656	4.381	49	33.804	112	40.98
9	0.9	-0.772	41	32.256	92	39.28

3.1. Signal to noise ratio (S/N)

Signal to noise ratio measures sensitivity of the quality investigated to those uncontrollable factors (error) in the experiment. Using “bigger is better” quality characteristic for mechanical properties investigation [11].

$$S/N = -10 \log_{10} \left[\frac{1}{n} \sum_{i=1}^n \frac{1}{y_i^2} \right] \quad (1)$$

Where:

n is the number of observations, and y_i is the data observed.

For all nine experiment readings, the S/N ratios were calculated and listed in Table 3. It can be seen that experiment number 8 has the largest modulus of elasticity and tensile strength S/N ratio. The parameters combined with their level in this experiment, are $A_3B_2C_1D_3$. However, experiment number 6 shows the most considerable value of bending strength with parameters combination, and their level is $A_2B_3C_1D_2$.

According to the results of combinations generated from the orthogonal array, the optimum levels were predicted by estimating the control factors for different three levels. The control factor levels were determined and listed in Table 4. Figure 4 shows the distributions of average S/N ratios. Where the goal for mechanical properties is “bigger is better.” From Figure 4, it can be noticed that the optimal parameters combined with their levels for higher modulus of elasticity are $A_2B_2C_1D_3$, equivalent to (75 μm grain size, 0.6% Olive filler, 0.6% Pomegranate filler, and 0.9% Siwak filler). While the optimal parameters combined with their levels for tensile strength and bending strength are $A_3B_2C_2D_3$ and $A_2B_3C_1D_3$, respectively.

3.2. Analysis of variance (ANOVA)

The analysis of variance method ANOVA objective is to analyze the influence of the design parameters on the total variance of results [12]. The parameter that is statistically more significant than others can be identified in ANOVA analysis. Table 5 shows the results of ANOVA analysis for the mechanical properties. For modulus of elasticity, it is noticed that the Siwak filler has the highest percentage contribution through the process by 52.29%, that it is considered the more significant parameter in comparison with the others. So, Siwak has the most significant influence on the modulus of elasticity, followed by the pomegranate filler with 35.47 % contribution. Also, for tensile strength, the Siwak filler has the greatest influence with 46.13%. While for bending strength, it is found that the Siwak filler shows an important contribution by a contribution of 24.37%, respectively. The ANOVA analysis shows the Siwak filler is the most influential parameter on the denture mechanical properties followed by pomegranate filler. While the grain size present in tensile strength.

Table 4. Average signal to noise ratio of different parameter levels.

Parameter	Symbol	Average of S/N (dB) of different level			Max-Min	Optimum level
		1	2	3		
Modulus of Elasticity						
Grain size (μm)	A	-0.639	0.539	-0.414	1.178	A ₂ B ₂ C ₁ D ₃
Olive filler	B	-1.09	0.52	0.06	1.61	
Pomegranate filler	C	1.83	-0.85	-1.49	3.32	
Siwak filler	D	-0.08	-2.37	1.93	4.30	
Tensile Strength						
Grain size (μm)	A	30.86	31.37	33.04	2.18	A ₃ B ₂ C ₂ D ₃
Olive filler	B	31.63	31.98	31.66	0.36	
Pomegranate filler	C	31.29	32.39	31.59	1.10	
Siwak filler	D	30.43	32.08	32.76	2.32	
Bending Strength						
Grain size (μm)	A	38.37	40.03	39.72	1.65	A ₂ B ₃ C ₁ D ₃
Olive filler	B	39.50	38.40	40.21	1.81	
Pomegranate filler	C	40.54	38.49	39.09	2.05	
Siwak filler	D	38.98	38.70	40.44	1.74	

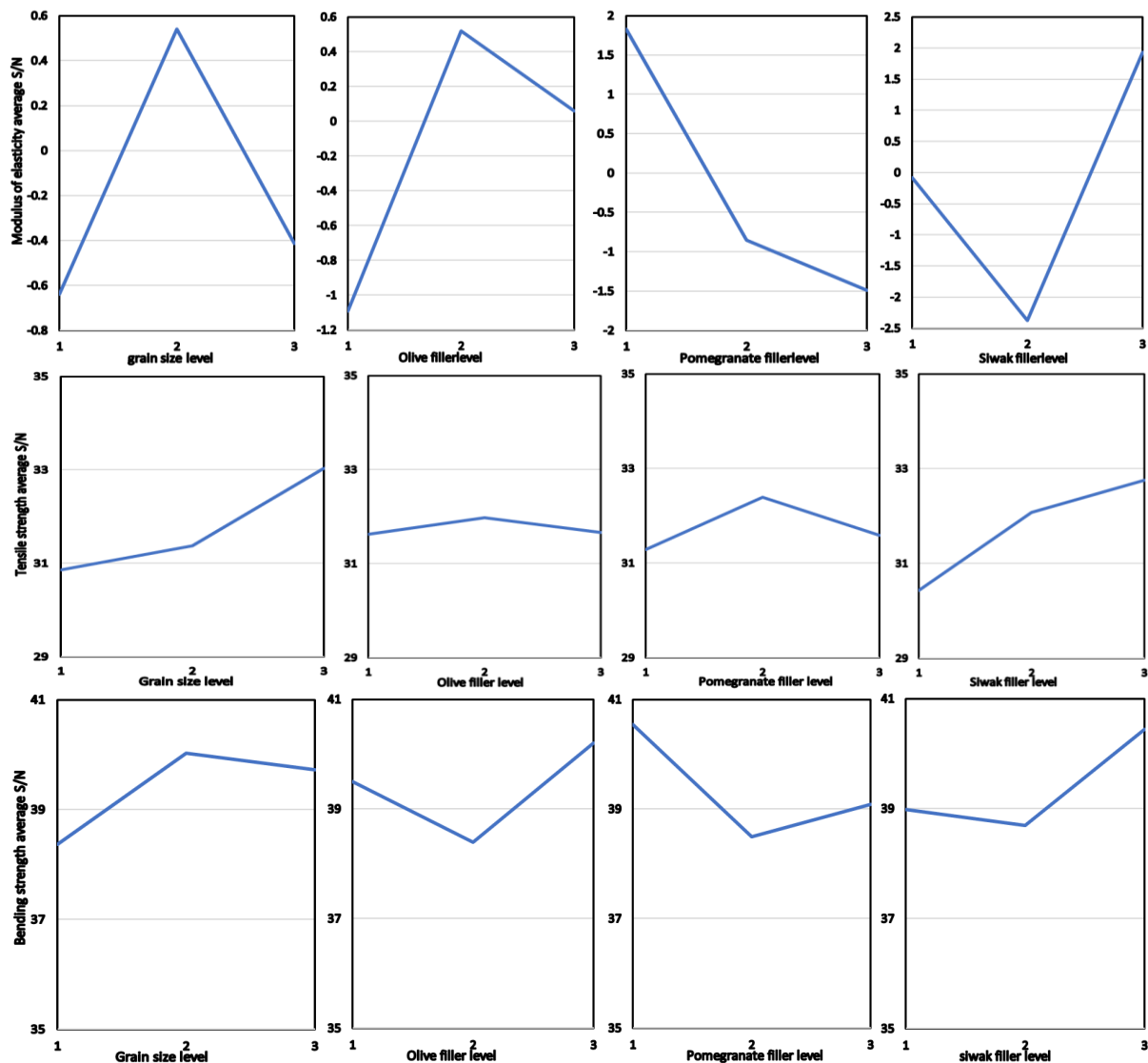
**Figure 4.** Average signal to noise ratio of Mechanical properties with different parameter levels.

Table 5. ANOVA Results of mechanical properties

Parameters	Sum of Squares (SS)	Degree of freedom (df)	Mean Squares (MS)	Contribution (%)
Modulus of Elasticity				
Grain size (μm)	2.421862	2	1.2109312	4.56
Olive filler	4.080943	2	2.0404717	7.68
Pomegranate filler	18.836976	2	9.4184882	35.47
Siwak filler	27.766961	2	13.883480	52.29
Error	0.000000	2	0.0000	0.00
Total	53.106744	2		100.00
Tensile Strength				
Grain size (μm)	7.820466	2.00	3.9102331	42.18
Olive filler	0.233437	2.00	0.1167187	1.26
Pomegranate filler	1.934300	2.00	0.9671498	10.43
Siwak filler	8.554220	2.00	4.2771100	46.13
Error	0.000000	2.00	0.0000	0.00
Total	18.542423	10.00		100.00
Bending Strength				
Grain size (μm)	4.634614	2	2.3173068	21.51
Olive filler	4.981019	2	2.4905095	23.11
Pomegranate filler	6.683420	2	3.3417100	31.01
Siwak filler	5.252175	2	2.6260877	24.37
Error	0.000000	2	0.0000	0.00
Total	21.551228	10		100.00

4. Confirmation Test

Taguchi method confirms the analysis results, and this is the final step in the method [13]. The optimum levels of the process parameters that have been obtained by analysis of the S/N ratio and experimental results of mechanical properties were used to develop the confirmation test. The values of optimum levels and the total mean value for each of S/N and experimental results shown in Table 4 have been used in Equation 2 [13, 14]; this model is called as predicted optimum mechanical properties.

$$\eta_{opt} = \eta_m + \sum_{i=1}^k (\eta_i - \eta_m) \quad (2)$$

Where:

η_{opt} : Predicted optimum value.

η_m : Total mean value.

η_i : Mean value at optimum levels from tables (4 and 5).

k : Number of main design parameters that affect the quality characteristics.

The optimal parameters combined with their levels for higher mechanical properties of modulus of elasticity, tensile strength, and bending strength are $A_2B_2C_1D_3$, $A_3B_2C_2D_3$, and $A_2B_3C_1D_3$, respectively. Table 6 summarizes the results of the confirmation test. The results show that the experimental and predicted ones are very close, with error not exceeding 4%. This verifies that the experiment result is correlated with the predicted result.

Table 6. Confirmation results.

Characteristic	Optimum Parameter			
	Level	Experiment	Predication	Difference (%)
Modulus of elasticity (GPa)	$A_2B_2C_1D_3$	1.52	1.455	4
Tensile strength (MPa)	$A_3B_2C_2D_3$	51.21	49.551	3.2
Bending strength (MPa)	$A_2B_3C_1D_3$	124.43	126.423	1.5

5. Conclusions

The following conclusions can be drawn:

1. The optimal parameters combined with their levels for higher modulus of elasticity appear by using grain size of 75 μm , with (0.6% Olive, 0.6% Pomegranate, and 0.9% Siwak) filler weight fraction. Whereas for, tensile strength and bending strength at (100 μm , with (0.6% Olive, 1.2% Pomegranate and 0.9% Siwak)) and (75 μm , with (0.9% Olive, 0.6% Pomegranate and 0.9% Siwak)) respectively.
2. The Siwak filler has the main influence on the mechanical properties with contribution ranged from (25-52) %, followed by pomegranate filler. Which have the largest contribution among the other parameters.
3. ANOVA analysis shows the grain size effect on tensile strength by 42%, which more than the other mechanical properties.
4. The experimental and predicted results are very close, with errors not exceed 4%.

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