

Effect of poly propylene fibers incorporation and their oxygen plasma treatment on tensile strength, wettability and wear resistance of heat cure denture base acrylic resin

Hasanain K.A. Alalwan, B.D.S. M.Sc. ⁽¹⁾

ABSTRACT

Background: Recently, Poly propylene fibers with and without plasma treatment have been used to reinforce heat cure denture base acrylic but, so far some of properties like tensile strength, wettability and wear resistance not evaluated yet, the aim of the study is to clarify the influence of incorporation of treated and untreated fibers on these properties.

Materials and methods: Twenty one specimens were fabricated for every tested property (tensile strength, wear resistance and wettability) that classified into three groups (control, untreated poly propylene fibers reinforced specimens and Oxygen plasma treated group) and for each test seven samples were used (n=7). Tensile strength was tested using Instron universal testing machine, wear resistance was evaluated by pin on disk wear measurement method while, a digital microscope supplied with high resolution camera was utilized to measure the contact angle reflecting wettability grade. One way ANOVA table and LSD were used to analyze the results.

Results: The tensile strength showed a paradoxical effect, plasma treated samples group demonstrated a negative influence in contrary, to the untreated group that revealed positive high significant impression. The plasma treated group had manifested a- positive significant difference regarding wear resistance whilst, the beneficial effect of treated and untreated Poly propylene fibers addition was obvious with high significant difference.

Conclusion: Incorporation of O₂ plasma treated and untreated poly propylene fibers to heat cure poly methyl methacrylate resin was beneficial regarding the tested properties except for addition of plasma treated Poly propylene fibers on tensile strength.

Key words: Poly propylene fibers, tensile strength, wettability, wear resistance, denture base. (J Bagh Coll Dentistry 2014; 26(4):32-38).

الخلاصة

الخلفية: حديثاً الألياف البولي بروبيلين مع وبدون معالجة بلازما الأوكسجين قد استخدمت في تقوية الراتنج الأكريلي الحراري لقاعدة الطقم لكن لحد الان لم يختبر تأثيرها على قوة الشد وقابلية الترطب ومقاومة البلى.

المواد والأساليب: إحدى وعشرين عينة قد اختبرت لكل صفة من الصفات الثلاثة بواقع سبعة نماذج اختيارياً لكل من مجموعة السيطرة ومجموعة النماذج المدعمة بالألياف المعالجة بلازما الأوكسجين والمجموعة الأخيرة للألياف غير المعالجة اختبرت قوة الشد بجهاز الإنسترون واختبرت قابلية الترطب بمايكروسكوب رقمي وفحصت مقومة البلى بطريقة المسار على القرص. النتائج: أظهرت النتائج تبايناً واضحاً عند فحص قوة الشد لصالح إضافة الألياف غير المعالجة بينما بينت تأثير سلبي للألياف المعالجة أما ما يخص الصفات الأخرى فقد أظهرت تأثيراً إيجابياً خصوصاً في صفة قابلية الترطب.

الاستنتاج: مع محدودية البحث يستنتج بأن إضافة الألياف المعالجة وغير المعالجة اثر إيجابياً بالصفات المدروسة في ما عدا قوة الشد مع الألياف المعالجة

INTRODUCTION

Fiber reinforced polymer resin composites have sorted several types of fibers with diverse traits. Carbon fibers revealed poor esthetics, glass fibers manifested hydrolytic degradation and corrosion leading to strength reduction and destruction of polymer fiber interface ^(1,2). Poor esthetics and polishability were apparent with aramid fibers ⁽³⁾. Ultra high modulus Poly ethylene Fibers reinforcement have been researched for several years and these studies showed controversial reinforcing effects ⁽⁴⁻⁶⁾. While, poly propylene fibers (PP fiber) has been harnessed to reinforce denture base acrylic resin recently, Polypropylene fiber is considered as one of the polyolefin synthetic fibers that characterized with strength, staining and abrasion resistance ⁽⁷⁾. Impact strength was the most obvious improved property moreover, Oxygen plasma treatment have been relied on to enhance fiber-matrix impregnation ^(8,9).

The influence of PP fiber incorporation on tensile strength, wear resistance and wettability have not tested yet. A low-temperature glow-discharge Oxygen plasma impact on the structure

(1)Assistant Lecturer. Department of Prosthodontics, College of Dentistry, University of Baghdad

and properties of exposed materials especially polymers results in etching and changes in a micro-structure of the surface. As a consequence of the change in a surface layer structure, a number of functional properties, in particular, adhesion, sorption and tribology are changed ⁽¹⁰⁾.

Noort have attributed inclination of dentures to fracture to low tensile strength which is of no more than 50 MPa, low elastic and flexural modulus and lack of fracture toughness ⁽¹¹⁾. Wear resulted from removal and relocation of materials via the contact of two or more materials leading to material loss. Wear resistance is deemed as one of the properties that be judiciously developed by composite ⁽¹²⁾. In bite splint fabrication, heat cure acrylic is utilized where high wear resistant material is usually preferred to keep the new occlusal relation and withstand opposing natural or artificial dentition ⁽¹³⁾. Denture base material wettability play an effective role in denture retention, complete wetting of denture surface increase the capillary force therefore, raising denture retention in static and dynamic situations ⁽¹²⁾. In this study, the reinforcing effect of poly propylene fibers incorporation to poly methyl methacrylate and their Oxygen plasma

treatment effect on tensile strength, wear resistance and wettability have evaluated.

MATERIALS AND METHODS

Incorporation of (6 mm length) 2.5 % (by weight) poly propylene fibers (PP fibers) (Cracecmfiber®) (figure1) to poly methylmethacrylate heat cure powder (PMMA)(SUPERACRYL® PLUS, Czech Republic-cross linked) in this study was according to recent study that have showed an incorporation of that percentage had the best effect regarding impact and transverse strengths which might considered main properties in measuring the strength of materials⁽⁹⁾.



Figure 1: Poly propylene fibers

The specimens were grouped to three groups for each one of the three tests (tensile strength, wear resistance and wettability test) of this study :control group, specimens with untreated PP fibers and the third group was specimens with Oxygen plasma treated PP fibers whereas the number of specimens for each group was seven(n=7).PP fibers was treated with Oxygen plasma using DC- glow discharge system device with 6 minutes exposure time (figure2), gas pressure was limited to 0.5×10^{-1} mlbar while the flow rate of gas was 10 ml/ min using flowmeter (Ministry of Science and Technology-Iraq, physics department).

FTIR (Fourier Transform Infrared Spectrophotometer, SHIMADZU, 8400S Japan) (figure3) was utilized to find out and identify functional groups PP fibers surface involvement (figure4a,b). Test specimens elaboration was done via making plastic patterns cut with highly precise laser turning machine. Conventional flasking method has undertaken after mould preparation to form specimens. All the specimens for all the three tests were finished and polished.



Figure 2: Plasma chamber



Figure 3: FTIR device

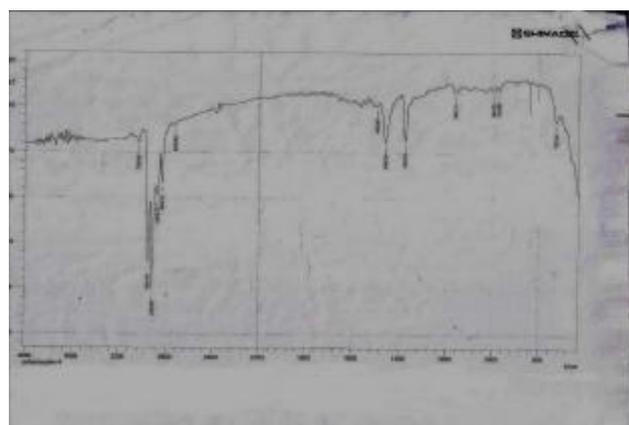


Figure 4a: FTIR of untreated PP fibers.

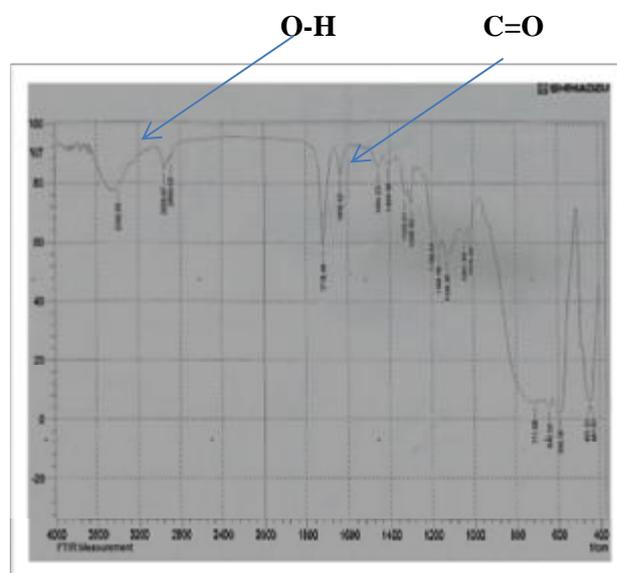


Figure 4b: FTIR of treated PP fibers with (C=O, 1719) and (O-H, 3382) functional groups.

Incorporation of polypropylene fibers

2.5% by weight added to the subtracted weighed powder. The required weight of the fiber and powder was measured by electronic balance (with accuracy 0.0001g). The weight of fibers was secured by the following equation:

Fiber reinforcement percentage % = weight of fiber/weight of fiber+ weight of matrix. Powder and PP fibers mixing were achieved using mortar and pestle till uniform mixture was obtained. Mixing with monomer was done incrementally for throughout dispersion of the fibers.

Tensile strength test

Dumbbell shaped specimens were fabricated according to ASTM specification D-638M (1986) with dimensions as explicitly illustrated in figure 5.

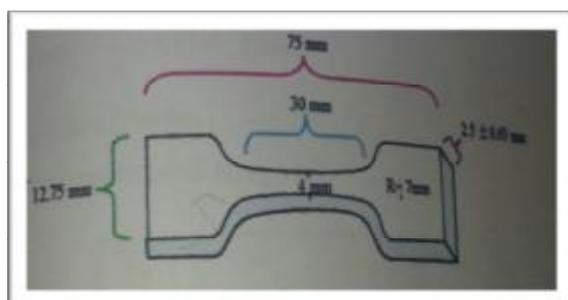


Figure 5: Dimensions of tensile test pattern.

Afterwards, they were conditioned in distilled water for two days at room temperature $25\text{c}^{\circ}\pm 2$ before testing according to ADA specifications. The testing procedure was accomplished using Instron universal testing machine (LARYEE, make test easy, china) with crosshead speed 0.5 mm/min and maximum loading of 20Kg. Tensile strength was calculated electronically via the software of the testing machine. (figure 6)



Figure 6: Tensile strength PP fiber reinforced specimen after submitting to the test.

Wear resistance test

Pin on disk wear testing device was used that recognized with high reproducibility of results^(14, 15), designed at University of Technology, Material engineering department, resistance laboratory-Iraq. The specimens were cylindrical in shape with dimensions of 20mm length and 10mm diameter according to the device requirement (figure 7). The pin was held specimen while the disk was stainless steel wheel which revolve 950 r/min, the specimen was weighed before and after the testing procedure, after securing the specimen to the holder and putting of 10N load on the horizontal arm, the device switched on for 10 minutes (wear testing time)⁽¹⁶⁾. The distance between the center of the disk and center of the specimen was 65mm. The wear resistance was calculated with the following equation: wear resistance (gr/mm) = change in weight/slide distance (slide distance = $2\pi \times \text{radius}$ distance between centers of disk and specimen \times number of cycles \times time of test)⁽¹⁶⁾. The disk was cleaned with abrasive grit paper after each test (figure 8). All specimens were immersed in distilled water for 48 hours before testing.



Figure 7: Wear specimens



Figure 8: Pin on disk mould with held specimen.

Wettability

Contact angle measurement was harnessed to determine the wettability; the angle was between the base of the sessile drop and the tangent line contacting the solid, liquid and air simultaneously. Dispersing of distilled water $10\mu\text{L}$ at the center of the fabricated sample using pipette. Dimensions of the sample $8 \times 30 \times 2\text{mm}$ width, length and thickness respectively⁽¹⁷⁾. Static sessile drop method was utilized in this study. As the water dispersed wait for 5 minutes to allow the drop to be in status of equilibrium. Measurement of the contact angle was accomplished employing a digital microscope (Dino-lite digital microscope pro -Taiwan) at magnification $45\times$ supplied with camera (high resolution 1024×768 pixel) and software (Dino capture 2.0 Version 1.3.3.) granted

a complete and precision analysis to the contact angle.(figure9)



Figure 9: Digital microscope with specimen.

Descriptive statistical analysis involving: arithmetic mean, standard deviation and standard error in addition to inferential statistical analysis including: F- test by Analysis of Variance and least significant difference (LSD) was

conducted for further verification. $P > 0.05$ NonSignificant (NS), $P \leq 0.05$ Significant (S) and $P \leq 0.001$ High Significant (HS).

RESULTS

Tensile strength

Untreated Polypropylene fiber reinforced samples showed the higher mean (58.2571) while the treated revealed the lowest (53.6857) as illustrated in table 1. ANOVA table- in table 2- displayed a statistical significant difference ($P=0.003$) among the studied groups. Table 3 demonstrated a positive significant effect of the untreated PP fiber reinforced samples over the control, in opposite to non-significant relation of control to treated PP fiber reinforced samples, while a high significant difference ($P=0.001$) between treated and untreated PP fiber samples in favor of untreated one.

Table1: Descriptive statistics of tensile strength test Mpa among the studied groups.

Group	N	Mean	S. D.	S. E.
Control	7	54.458	2.2063	0.8339
Untreated PP F	7	58.257	2.9102	1.0999
Plasma treated PP F	7	53.685	1.4690	0.5552
Total	21			

Table2: ANOVA table of tensile strength test among the studied groups.

F	P-value	Sig.
8.114	0.003	S

Table 3: LSD of tensile strength test groups

Study group	Study group	Mean Difference	P-value	Sig.
Control	Untreated PP fiber	-3.79857	.006	S
Control	treated PP fiber	0.77286	.533	NS
Untreated PP fiber	Plasma treated PP fiber	4.57143	.001	HS

Plasma treated PP fiber reinforced specimens exhibited the highest wear resistance because they showed the lowest weight loss per millimeter (0.000010), whilst the control manifested the lowest wear resistance (0.000012) moreover, ANOVA table presented a significant difference

among tested groups ($P=0.030$) as shown in table 4 and table 5. Multiple comparison with LSD (table 6) revealed a non-significant difference between groups except between control and plasma treated PP fiber reinforced specimens ($P=0.009$).

Table 4. Descriptive statistics of wear resistance (gm/mm) test among the studied groups.

Group	N	Mean	S. D.	S. E.
Control	7	0.000012	.0000008	.0000003
Untreated PP F	7	0.000011	.0000013	.0000005
Plasma treated PP F	7	0.000010	.0000018	.0000007
Total	21			

Table 5: ANOVA table of wear resistance test among the studied groups.

F	P-value	Sig.
4.311	0.030	S

Table 6: LSD of wear resistance test among studied groups

Study group	Study group	Mean Difference	P- value	Sig.
Control	Untreated PPfiber	.0000013	.097	NS
Control	treated PPfiber	.0000021	.009	S
Untreated PP fiber	Plasma treated PPfiber	.0000009	.259	NS

Wettability

Table 7 offers the effect of incorporation on the mean contact angle among studied groups, the highest mean was in control group so, represented the lowest wettability however, the Oxygen plasma treated PP fiber reinforced specimens showed the contrary. The evidence on the strong

effect of PP fibers incorporation and their plasma treatment on wettability of PMMA has proved by ANOVA table and further affirmed statistically with LSD, where a high significant difference between each two different groups as illustrated in table 8 and table 9.

Table7: Descriptive statistics of wettability test among the studied groups.

Group	N	Mean	S.D.	S.E.
Control	7	45.6039	2.701025	1.020892
Untreated PP F	7	35.7269	3.355893	1.268408
Plasma treated PP F	7	27.8051	2.753189	1.040608
Total	21			

Table 8: ANOVA table of wettability test among the studied groups.

F	P-value	Sig.
63.887	.000	HS

Table 9: LSD of wettability test among studied groups

Study group	Study group	Mean Difference	P-value	Sig.
Control	Untreated PP fiber	9.877000	.000	HS
Control	Plasma treated PP fiber	17.798714	.000	HS
Untreated PP fiber	Plasma treated PP fiber	7.921714	.000	HS

DISCUSSION

The negative effect of plasma treatment to PP on the tensile strength was strong enough to clearly reduce it, it could be inferred to the water absorption of PP fibers was improved by plasma treatment. This fact was caused by the creation of polar C=O and O-H groups on the surface after plasma treatment. It happens due to plasma probably breaks the C-H and/or C-C bonds creating free radicals, which can react with the activated oxygen species in the discharge leading to the formation of these moieties, this is in agreement with Skacelova et al and Morent et al (18, 19).

Absorption of water by PMMA accomplished slowly over a period of time, essentially because of the polar properties of the resin molecules. High water uptake equilibrium can soften an acrylic resin because the absorbed water acts as a plasticizer and reduce the strength of the material (20). The incidence of water uptake into PMMA networks are substantially controlled by resin polarity, affected by the concentration of polar groups available to form hydrogen bonds with water and network topology (21), therefore, incorporation of oxygen plasma treated poly

propylene fibers to PMMA had increased the water absorption significantly (9) and PMMA tensile strength is negatively affected by water absorption and ambient moisture, this is in agreement with Ishiyama et al (22), in addition to the acrylic used in this study was of cross linked type that might exaggerate the water absorption (23). Furthermore, a previous study conducted to review all the articles related to fiber reinforced denture base materials had noticed that plasma treatment for UHMPE fiber had not increased PMMA strength in comparison to untreated (2). Generally, plasma treatment for fibers can lessen the tensile strength of the fibers themselves due to their loss of weight and diameter reduction (24,25).

On the other hand, two essential properties of PP fiber are moisture absorption very low capability and good toughness (26) thus, the incorporation of untreated PP fiber has increased the tensile strength while, the plasma treated was equivalent to control group where the benefit of PP fiber addition had cancelled after changing the surface of the fibers and making them more hydrophilic, that encouraged water absorption and weakened the material. Poor impregnation of the untreated fibers did not precipitate a negative

influence, it could be attributed to solicitude during fiber-powder blending and homogenous mixing with monomer that distribute the fibers and prevent their accumulation.

Wettability of acrylic reinforced with untreated and Oxygen plasma treated PP fiber was highly magnified, regarding the treated PP, it might be elucidated by increasing the water affinity of the PP fiber itself via plasma treatment this is in agreement with several researchers⁽²⁷⁻²⁹⁾, that overshadowed on PMMA-PP fiber composite wettability and occurrence of new functional groups as annotated above guiding the change in a surface layer structure. Wettability of the untreated PP fibers incorporated samples was increased considerably because the addition of randomly oriented PP fibers elevates the surface roughness even non-significant statistically⁽⁹⁾, the surface roughness and wettability are directly proportional⁽³⁰⁾, consequently, the untreated PP fiber samples wettability had raised.

High abrasion resistance of PP fiber⁽²⁶⁾ had increased the untreated PP fiber reinforced samples wear resistance yet, that not significant statistically however, after plasma treatment it significantly improved in comparison to control group and this may be attributed to formation of functional groups on the pendant methyl group of the isotactic PP fiber leading to increased crosslinking that decreased plastic flow which in turn increased the wear resistance and that was consistent with Kyomoto et al and Drobny^(31, 32).

Resin composite capability to withstand wear relies on filler–matrix interactions and highly sensitive for spacing between them, the Oxygen plasma treatment had played that role and increased the wear resistance⁽³³⁾. Finally, it can be concluded that incorporation of O₂ plasma treated and untreated PP fibers to heat cure PMMA was beneficial regarding the tested properties except for addition of treated PP fiber on tensile strength.

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