The effect of addition of untreated and oxygen plasma treated polypropylene fibers on some properties of heat cured acrylic resin

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ABSTRACT

Background: The polymethyl methacrylate is the most reliable material for the construction of complete and partial dentures, despite satisfying esthetic demand itsuffered from having unsatisfactory properties like impact strength and transverse strength.

This study was designed to improve the impact strength and transverse strength of heat cure acrylic resin by adding untreated and oxygen plasma treated polypropylene fibers and investigate the effect of this additive on some properties of acrylic resin materials.

Materials and methods: Untreated and oxygen plasma treated polypropylene fibers was added to PMMA powder by weight 2.5 %. Specimens were constructed and divided into 5 groups according to the using tests; each group was subdivided in to 3 subgroups. The tests conducted were impact strength, transverse strength, surface hardness, surface roughness, water sorption and solubility. Data were analyzed by one way analysis of variance (ANOVA) and least significant differences (LSD).

Results: After incorporation of untreated and oxygen plasma treated polypropylene fibers there was a highly significant increase in impact strength and surface hardness; while there was a non significant difference in transverse strength. Also the value of water sorption increase significantly but with the limit of ADA specification.

Conclusion:Within the limit of this study it can be concluded that the reinforcement with polypropylene fibers effective method to increase fracture resistance of denture base; while reinforcement with oxygen plasma treated polypropylene fibers further increase fracture resistance.

Key words: Acrylic resin, Impact strength, Transverse strength, Polypropylene fibers, Plasma treatment. (J Bagh Coll Dentistry 2013; 25(4):33-38).

INTRODUCTION

Polymethyl methacrylate has proved to be the most satisfactory denture base material currently available. Despite satisfying esthetic demands it is far from ideal in fulfilling the mechanical requirements of prosthesis. The main problem associated with PMMA as a denture base material, is unsatisfactory strength particularly under fatigue failure inside the mouth caused by occlusal biting force and impact failure outside the mouth by dropping the dentures [(1)].

Numerous attempts have been used to strengthen PMMA denture base resin such as incorporation of metal wire, the primary problem of using metal wire is poor adhesion between wire and acrylic resin matrix [(2)], production of alternative polymer slike polystyrene and polycarbonate, but have not been shown to produce dentures of greater accuracy with better performance [(3)], and incorporation of rubber phase in the bead polymer has improved the impact strength but result in increased cost [(4)]. The other approach is the reinforcement of PMMA denture base resin with fibers such as glass fibers, carbon fibers, aramide fibers, nylon fibers and polyethylene fibers [(5)].

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In this study is going to use type of olefin fibers named polypropylene (pp) as reinforcing filler to PMMA denture base resin which has many properties like high strength, good surface finish and polish, low cost and excellent biocompatibility [(6)].

However, these fibers break- up the homogenous matrix of acrylic resin due to poor interface between fiber and resin affecting the mechanical properties. In order to avoid this, the polypropylene fiber surface energy increased by chemical or plasma treatment [(7)]. A method based on cold plasma treatment represents an environmentally attractive alternative able to replace chemical methods, with plasma treatment surface chemistry and topography may be influenced to result in improved adhesion [(8)].

MATERIALS AND METHODS

One hundred fifty acrylic specimens were constructed by conventional flasking technique using heat cure acrylic resin (SUPER ACRYL®PLUS) the samples were divided into five groups according to the using tests and each group sub divided into three subgroups.

Three different plastic patterns were constructed by cutting plastic plates with different thickness into desired shape and dimension using highly accurate laser cutting machine. These plastic patterns were used in formation of mold

for construction of the specimens by conventional flasking technique [9]. The required weight of the powder of the polymer and polypropylene fibers was weighted by using digital electronic balance for each group. Mixing of polymer powder and fibers was done randomly by using mortar and pestle until homogenous mixture was attained.

Mechanical and physical tests

A- Impact strength test

The specimens were prepared with dimensions (80mm x 10mm x 4mm) (ISO 179, 2000) for unnotched specimens. Specimens were stored in distilled water at 37^0C for 48 hours before being tested [9].

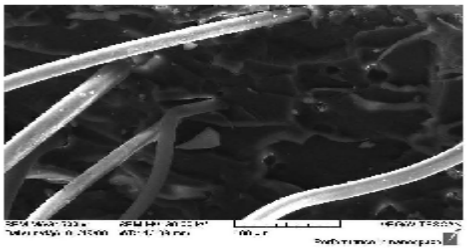
The impact strength test was evaluated following the procedure recommended by the ISO 179 with impact testing device. The specimens were supported horizontally at each end and struck by free swinging pendulum of 2 Joules. The scale readings give the impact energy in Joules. The charpy impact strength of unnotched specimens was calculated in Kilo joules per square meter by the following equation: Impact strength = $\frac{E}{b \cdot d} X10^3$ (ISO, 2000)

E: The impact energy in Joules

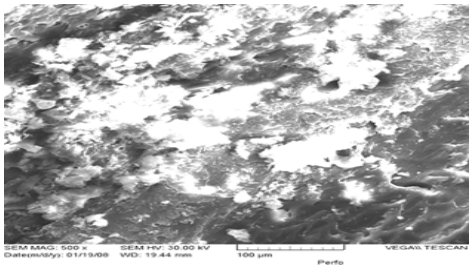
b: Is the width of the specimens in millimeters

d: Is the depth of the specimens in millimeters

Then the fracture surface of specimen examined and photographed using Scanning Electroning Microscope (SEM) to study the difference in adhesion before and after plasma treatment.



A-before treatment



B- after treatment

Figure 1: Scanning Electroning Microscope for fracture surface of specimen (A, B)

B- Transverse strength test

Specimens were prepared with dimensions (65mm x 10mm x 2.5 ± 0.1mm). All specimens stored in distilled water at 37 ^{0}C for 48 hours before being tested [9].

The test was performed using Instron universal testing machine (WDW-200 E), each specimen was positioned on the bending fixturewhich consist of two parallel supports (50 mm apart), the full scale was 50 Kg andthe load was applied with across headspeed of1mm/min. by a rod placed centrally between the supports making deflection until fracture occurs.

C- Surface hardness test

Specimens of heat cure acrylic resin were prepared with a dimension (65mm x 10mm x 2.5 ± 0.1mm). All specimens were stored in distilled water at 37^0C for 48 hours before being tested [9]. Surface hardness was determined by using (Shore D) durometer hardness tester which is suitable for acrylic resin material.

The instrument consist of spring - loaded indenter (0.8mm in diameter), the indenter is attached to digital scale that is graduated from 0 to 100 units. The usual method is to press down firmly and quickly on the indenter and record the reading. Three readings were done on each specimen (one in the center and other at each end) then the mean of three readings was calculated.

D- Surface roughness test

Specimens with dimensions (65mm x10mm x 2.5± 0.1mm) were prepared to be used for surface roughness test. All the specimens were stored in distilled water at 37^0C for 48 hours before being tested [9].

The profilometer device was used to study the effect of fiber reinforcement on the microgeometry of the test surface. This device is supplied with sharp stylus surface analyzer from a diamond to trace the profile of the surface irregularities by recording of all the peaks and recesses which characterized the surface by its scale. The acrylic specimen was placed on its stable stage and the location of the tested area was selected (The specimen was divided into three parts) then the analyzer was traversed along the tested area and the mean of three readings was calculated.

E- Water sorption and solubility test

Acrylic disc specimens were prepared by using plastic pattern having dimensions of (50mm±1mm in diameter and 0.5 mm ±0.1 mm in thickness).

The specimens were dried in desiccators containing freshly dried silica gel .The desiccator was stored in an incubator at a37^{0}C $\pm$2^{0}C for 24 hours after that the specimens were removed to room temperature for one hour then weighted with electronic balance with accuracy of (0.0001g). This cycle of weighting was repeated every day until a constant mass (M1) (conditioned mass) was reached [9].

All discs of all groups were immersed in distilled water for 7 days at 37^{0}C $\pm$ 2^0 C [9]. The discs were removed from the water with a dental tweezers wiped with a clean dry towel until free from visible moisture, waved in the air for 15 seconds and weighted; this mass was recorded as (M2).

The value of water sorption was calculated for each disc from the following equation:

$$WSP = \frac{M2 - M1}{S}$$ (ADA specification No.12, 1999)

WSP = Water sorption in mg/cm^2

$M2$ = The mass of the disc after immersion in distilled water (mg)

$M1$ =The mass of the disc before immersion in distilled water (conditioned mass) (mg).

S = Surface area of the disc (cm^2)

In order to obtain the value of water solubility the discs were again reconditioned to a constant mass in the desiccator at 37^{0}C $\pm$ 2^{0}C as done in the first time for sorption test and the reconditioned mass was recorded as (M3). The solubility during immersion was determined for each disc by the following equation:

$$WSL = \frac{M1 - M3}{S}$$ (ADA specification No.12, 1999)

WSL= Water solubility in mg/ cm^2.

M1= the conditioned mass (mg).

M3= the reconditioned mass (mg).

S= the surface area of the disc (cm^2).

RESULTS

Statistical analysis was done by using SPSS version 20.The results obtained from the measured data were classified according to the followings experimental groups:

- Group (A) Control group
- Group (B) Acrylic resin+ untreated polypropylene fibers
- Group (C) Acrylic resin + 4 minutes oxygen plasma treated polypropylene fibers

Impact strength test

The result of this test showed that group (C) exhibited the highest impact strength mean value (10.986 Kj/m^2); while the group (A) exhibited the lowest one (7.190 Kj/m^2).

One way analysis of variance (ANOVA test) indicated a highly significant difference among the studied groups (P< 0.01).

Table 1: Descriptive data and ANOVA test of impact strength test among studied groups

Studied groups		Descriptive		ANOVA test	
	N	Mean Kj/m^2	SD	Between groups	
Control	10	7.190	0.864	df	2
Untreated polypropylene fiber	10	9.705	0.671	F	69.976
Plasma treated polypropylene fiber	10	10.986	0.634	P- value	0.000 **HS**

Transverse strength test

One way analysis of variance (ANOVA test) indicated a non significant difference among all groups (P >0.05)

Surface hardness test

The result of this test showed that group (C) had the highest mean value (86.517); while group (A) had the lowest one (83.318).

One way analysis of variance (ANOVA test) indicated a highly significant difference among studied groups (P < 0.01).

Table 2: Descriptive data and ANOVA test of transverse strength test among studied groups

Studied groups		Descriptive		ANOVA test	
	N	Mean N/mm^2	SD	Between groups	
Control	10	95.63	3.271	df	2
Untreated polypropylene fiber	10	96.801	3.766	F	0.646
Plasma treated polypropylene fiber	10	97.523	4.187	p- value	0.532 **NS**

Table 3: Descriptive data and ANOVA test of surface hardness test among studied groups

Studied groups		Descriptive		ANOVA test	
	N	Mean	SD	Between groups	
Control	10	83.318	2.172	df	2
Untreated polypropylene fiber	10	86.447	1.098	F	10.746
Plasma treated polypropylene fiber	10	86.517	1.843	p- value	0.000 **HS**

Surface roughness

The result of this test showed that group (C) had the highest mean value **(**0.915µm**);** while group (A) had the lowest one (0.903 µm).

One way analysis of variance (ANOVA test) indicated a highly significant difference among studied groups (P < 0.01).

Table 4: Descriptive data and ANOVA test of surface roughness test among studied groups

Studied groups		Descriptive		ANOVA test	
	N	Mean µm	SD	Between groups	
Control	10	0.903	0.007	df	2
Untreated polypropylene fiber	10	0.908	0.008	F	7.135
Plasma treated polypropylene fiber	10	0.915	0.006	p- value	0.003 **HS**

Water sorption test

The result of this test showed that group (C) had the highest mean value **(**0.409 mg/cm^2**);** while group (A) had the lowest one (0.357mg/cm^2).

One way analysis of variance (ANOVA test) indicated a highly significant difference among studied groups (P < 0.01).

Table 5: Descriptive data and ANOVA test of water sorption test among studied groups

Studied groups		Descriptive		ANOVA test	
	N	Mean mg/cm^2	SD	Between groups	
Control	10	0.357	0.017	df	2
Untreated polypropylene fiber	10	0.386	0.026	F	12.571
Plasma treated polypropylene fiber	10	0.409	0.026	p- value	0.000 **HS**

Water solubility test

The result of this test showed that group (B) had the highest mean value **(**0.02 mg/cm^2**);** while group (A) had the lowest one (0.018mg/cm^2).

One way analysis of variance (ANOVA test) indicated significant difference among studied groups (P < 0.05).

Table 6: Descriptive data and ANOVA test of water solubility test among studied groups

Studied groups		Descriptive		ANOVA test	
	N	Mean mg/cm^2	SD	Between groups	
Control	10	0.018	0.001	df	2
Untreated polypropylene fiber	10	0.02	0.002	F	3.520
Plasma treated polypropylene fiber	10	0.019	0.002	p- value	0.044 **S**

DISCUSSION

In this study used plasma treatment rather than chemical treatment for polypropylene fibers since plasma a convenient procedure and environmentally friendly technique [8].

Oxygen- containing plasmas were most commonly employed to improve polymer surface properties, these results might be due to the effects of chemical oxidation reactions and/ or chemical etching process. During the oxidation reactions, plasma promotes adhesion by inducing further chemical reactions with generated new chemical functional groups such as the hydroxyl group which increased the surface energy; while during the chemical etching process, this process result in chemical removal of surface material that increased the effective surface area of the polymer (i.e., surface roughning) this roughing in turn promote more intimate molecular contact between the plasma exposed fiber surface and the matrix allowing for stronger bond to occur [10].

Impact strength

The results revealed that the addition of untreated polypropylene fibers produced a highly significant increase in impact strength mean value

compared with control group, this increase which could be related to the presence of fibers which prevent the crack propagation and change in direction of cracks resulting in smaller cracks between the fibers, this can be correlated to the increased impact strength of fiber-reinforcedspecimens compared to the unreinforced specimens where there is unobstructed crack propagation .

These results are in agreement with results obtained by Mowade et al [6]. There was also a highly significant increase in impact strength mean value of specimens after incorporation of plasma treated pp fibers compared with control group, this increase could be attributed to the fact that plasma introduce functional groups on the surface of fibers there by making the surface polar, which improve the surface energy of the fiber and its compatibility with other materials [11] therefore, enhance the impact strength. These results are in agreement with results obtained by Mowade et al [6].

Transverse strength

The results revealed that the addition of untreated polypropylene fibers produced non significant difference in transverse strength mean value compared with the control group, this may be related to the fact that the random orientation of fibers allows only small portion of the reinforcement to be directed perpendicular to the applied stress.

Unalan et al [12] and Kamble et al [13] found that reinforced acrylic with 2% by weight of glass and polyethylene fibers improved the flexural strength of the specimens compared to unreinforced PMMA and bis- acryl composite resins; while Al-Momen [14] found after the addition of 5% and 10% Styrene Butadiene Rubber into acrylic resin produce a significant decrease in transverse strength was observed duo to increase in flexibility of composite containing SBR.

There was also non significant difference in transverse strength mean value after incorporation of plasma treated polypropylene fibers into PMMA resin compared with control group, this due to the internal voids formed in the resin- fiber composite caused by poor wetting of fibers with resin (perhaps the using fibers not undergo changes from plasma treatment), these voids were oxygen reserves that allowed oxygen to inhibit radical polymerization of the acrylic resin inside composite,this can lead to higher residual monomer content of fiber composite and affect strength.

Surface hardness

The results revealed that the addition of untreated polypropylene fibers produce a highly significant increase in surface hardness mean value compared with control group; this increase could be related to the presence of these fibers near or at the surface of the composite which extremely hard and stiff.

Al- Momen [14] and Salih [15] they found a remarkable increase in the hardness observed when the randomly oriented form of Kevlar, glass and carbon fibers were added to resin.

There was also a highly significant increase in surface hardness mean value after incorporation of plasma treated pp fibers to PMMAresin compare with control group this could be attributed to that treatment increase the fiber hardness.

Ahmad and Wel [16] showed that addition of saline coupling agent only improved the interfacial bonding between the matrix and glass fibers without giving any perceptible impression to the value of hardness.

Surface roughness

The results revealed that the addition of untreated polypropylene fibers produce non significant difference in surface roughness compared with control group, this could attributed to smooth surface of polypropylene fibers.

Waltimo et al [17] found significant increase in surface roughness with glass fibers reinforcement.

There was a highly significant increase in surface roughness mean value of specimens after incorporation of plasma treated pp fibers compared with control group, this increase could be attributed to fact that oxygen – plasma treatment increase the surface roughness of treated polymer [11].

Cvelbar et al [18] and Wei et al [19] revealed by using Atomic Force Microscope (AFM) that oxygen plasma treatment usually creates micro roughness on the treated surface due to an etching effect.

Water sorption

The results revealed that the incorporation of untreated polypropylene fibers produced a highly significant increase in the water sorption of acrylic resin when compared with control group and this increase could be related to the voids and defects formed at fiber/ matrix interface in poorly impregnated regions which more readily encourage water sorption.

There was also a highly significant increase in water sorption mean value of specimens after incorporation of plasma treated pp fibers

compared with control group; this increase could be attributed to fact that plasma treatment changed totally hydrophobic surface of untreated pp fibers to hydrophilic surface due to incorporating of functional groups [7].

Water solubility

The obtained results revealed that there was a significant increase in water solubility of the specimens reinforced with untreated polypropylene compared with control specimens, this increase could be attributed to the presence of air voids in the composite structure and the polymerization reaction inhibited by oxygen resulted in higher residual monomer content in the polymer [20] subsequent greater solubility of the polymerwill occur.

There was a non significant difference in water solubility of the specimens reinforced with plasma treated polypropylene fibers compared with control group, this attributed to the transverse interlocking occurred between the reinforced plasma treated polypropylene fibers and acrylic resin may lead to decrease in the residual monomer content subsequent lesser solubility of the polymer will occur.

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