Improved Wear Rate Resistance, Compression Strength and Hardness of Polymethylmethacrylate Resin with Orange Peel Powder for Artificial Denture Base

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ABSTRACT  
The material utilized for manufacturing artificial dentures should have high-grade mechanical properties in order to resist heavy forces inside the mouth. This study includes investigation of some of the mechanical properties (wear rate resistance, compression strength, and hardness) of the specimens prepared by (hand lay-up) method. The wear behavior experiments were performed on (a pin-on-disk tester) under various factors 5%, 8%, 11%, 14%, 17% weight fraction of orange peel, (10, 15, 20 N) load applied and (5, 10, 15 minutes) sliding time, and analysis these experimentally by using the Taguchi’s experimental design (L9) (MINITAB 16). Tests explicated that the specimens (polymethylmethacrylate - 17% orange peel) composites have the best wear rate resistance, compression strength and hardness shore D (0.040×10−5 cm3/N.mm, 142 MPa, 86 shore D) respectively than other specimens (polymethylmethacrylate - 5%, 8%, 11%, 14% wt. orange peel) and these specimens better than the specimens standard polymethylmethacrylate, which could be attributed to the homogeneous dispersion of orange peel particles in the polymethylmethacrylate resin matrix. The results (signal to noise ratio) showed the factors (17% weight fraction) orange peels, (20 N) load applied, and (5 min) sliding time gives the best wear rate resistance. The results of the analysis of variance showed the sliding time (C) is the essential factor effect on the wear rate resistance followed by (A) weight fraction of orange peels and (B) load applied were less affected on wear behavior rate.

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1. Introduction
The composite polymer is a new composite material consisting of the interconnection of two or more materials whose engineering performance and characteristics better the individual constituents [1]. The polymer can be divided into two classes: thermosetting and thermoplastic resin [2], polymethyl methacrylate is an example of the thermoplastic resin and generally used for the artificial denture. In day now life, people are handling wear difficulty in the denture base, where the artificial denture base is the product, which is enveloped by soft and hard tissues. The artificial denture base changes depending upon the patient’s case that is complete denture and fixed partial denture [3]. In general, aged humans each need of the complete denture with beautiful. Several attempts have been performed to enhance the polymethyl methacrylate characteristics to wear behavior and improve the material strength by creating the composites (filler or particles) with polymethyl methacrylate [4]. Wear behavior of teeth, especially on the occlusal facades, is taken into consideration physiological and real processes. The wear behavior in the artificial denture occurs due to multifactorial etiology, including the interplay of attrition, erosion, and abrasion [5]. Further, each tooth wear method includes chemical and physical, numerous factors such as approach force, and time of contact, effect on the wear behavior. Numerous researches have been conducted to enhance the features of polymethylmethacrylate, which has such a wide variety of applications. Liu et al. [6] evaluated the wear rate and hardness of specimens prepared from polymethylmethacrylate resin with 5%, 10%, 15%, and 20% wt. nanohydroxyapatite and mineral trioxide aggregate. Wear rate evaluated under factors (20, 40, 60, 80, 100 N) load applied, (2.5, 5.0, 1.5, 10.0, 12.5 mm/Sec) speed and (4000, 8000, 12000, 16000, 20000 cycles. Results analysis by using the Taguchi experimental design L16. The results indicated the hardness of the specimens reinforced with 20% wt. M.T.A. better than specimens reinforced with 20% wt. nano-HA, while the specimens reinforced with HA give the wear resistance than specimen reinforced with (M.T.A.). Bdaiwi [7] study the compression, impact, hardness, bending, and tensile tests of polymethylmethacrylate with 0.5% to 2.5% wt. of nano ZrO$_2$Y$_2$O$_3$. Results indicated the weight fraction of 1% ZrO$_2$Y$_2$O$_3$ give better mechanical properties than comparison with weight fraction (0.5%, 1.5%, 2%, and 2.5%). Mohammed et al. [8] have studied the effect of different weight fractions (2%, 4%, 6% & 8%) cinnamon filler reinforced with polymethylmethacrylate resin in an impact test, flexural and tensile test. It can be noted from the results the specimens reinforced with 8% wt. The cinnamon filler gives a better values of flexural strength and tensile strength, but the specimens reinforced with 6% wt cinnamon gives better value of impact strength than other weight fractions.

2. Objective of the Research
The idea of the study: It was suggested to add a filler material (Orange peels) into polymethylmethacrylate in order to improve some mechanical properties (wear behavior, compression, and hardness shore D) due to weakness the mechanical properties of polymethylmethacrylate polymer resin when utilized in the artificial denture base without any addition.

3. Scientific Part
I. Materials and methods
Orange peels were obtained from local agricultural fields utilized as filler materials. Polymethylmethacrylate resin (Castlevania) equipped by (Vertes Dental) utilized as a matrix to prepare specimens composites.

II. Preparation of orange peels powder
Orange peels are agricultural waste and available in many places like (houses and restaurants). These agricultural wastes are natural, renewable and environmentally friendly sources that can be utilized in several medical and industrial fields since recycling of agricultural waste became is immediately necessary [9]. The orange peels washed in water to remove any dirt, sand, and other impurities, then dried below the sunlight for two days and grinding for 3 hours by using a mill to obtain a fine powder.
III. Manufacture of polymethylmethacrylate specimens

The Polymethylmethacrylate polymer consists of “monomer liquid” and “polymer powder” used in artificial denture base, the mixture ratio is commonly almost (17 gm) powder and (9.5 gm) liquid polymer. Prepares composite specimens by using hand lay-up casting method, the mold with dimensional (220 mm× 220 mm× 4 mm) was covered with a layer of nylon for easy removal of the specimens composites from the mold. At the beginning of the casting process are mixed the amount of orange peels powder (5%, 8%, 11%, 14%, 17%) with “monomer liquid” in a clean container and stirred well for (4-6 minutes), then add the polymer powder gradually and stirred for (7 minutes) to obtain an identical mixture. Finally, the mixture was poured into the mold and left at room temperature for (30 minutes) until the mixture was hardened. The specimens removed from the mold and put into an oven for 30 minutes at 55 Cº for curing in order to eliminate the residual stresses and improve the bonding among the base and reinforced material [10]. In addition, cutting specimens to wear test, compression test, hardness test according to ASTM G99-04, ASTM D 695, ASTM 2240 standards. Tables 1 offer the composition of the composite specimens.

IV. Mechanical tests

1. Wear Test

The “pin-on-disc” was utilized to estimate wear rate resistance of the artificial denture base composites. The dimensions of the specimens in this test were (10 mm diameter and 20 mm length) depending on the ASTM (G99-04) standards [11]. Each the tests wear was performed under factors includes applied loads (10N, 15N, 20N), sliding times (5, 10, 15 minutes), constant sliding velocity (2 m/s), and constant sliding distance (65 mm). The loss weighing of specimens was registered in all the wear tests, in order to calculate the wear rate by employing the following equation (1) [12].

\[
W.R = \frac{\Delta m}{L \cdot \rho \cdot F}
\]

Where: W.R: Wear rate (cm³/N.mm); Δm: The difference weight (gm); L: distance of sliding (mm); \(\rho\): density of specimen (gm/cm³); F: load applied (N)

2. Compression Test

The specimens for compression tests were cut depending on standard ASTM D695 [13] by utilizing the same tensile machine at strain rate (speed of cross head) was (5mm/min), manufactured by (Laryee Company in China), type (WDW-50).

3. Hardness Shore (D)

The specimens for hardness shore D were cut into the thickness (5 mm) and diameter (40mm) depending on standard ASTM D2240 [14] by utilize durometer hardness test, type (Shore D) at load applied equally to 50 N and depressing time of measuring equal to (15 sec).

4. Taguchi Experimental Design

The Taguchi approach is a robust methodology for responding with different variables where Taguchi design is employed to devise for the test layout and investigates the effect of each variable and determine the optimal level for any specific variable. In addition, this approach decreases total test costs and test time [15]. In this research, the Taguchi L9 orthogonal array was adopted and converted into S / N (signal to noise) data were used to measure the characteristics and to identify the important parameters through analysis of variance (ANOVA) [16]. There are three kinds of “signal-to-noise” analysis appropriate: 1) less is better, 2) nominal is better, and 3) bigger is better. In this research, the principal purpose is to decrease the wear rate; therefore, it was applied that the S/N lower is better, according to equalization 2 [17]. The optimal scale for a variable is the level that results in the highest “S/N” ratio value in the empirical space. Table 2 displays the levels of the factors utilized in the test of the wear behavior.

4. Results and Discussion

I. Particle size and x-ray fluorescence of the orange peels powder
Figure 1 displayed the median particle size for orange peels powder, which was approximately (27µm) and measured using (MASTERSIZER 2000) technique. Table 3 shows the content of the elements in orange peel powder after milling using the X-ray Fluorescence technique by using reagents Compton secondary molybdenum and Barkla scatters HOPG.

Table 1: Composition of the specimens composite

| Specimens | Composition      |
|-----------|-----------------|
| S1        | 100% PMMA       |
| S2        | 95% PMMA + 5% O.P |
| S3        | 92% PMMA + 8% O.P |
| S4        | 89% PMMA + 11% O.P |
| S5        | 86% PMMA + 14% O.P |
| S6        | 83% PMMA + 17% O.P |

Table 2: Levels of variables utilized in the test of wear behavior

| Factors | Level | Unit |
|---------|-------|------|
| (A) Weight fraction of orange peels powder | I (S1) | II (S2) | III (S3) | Unit (S4, S5, S6) |
| (B) Load applied | 10 | 15 | 20 | N |
| (C) Sliding time | 5 | 10 | 15 | Min |

Figure 1: Indicates the median particle size for orange peels powder after milling

Table 3: Content of the elements in orange peel powder

| Elements | Ratio % |
|----------|---------|
| K        | 1.6     |
| Ca       | 1.3     |
| Na       | 0.27    |
| Fe       | 0.20    |
| K₂O      | 1.90    |
| CaO      | 1.9     |
| Na₂O     | 0.40    |
| Fe₂O₃    | 0.39    |

II. Wear test

Table 4 displays the wear rate for each (Taguchi design L9 tests) along with value S/N ratios for specimen polymer composite reinforced by the different weight fractions (orange peels powder). The results were analyzed employ (MINITAB-16) which exhibit the wear rate dwindling with an added weight fraction of (orange peels powder), where the minimum wear rate was found in experiments test (15 and 17) under effect factors of (14%, 17% weight fraction orange peels powder - PMMA), (20 N, 15N load applied) and (5 min sliding time). Due to the homogeneous dispersion of fillers with the polymeric matrix, leading to a greater number of covalent bonds that improve the mechanical
properties [18]. Figure 2 displays the (major influence plot) control factors for weight fraction, the load applied, and the sliding time of all specimens. The figure shows that if S/N ratio is steady for the full range, suggest that these factors do not have much significance, while if S/N proportion is not steady for the full range, suggest that these agents have much significance as far as wear behavior rate is concerned. The results showed that the combination of the factors (17% weight fraction orange peels powder) (20 N load applied) and (5 min time sliding) contributes to the least wear rate. In addition, among all factors, it is possible to observe the weight fraction of orange peels powder and time sliding are more sentient with the (S/N ratio) for wear rate more than applied load. Figures 3 shows interaction plot for wear rate between the weight fraction of orange peels powder, applied load, and time sliding for all specimens. All factors are highly interactive because these composites tend to become more brittle with the addition of the orange peels powder from 8% to 17% weight fraction.

Table 4: Exhibit results of wear rate and S/N values of all specimens (PMMA - orange peels)

| Exp. | Weight fraction of Filler (A) | Load applied (B) | Time sliding (C) | Wear Rate cm³/N.mm | S/N |
|------|-----------------------------|------------------|------------------|-------------------|-----|
| 1    | 0%                          | 10               | 5                | 0.85×10⁻⁵         | 2.1672 |
| 2    | 0%                          | 15               | 10               | 1.02×10⁻⁵         | 1.4116 |
| 3    | 0%                          | 20               | 15               | 1.44×10⁻⁵         | 0.1720 |
| 4    | 5%                          | 10               | 10               | 0.64×10⁻⁵         | 3.8764 |
| 5    | 5%                          | 15               | 15               | 0.74×10⁻⁵         | 2.6154 |
| 6    | 5%                          | 20               | 5                | 0.41×10⁻⁵         | 7.7443 |
| 7    | 8%                          | 10               | 15               | 0.58×10⁻⁵         | 4.7314 |
| 8    | 8%                          | 15               | 5                | 0.30×10⁻⁵         | 10.4576 |
| 9    | 8%                          | 20               | 10               | 0.35×10⁻⁵         | 9.1186 |
| 10   | 11%                         | 10               | 5                | 0.14×10⁻⁵         | 17.0774 |
| 11   | 11%                         | 15               | 10               | 0.16×10⁻⁵         | 15.9176 |
| 12   | 11%                         | 20               | 15               | 0.18×10⁻⁵         | 14.8945 |
| 13   | 14%                         | 10               | 10               | 0.13×10⁻⁵         | 17.7211 |
| 14   | 14%                         | 15               | 15               | 0.18×10⁻⁵         | 14.8945 |
| 15   | 14%                         | 20               | 5                | 0.088×10⁻⁵        | 21.1103 |
| 16   | 17%                         | 10               | 15               | 0.045×10⁻⁵        | 26.9357 |
| 17   | 17%                         | 15               | 5                | 0.040×10⁻⁵        | 27.9588 |
| 18   | 17%                         | 20               | 10               | 0.041×10⁻⁵        | 27.7443 |
Figures 2: Major influence plot of control factor for weight fraction, load applied and sliding time for all specimens
The wear rates for all specimens PMMA has been offered in Figure 4. It is apparent from this figure the specimens (PMMA- 14%, PMMA- 17% orange peels) gives less wear rate (0.088 ×10^{-5}, 0.040×10^{-5} cm³/N.mm) respectively, the wear resistance increased significantly for two reasons: (i) the filler (orange peels) is uniformly dispersed with PMMA polymer resin, (ii) improves the strength of the friction surface through formation a dense and protective friction layer, thereby these reasons reducing the wear rate [19 and 20]. In specimens reinforced with lower concentrations of orange peels give less wear resistance because these concentrations can be easily separated from the PMMA during the wear behavior process, and thus reduce bonding between polymer matrix and filler, increasing the wear rate.
Figure 5 shows the effect load applied to the wear rate for all specimens. It was observed wear rate increase from \(0.85 \times 10^{-5}\) to \(1.44 \times 10^{-5}\) cm\(^3\)/N.mm with increase load applied from (10 to 20 N) for standard specimens (PMMA) because increasing the applied load leads to increased friction on the surface of the polymeric and thus leads to greater weight loss [21]. Also from the graph, the specimens (PMMA- 5% to 17% orange peels) have wear resistance at loads applied (10, 15, 20 N) better than specimens unfilled with orange peels, due to when applied the load on these supported specimens the load is carried to the filler by a shearing mechanism among the filler and polymer, for the bond was great the shearing mechanism produces the stress concentration across the filler/matrix interface [22]. For all specimens. It was observed wear rate increase from \(0.85 \times 10^{-5}\) to \(1.44 \times 10^{-5}\) cm\(^3\)/N.mm) with increase load applied from (10 to 20 N) for standard specimens (PMMA) because increasing the applied load leads to increased friction on the surface of the polymeric and thus leads to greater weight loss [21]. Also from the graph, the specimens (PMMA- 5% to 17% orange peels) have wear resistance at loads applied (10, 15, 20 N) better than specimens unfilled with orange peels, due to when applied the load on these supported specimens the load is carried to the filler by a shearing mechanism among the filler and polymer, for the bond was great the shearing mechanism produces the stress concentration across the filler/matrix interface [22]. Figure 6 presents the effect of sliding time on the wear test. From the Taguchi test shown, the wear rate increased with an increase sliding time from (5 to 15 min) due to with increase sliding time leads weakness in the typical polymer composite material and thermal softening, thus causes increased wear rate, these results agree with [23].

![Figure 5: Effect load applied on the mean wear rate for all specimens](image1)

![Figure 6: Effect sliding time on the mean wear rate for all specimens](image2)

### III. Analysis ANOVA for results wear rate

In order to find out the statistical importance of different factors weight fraction orange peels (A), the load applied (B), and sliding (C) on wear rate, analysis of variance (ANOVA) is performed on empirical data. Table 4 shown ANOVA analysis of variance wear rate for all specimens. The latest column of every table indicates (P-value) for the individual control factors and the possible interactions, when it is the (P-value) of the factor less than 0.05 this indicates that it has a clear effect on the wear rate, while when the (P-value) of the factor more than 0.05 this indicates that it has a less effect on the wear rate [24]. The
ANOVA results for specimens (pure PMMA, PMMA-5% O.P, PMMA-8% O.P) shown in Table 5 (A), indicate that, sliding time (P=0.033), weight fraction of orange peels (P=0.046) and load applied (P=0.060), in this order, sliding time followed the weight fraction of orange peels are the significant control factors on wear rate behavior. The ANOVA results for specimens (PMMA-11% O.P, PMMA-14% O.P, PMMA-17% O.P) shown in Table 4 (B), indicate that, sliding time (P=0.022) followed weight fraction of orange peels (P=0.037) followed load applied (P=0.046), in this order, for the weight fraction of orange peels, load applied and sliding. Time are significant control factors on wear rate behavior.

IV. Compression test

Figure 7 displays the results of the compression test of the (PMMA- as a function of orange peels). According to the results can be observed the compressive strength increase with an added weight fraction of the orange peels, therefore the specimen (PMMA containing 17% weight fraction orange peels) had the maximum value of ultimate compressive strength among all other weight, but these weight fraction (5%, 8%, 11%, 14% wt. Orange peels) have compressive strength values better than pure specimen PMMA. Because the additives have compression resistance better than the polymer base material, also the additives are easy to penetrate between the polymer chains leads to a reduced vacancy within the matrix material and thus raise the compressive strength [25 and 26].

IV. Hardness shore D

Figure 8 presents the influence of different weight fraction orange peels on the hardness of filling PMMA. This Figure indicates the hardness shore D of specimens PMMA filled with orange peels increases with increasing filler content because when added the fillers, the composite becomes stiffer and harder [27]. In addition, these additions of fillers improve the resistance to plastic deformation and lower in the matrix surface resistance to the indentation [28]. Therefore the hardness shore D values arise from (79) for specimens (PMMA without any filler) to (86) for specimens (PMMA-17% wt. orange peels).

| Source                  | DF | Seq SS | Adj SS | Adj MS  | F      | P      |
|-------------------------|----|--------|--------|---------|--------|--------|
| A) Specimens (100% PMMA, PMMA-5% O.P, PMMA-8% O.P) |    |        |        |         |        |        |
| Orange Peels (A)        | 2  | 0.77227| 0.77227| 0.38613 | 20.72  | 0.046  |
| Load Applied (B)        | 2  | 0.00407| 0.00407| 0.00203 | 0.11   | 0.060  |
| Sliding Time (C)        | 2  | 0.24500| 0.24500| 0.12250 | 6.57   | 0.033  |
| Error                   | 2  | 0.03727| 0.03727| 0.01863 |        |        |
| Total                   | 8  | 1.05860|        |         |        |        |

| Source                  | DF | Seq SS | Adj SS | Adj MS  | F      | P      |
|-------------------------|----|--------|--------|---------|--------|--------|
| B) Specimens (PMMA-11% O.P, PMMA-14% O.P, PMMA-17% O.P) |    |        |        |         |        |        |
| Orange Peels (A)        | 2  | 0.0228916| 0.0228916| 0.0114458 | 25.77  | 0.037  |
| Load Applied (B)        | 2  | 0.0010336| 0.0010336| 0.0005168 | 1.16   | 0.046  |
| Sliding Time (C)        | 2  | 0.0031349| 0.0031349| 0.0015674 | 3.53   | 0.022  |
| Error                   | 2  | 0.0008882| 0.0008882| 0.0004441 |        |        |
| Total                   | 8  | 0.0279482|        |         |        |        |

Figure 7: Compressive strength of all specimens
5. Conclusions

1. Improved the wear rate resistance significantly with an additional weight fraction of orange peels to PMMA compared with the wear rate resistance for the standard specimen (PMMA).
2. Weight fraction (17%) of filler orange peels, (20 N) load applied, and (5 min) sliding time give the best wear resistance.
3. Sliding time (C) was observed to be the great significant factor effect on the wear rate behavior followed by (A) weight fraction of orange peels and (B) load applied.
4. Specimens (PMMA- 17% orange peels) give the best value of compressive strength and hardness shore D (142 MPa, 86), respectively, compared with the standard specimen (PMMA) (94 pmma, 79).

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