Studying the impact strength of layered denture base resin
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Abstract
This research investigate the effect of uncoated and coated chopped carbon fibers with alumina Al$_2$O$_3$ or Tri calcium phosphate (TCP) on the impact value of acrylic poly methyl methacrylate (PMMA) denture base resin. To improve bonding between carbon fibers and coating materials powders, the surface of carbon fibers has been treated with Para amino benzoic acid (C$_7$H$_7$NO$_2$) and poly vinyl alcohol (PVA) was also used. The morphology of the coating layers has been examined by field emission scanning electron microscope (FE-SEM). According to the tests results, PMMA reinforced with uncoated chopped carbon fiber presents high impact strength value with bad aesthetic. Samples prepared by coated carbon fiber with Al$_2$O$_3$ or TCP have high impact strength values when compared to control group with good aesthetic. The measured value of impact strength shows a noticeable increase accordingly with the increase of PVA and decrease of used fiber.

Key words
Poly (methylmethacrylate) (PMMA), chopped carbon fiber (C.F.), Alumina (Al$_2$O$_3$), Tri calcium phosphate (TCP) powder.

Introduction
Poly methyl methacrylate (PMMA) is the most well known denture base material, due to its different advantages, including low cost, stability in the oral environment, biocompatibility, ease of processing and acceptable aesthetics. It is not considered as a perfect material because of its bad mechanical and physical properties [1-3]. The main drawbacks of poly methyl methacrylate are deficient ductility, viscoelastic behavior and inferior mechanical strength [4-6]. Rapid and quick development concepts of composite technology promoted the reinforcement of materials utilizing fibers [7-9]. Addition of polyethylene, glass and carbon fibers produced a
significant increase in the impact strength. Otherwise the addition of silk fibers did not produce an improvement in impact strength [10]. Carbon fiber was utilized in denture base reinforcement due to many reasons such as low density, excellent tensile properties, high thermal and chemical stabilities in the absence of oxidizing operators. Also, offer both the highest specific strength and highest specific modulus comparing to all reinforcing fibers. Carbon fibers have been utilized in composites as continuous fibers, woven and chopped fibers [11]. Carbon fibers weren’t used alone because of poor aesthetics due to color of the fibers [12-14], problems with polishing [12, 14, 15], and potential toxicity [12, 16, 17]. Denture base material reinforced with carbon fibers in (0.5, 1, and 2) wt% showed an increase in impact resistance value than that of control material. The highest value was recorded to the reinforcement at 2 wt% fibers content. The researcher used coated carbon fiber with CaP by using sol gel method, and reinforced PMMA with (0.5, 1, 2) wt%. On the other hand the value of transverse strength decreased [18].

In this study carbon fiber was used to enhance the impact strength value of PMMA, but still samples have poor aesthetic. The aesthetic was improved after incorporation of biomaterials coated fibers. Al₂O₃ or TCP were utilized to coat the dark carbon fibers with white color.

**Experimental part**

**A-Coating of carbon fibers**

Chopped carbon fibers (300 GSM CFRP FABRIC, weight: 300 gm/sqm, thickness: 0.167 mm, density: 1.8 g/cm³, England) was immersed in ethanol solution for 15 minutes for riddance from impurities and oxidation, after that dried at 50°C for 60 minutes. (3 gm) of benzoic acid (4-Amino benzoic acid, PABA, pure; minimum Assay: 99.0 %, Mumbai-400088, India) was added to (150 ml) of distilled water. The mixture was added to the fibers that were pre-dried in the oven, and then left for 4 h. The fibers were washed with distilled water several times and dried in oven for 4 h. at 50°C. Fig.1 shows chopped carbon fibers before coating.

![Fig. 1: Chopped carbon fibers.](image)

Coating of carbon black with aluminium oxide or Tri calcium phosphate:
PVA was dissolved in 20ml of distilled water with heating and Al₂O₃ or TCP powder was added to the solution according to Table 1. Chopped fibers was immersed in the coating solution immediately and left over night. The resulted fibers were shown in Fig.2.
Table 1: Groups number according to the reinforcement material.

| Groups no. | Black chopped C.F. (Weight fraction $w_i$) | PVA ($w_i$) | Powder ($w_i$) | PMMA |
|------------|------------------------------------------|-------------|----------------|------|
|            |                                          |             | $\text{Al}_2\text{O}_3$ | TCP |
| G1         | 0                                        | 0           | 0              | 0    | 1   |
| G2         | 0.03                                     | 0           | 0              | 0    | 0.97|
| G3         | 0.03                                     | 0.02        | 0.06           | 0    | 0.89|
| G4         | 0.03                                     | 0.02        | 0              | 0.06 | 0.89|
| G5         | 0.005                                    | 0.09        | 0.06           | 0    | 0.845|
| G6         | 0.005                                    | 0.09        | 0              | 0.06 | 0.845|
| G7         | 0.0075                                   | 0.09        | 0.06           | 0    | 0.8425|
| G8         | 0.0075                                   | 0.09        | 0              | 0.06 | 0.8425|

Fig. 2: Coated chopped C.F. with a) $\text{Al}_2\text{O}_3$, b) TCP.

B-Preparation of the flask

Denture base resin was set up by flask, the flask has 2 parts. One half of the flask filled with dental stone, while the other half contains kaolin with (70*50*3) mm dimension, the stone poured to fill the flask. The internal surface of each flask half were covered with vaseline to keep the dental stone from connecting to the cast. Finally, the flask left to dry over night, then the two parts opened and cleaned from kaolin.

C-Preparation of the samples

Eight groups of PMMA samples were manufactured as appeared in Table 1. The samples were set up by heat polymerised acrylic resin; the powder to liquid ratio was (1.8g/0.72ml), mixed and left for 15 min. until reaching at the dough. In G1 (pure) the dough was put in the mold directly. Chopped carbon fibers with or without coating was wetted with MMA monomer. In group (2 to 8) the dough was cut into two equal parts, carbon layered placed between them. The flask compressed by hydraulic press at 0.5 Ton for five minutes. Then the flask were put in water bath, heated to 100°C and left at boiling temperature for 60 min. and then kept to room temperature. The samples removed from the flask, cut to specific measurement (3*10*65) mm for impact test and smoothed with 400, 600, 800 grit SiC papers. All specimens were shown in Fig.3.
Fig.3: manufactured samples
1) G1 Pure   2) G2 uncoated chopped C.F.    3) G3 coated with Al2O3    4) G4 coated with TCP 
5) G5 coated with Al2O3  6) G6 coated with TCP   7) G7 coated with Al2O3    8) G8 coated with 
TCP.

D- Impact strength test
Impact strength test Charpy type was used by impact testing tool (ISO-179 impact tester N.43-1). A pendulum
of two Joule was utilized. The sample was put horizontally at its ends and struck by a free swinging pendulum
from a fixed height in the center. charpy impact strength was measured in kJ/m², by applying the following
formula:

\[
I.S. = \frac{(E)}{(b \times d)^2}
\]

where, \(I.S\) = impact strength in kJ/m².
E: Energy of fracture.
b: width of specimen.
d: thickness of specimen.

Results and discussion
Impact strength
Impact failures generally happen when abrupt hit to the denture because of accidental dropping. Within the oral
cavity PMMA is a brittle material, has low impact strength. To improve the impact strength of PMMA, the carbon
fiber was applied.

A) Effect of uncoated and coated chopped C.F. on impact strength
Table 2 appears the experimental results of impact strength for control sample; samples strengthened with
uncoated and coated chopped C.F. with Al2O3 or TCP respectively.

Table 2: Descriptive information of impact strength values of pure and coated and uncoated chopped fibers test.

| Groups no. | Impact Strength (kJ/m²) |
|------------|------------------------|
| G1         | 7.46                   |
| G2         | 9.28                   |
| G3         | 8                      |
| G4         | 11.25                  |
From Table 2, each reinforced group show an improvement in impact strength when compared to control group as shown in Fig.4. This attributed to carbon fibers which have the highest specific modulus and highest specific strength of all reinforcing fibers [11]. The applied stress transfer from the weakly polymer matrix to the fibers that have a high tensile strength [19]. Fibers reduce the crack propagation and change direction of cracks [20]. In spite of the increase in impact strength of the composite, the aesthetic is bad. To improve aesthetic, coating of carbon fibers with Al₂O₃ or TCP was tried. Al₂O₃ and TCP powders are biocompatible materials, were utilized to coat the dark carbon fibers with white color. Samples manufactured coating chopped C.F. with Al₂O₃ or TCP, had high impact strength value when compared to control group. In G4 the impact strength increased to (11.25) kJ/m² after carbon fiber coating with TCP. This may be due to TCP particles are hard and have much higher fracture strength [21]. And because of uniform distribution of TCP particles on fiber. Impact strength value for samples manufactured by chopped C.F. coated with TCP was higher than Al₂O₃; this is due to random distribution of Al₂O₃ [22].

![Fig.4: Impact strength for PMMA reinforced with coated and uncoated chopped C.F.](image)

**B) Effect of fiber loading on impact strength**

Table 3 appears impact strength values for samples manufactured by chopped C.F. have different fiber content and have the same weight fraction (wᵢ) of coated materials (6 wᵢ of Al₂O₃ or TCP) and PVA (9 wᵢ).

| Groups no. | I.S.(kJ/m²) of samples prepared by chopped C.F. coated with Al₂O₃ | Groups no. | I.S.(kJ/m²) of samples prepared by chopped C.F. coated with TCP |
|------------|---------------------------------------------------------------|------------|---------------------------------------------------------------|
| G5         | 8.67                                                          | G6         | 9.45                                                          |
| G7         | 10.17                                                         | G8         | 12.26                                                         |

*Table 3: Result of impact strength for samples with different fibers content*
From Fig. 5 impact strength values for all samples significantly increased with increasing of fiber content (0.5, 0.75) wt. The weight fraction of PVA is sufficient to wet the low content of chopped C.F. and make them bonded together, leading to good interface bonding among fibers, powders and the resin. In addition, low filler contents leads to good dispersion; this dispersion restrict crack propagation paths as well as enhance absorption of the load energy [23].

Field Emission Scanning Electron Microscope (FE-SEM)

Field emission scanning electron microscopic analysis for coating of carbon fibers surface was imaged. The images of Al₂O₃ coated fiber displayed irregular distribution of Al₂O₃ particle on the fiber surface, with irregular shape, agglomerations of Al₂O₃ particle on some fibers, while other fibers were not coated, as shown in Fig. 6. TCP coated fibers displayed good deposition distribution on the surface of carbon fiber, from the images spherical particles was also seen without any agglomerations on fibers surfaces as appeared in Fig. 7.
Conclusion
1- The color of dark carbon fibers was covered effectively.
2- It is probably to synthesis a denture base resin with great aesthetic and high impact strength from coated carbon fibers by Al₂O₃ or TCP powders.
3- Weight fraction of coated carbon fiber has positive effect on impact strength.
4- Samples prepared by incorporation of TCP to carbon fiber possess the highest impact strength value (12.26 kJ/m²).

References
[1] R. Alla, K. Raghavendra, R. Vyas, & Konakanchi, Int J Appl Dent Sci, 1, 4 (2015) 82-89.
[2] M. Gad, S. M. Fouda, F. A. Al-Harbi, R. Nääpänkangas, Raustia, Int J Nanomedicine, 12 (2017) 3801-3812.
[3] M. M. Gad & R. Abualsaud, Int. J. Biomater, 2019 (2019) 1-14.
[4] A. Alrahlah, H. Fouad, M. Hashem, A. Niazy, AlBadah. Materials, 11, 7 (2018) 1-15.
[5] T. R. Meng and M. A. Latta., J Contem- porary Dental Practice, 6, 4 (2005) 93-100.
[6] N. Murakami, N. Wakabayashi, R. Matsushima, A. Kishida, Y. Igarashi, J. the Mechanical Behavior of Biomedical Materials, 20 (2013) 98-104.
[7] G. Uzun, N. Hersek, & T. Tincer, J. prosthetic dentistry, 81, 5 (1999) 616-620.
[8] S. K. Garoushi, L. V. Lassila, P. K. Vallittu, J. Contemporary Dental Practice, 7, 5 (2006) 10-7.
[9] H. D. Stipho. J Prosthetic Dentistry, 79, 5 (1998) 580-584.
[10] Amjad Rahamneh, Pakistan Oral & Dental Journal, 29, 1 (2009) 181-183.
[11] Xiaosong Huang, Materials, 2, 4 (2009) 2369-2403.
[12] D. C. Jagger, A. Harrison, K. D. Jandt, J. of Oral Rehabilitation, 26, 3 (1999) 185-194.
[13] S. Y. Chen, W. M. Liang, P. S. Yen, J. Biomedical Materials Research, 58, 2 (2001) 203-208.
[14] I. H. Tacir, J. D. Kama, M. Zortuk, S. Eskimez, Australian Dental J., 51, 1 (2006) 52-56.
[15] T. Kanie, K. Fujii, H. Arikawa, K. Inoue, J. Dental Materials, 16, 2 (2000) 150-158.
[16] N. Yazdanie & M. Mahood, J. Prosthetic Dentistry, 54, 4 (1985) 543-547.
[17] J. Özen, C. Sipahi, A. Çağlar, M. Dalkiz, Turkish J. Medical Sciences, 36, 2 (2006) 121-126.

[18] Russel Rushdi, "Denture Base Modification by Reinforcement of Carbon Fiber/Hydroxyapatite and Study its Properties", Department of Applied Sciences, Master Thesis, University of Technology, Iraq, (2014).

[19] T. Nohrström, P. Vallittu, Yli-Urpo, Int. J. Prosthodont, 13 (2000) 72-78.

[20] R. A. Jaikumar, S. Karthigeyan, S. A. Ali, N. M. Naidu, R. P. Kumar, K. Vijayalakshmi, J. Pharmacy & Bioallied Sciences, 7, 2 (2015) 461-464.

[21] J. Engstrand, C. Persson, H. Engqvist, J. the Mechanical Behavior of Biomedical Materials, 29 (2014) 81-90.

[22] A. O. Alhareb, H. M. Akil, Z. A. Ahmad, J. Thermal Analysis and Calorimetry, 134, 2 (2018) 941-951.

[23] A. O Alhareb & Z. A. Ahmad, J. Reinforced Plastics and Composites, 30, 1 (2011) 86-93.