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# Effect of Mixing Silanized Poly Propylene and Siwak Fibers on Some Physical and Mechanical Properties of Heat Cure Resin Denture Base

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## Abstract:

PMMA is used in construction of denture base due to several advantages, including biocompatibility and optimal appearance**.** Operators and patients complain from fracture of acrylic denture base material attributed to decreased resistance to impact, flexural or fatigue stresses. The aim of the research is to assess the effect of addition of silanized mixture of siwak and poly propylene fibers on PMMA denture base.

Fourteen acrylic resin specimens were prepared for each test (Impact strength, Transverse strength, Shear bond strength, Thermal conductivity, Shore D hardness, Surface roughness and Water sorption) the specimens were grouped as control group (n=7) :no fibers additive and experimental group (n=7):(2 %) silanized mixture of siwak and poly propylene fibers, 4 mm length, FT-IR was done to investigate the presence of functional groups of coupling agent (TMSPM) on the tested fibers, independent t-test was used for statistical analysis of the resulted data.

Statistical analysis indicated that silanized mixture of poly propylene and natural fibers (siwak) produced significant increase ($p \leq 0.05$) of impact strength and highly significant increase ($p \leq 0.01$) of shear bond strength, transverse strength, thermal conductivity and shore D hardness of heat cure acrylic resin while a non significant increase ($p > 0.05$) of surface roughness and water sorption properties was observed in comparison to non fiber reinforced resin specimens.

The addition of silanized mixture of siwak and polypropylene fibers into heat cure PMMA improve the tested physical and mechanical properties.

**Keywords**: *reinforcing fibers, acrylic resin, silane coupling agents, properties of resin denture base.*

## تأثير إضافة خليط من ألياف البولي بروبيلين والسواك بعد المعالجة السطحية على بعض الخصائص الفيزيائية و الميكانيكية لقاعدة طقم من الأكريل الحراري

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## الخلاصة:

يستخدم البولي مثيل ميثاكريليت في صناعة قاعدة أطقم الأسنان بسبب العديد من المزايا مثل التوافق الحيوي مع الأنسجة والمنظر الجميل الأمثل ولكن المرضى يعانون من كسر طقم الأكريلك بسبب إنخفاض المقاومة للصدمة وللقوة العرضية.

الهدف من هذه الدراسة هو تقييم إضافة خليط من ألياف البولي بروبيلين وألياف السواك الطبيعية المضادة للميكروبات على قاعدة طقم الأكريلك.

تم إعداد أربعة عشر من عينات راتنج الأكريلك الحراري لكل من الإختبارات التالية: قوة الصدمة, قوة المستعرضة, التوصيل الحراري, الصلادة السطحية, خشونة السطح, قوة الالتصاق القصي لطقم الأسنان وقابلية إمتصاص الماء. تم تقسيم العينات الى مجموعة السيطرة (7 عينات) والمجموعة التجريبية (7 عينات) التي تحتوي على 2% من

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خليط الألياف المعالجة سطحيا وبطول 4 ملم وقد تم إجراء فحص FTIR للتحقق من وجود مادة silane على سطح الألياف وتم تحليل النتائج بواسطة إختبار t- test للتحليل الاحصائي للبيانات.

أظهرت النتائج زيادة معنوية في قوة الصدمة, قوة المستعرضة, قوة الإلتصاق القصي لقاعدة الطقم, الصلادة والتوصيل الحراري لمادة راتنج الأكريلك الحراري المدعم بالألياف كما أظهرت النتائج إنخفاض غير معنوي في خشونة السطح وخاصية إمتصاص الماء عند المقارنة مع مجموعة السيطرة.

نستنتج من هذه الدراسة أن إضافة خليط من ألياف البولي بروبيلين وألياف السواك المعالجة سطحيا يؤدي الى تحسين الخصائص الفيزيائية والميكانيكية لمادة راتنج الأكريلك.

**الكلمات المفتاحية:** *الالياف الداعمة, راتنج الاكريلك, مادة السلين, خصائص راتنج قاعدة الطقم.*

## Introduction:

Most of the dental prosthesis is composed of poly methyl methacrylate (PMMA) due to many properties, including its color, durability, solubility and biocompatibility. Fracture of poly methyl methacrylate dentures occurs due to several reasons involving occlusal interferences, excessive load, careless handling and falling of dentures on hard surfaces[1,2,3].

Enhancement on mechanical properties of denture base materials were made either by cross linking agents like poly ethylene glycol dimethacrylate or by mixing rubber, metal oxides, metal wires or fibers [4, 5, 6, 7].

Many studies found that the reinforcing fibers such as Kevlar, carbon, glass, poly ethylene(PE) , poly propylene (PP) and silane treated glass have been added into (PMMA) to improve its mechanical properties, some commercially acrylic resin denture bases are mixed with reinforcing fibers [6, 9, 10].

Poly propylene fibers are considered as one of poly olefin synthetic fibers that characterized with strength, staining and abrasion resistance. These fibers are inert with high impact resistance, high ductility, neutral color low density and good biocompatibility [11, 12, 13].

Rigid fibers like glass fibers which are used to reinforce denture bases, it is necessary to coat the glass fibers with silane to increase the surface area and also to make chemical changes in the fiber creating covalent bond with (PMMA)[5,6,26].

Microbial adhesion and plaque accumulation on the surface of polymeric restorations are the main source of oral inflammations and fungal infections. Several studies review the inhibitory action of Miswak on gram-positive and gram-negative bacteria and fungi living in the mouth have been conducted. The findings confirmed that Miswak can be used as a dental hygiene strategy acting against various oral diseases. Recommendations and encouragement from the World Health Organization has been proved concerning the usage of chewing sticks as an effective and substitute method for oral hygiene (1984 and 2000 international consensus)[27,28].

Various projects have performed to strengthen the PMMA denture base involving macro and micro additives and fibers but no studies have been conducted to evaluate the effect of incorporation of anti microbial natural fibers (siwak) and synthetic poly propylene fibers on physical and mechanical properties of heat cure resin denture base (PMMA) has not been investigated.

The aim of this study was to add mixture of silanized poly propylene and siwak fibers (elastic synthetic and rigid antimicrobial natural fibers) to heat cure acrylic resin denture base material and evaluate the efficiency of these additives on certain mechanical and physical properties of the resulted polymer.

## Materials and Methods:

### Preparation of siwak fibers:

Salvadora persica (miswak) were dried by storing these sticks in a desiccator for three weeks. After each week the sticks

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were weighted by electrical sensitive balance with accuracy of (0. 0001g). When a constant weight was obtained it was indicated that the sticks were dried completely. Then these sticks were ground by using an electrical grinding machine. Selected sieves were used to separate the siwak powder from siwak fibers. The collected fibers were stored in screw top containers with sacs of silica gel granules in the tops.

**Preparation of acrylic resin specimens:**

Acrylic resin specimens (n=14) were prepared for each test, the specimens were grouped as follows:

A - Acrylic resin specimens with no fiber mixture addition (n=7, control group).

B - Acrylic resin specimens with fiber mixture addition 2% wt silanized siwak and poly propylene fibers, 4 mm in length (n=7, experimental group).

**Silanation of siwak and poly propylene fibers:**

Measured amount of pure toluene (200 ml) and 30 g of fibers mixture (in equal ration) were placed into a flask and sonicated for 20 minutes, then the mixture was placed into a flask on magnetic stirrer and 1.5g of TMPSM solution, (trimethoxy-silyl propyl methacrylate). (Silane, Sigma-Alderich Germany) was added into the toluene fibers mixture in order to create 5% wt silane addition, the mixture was left in the covered flask for 2 days, then placed in the rotary evaporator for 30 min, the modified fibers were left to dry in vaccum oven at 60˚C for 20 hours [15].

**FT-IR** analysis (Fourier Transform Infra photometer, SHIMADZU, Japan) was applied to assess the foundation of functional groups of TMPSM on the surface of tested fibers by identification of specific vibrations of functional groups[15], (Figure-9).

**The addition of silanized mixture of siwak and poly ethylene fibers to acrylic resin:**

Silanized Siwak and PP fibers (4 mm length)[37] were added to the PMMA powder (PYRAX, heat cure denture base polymer, India) in concentration of 2% wt, figure-1, table-1. The weight was measured by electrical balance with accuracy (0.0001g), mortar and pestle was used for even distribution and mixing of fibers with acrylic resin powder.

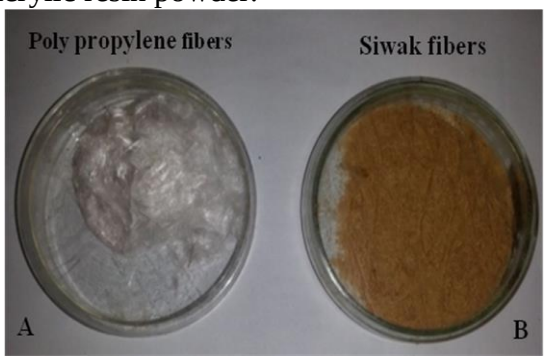


**Fig.1: Silanized. A, poly propylene fibers; B, siwak fibers.**

**Table-1: Ratio for mixing silanized siwak and PP fibers with PMMA powder.**

| Conc. Of addition % | Amount of fibers (g) | Amount of polymer (g) | Amount of monomer (ml) |
|---|---|---|---|
| 0 | 0 | 20 | 10 |
| 2% | 0. 2g : 0. 2g | 19. 6 | 10 |

PP: poly propylene fibers

The control and experimental acrylic resin specimens were constructed, finished regarding the manufacturer instructions. The Specimens were tested after being conditioned in distilled water at 37°C for 48 hours[8].

**Mechanical and physical tests evaluated in the research:**

The following mechanical and physical properties of modified acrylic resin were evaluated:

1 - Impact strength.

2 - Transverse strength.

3 - Shear bond strength.

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4 - Thermal conductivity.
5 - Shore D hardness.
6 - Surface roughness.
7 - Water sorption.

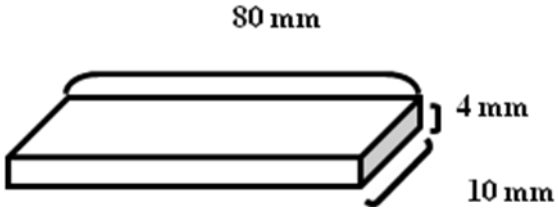
**Impact strength test:**

The resin specimens used were constructed with dimensions (80mmX 10mm X 4mm ± 0. 2mm), according to ISO. 179-1. 2000 (figure-2). 14 resin specimens were formed for the control and experimental groups for impact strength measurements.

The charpy impact strength of un notched specimen was measured in $KJ/m^2$according to the equation below:

Impact strength = $\frac{E}{b.d} X10^3$ (Anusavice, 2008)

E= energy in joules.
b= width of the resin specimens (mm).
d= thickness of the resin specimens (mm).




**(A)**

**(B)**

**Fig.2.A: Dimensions of acrylic resin specimen for impact strength.**
**B: Charpy impact testing device.**

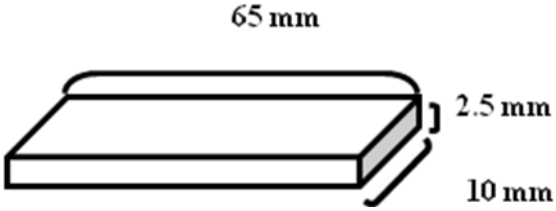
**Transverse strength test:**

The resin specimens were constructed with (65mm X 10mmX 2. 5mm ± 0. 2mm) length, width and thickness (international standard ISO-179) (figure-3). 14 specimens were formed for the control and the study groups for measurements of transverse strength. The test was conducted by positioning the specimen on the bending fixture using Ley-bold-Harris hydraulic press, consisting of 2 parallel supports, 50mm distance between the supports, the load was (7. 5 KN) for deflection.

The transverse strength was measured according to the following formula:

Transverse strength = $\frac{3Pl}{2bd^2}$

P= peak load.
l= span length.
b= width of resin specimen.
d= is the thickness of resin specimen. (Anusavice 2008).




**(A)**

**(B)**

**Fig.3.A: Dimensions of acrylic resin specimen for transverse strength.**
**B: hydraulic press.**

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**Shear bond strength test:**

The dimensions of acrylic block were (75, 13, 13 ± 0. 2mm) length, width and thickness with stopper of about 30 mm depth, two blocks were approximated with the cold silicone soft liner in between, (figure-4). Mollosil soft liner was mixed according to manufacturer instructions, after soft liner application between 2 acrylic resin blocks, 200g weight above the specimen and the material was left to set (5 min)[29].

The acrylic specimens were undergone to shear load with speed (0. 5mm/min) and load (50 Kg), the shear bond strength was measured by applying the following formula:

Bond strength= F (N)\A($mm^2$)
(ASTM specification, D-638m, 1986)
F= force of failure (Newton)
A= surface area of cross section ($mm^2$)

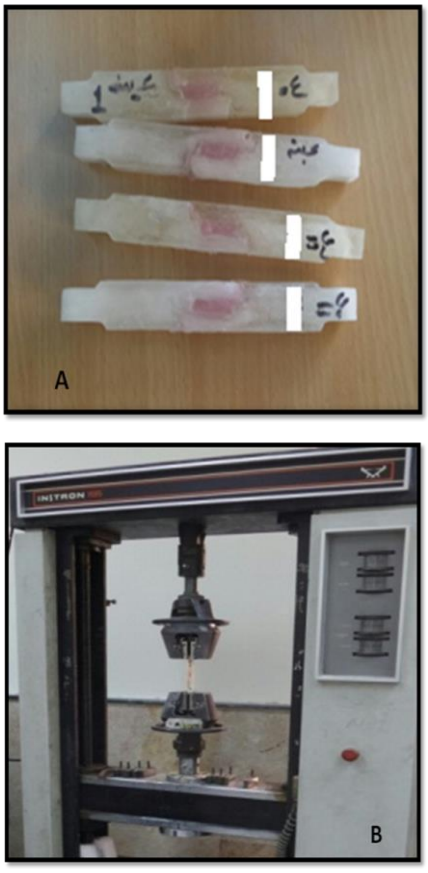


**Fig.4.A: Acrylic resin specimen with silicone soft liner in between.**
**B: Instron machine for shear bond strength measurement.**

**Thermal conductivity test:**

Seven specimens were prepared for each group (total: 14 specimens). The discs have a diameter (40mm) and thickness (2. 5mm) according to instrument specifications as shown in figure-5.

Thermal conductivity is calculated from the following equations:

$$K=\frac{e\ d}{2\pi\ r2\ (TB-TA)}\ [as\ TATB+2aA+TA]$$ [25]

as= surface area of specimen
e= the amount of thermal energy per unit area per second (W\m2. C)
K= thermal conductivity in w/m.°C
d= thickness of the specimen in mm
r= diameter of the specimen in mm
TA TB TC: temperature in disc A, B and C measured in °C.

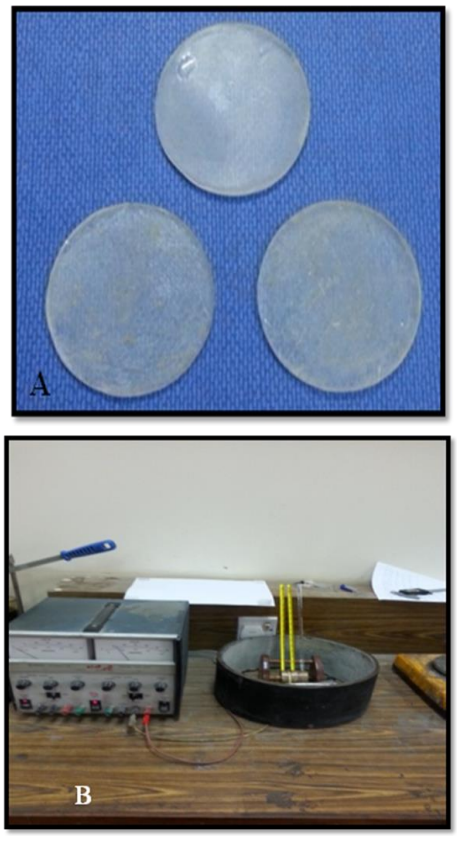


**Fig.5. Thermal conductivity: A, resin discs; B, testing device.**

**Shore D hardness:**

The specimens for this test were used with the dimensions (65×10×2. 5mm ± 0. 2mm). Surface hardness was assessed with durometer hardness tester (shore D)

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according to (ANS/ADA) No. 12, 1999) for acrylic resin material (figure-6). The device composed from blunt-pointed indenter 0. 8mm in diameter that tapers to a cylinder 1.6 mm. The readings were recorded from the digital scale of the instrument. Five recordings were registered on various regions of the resin specimen and an average of five recordings was calculated.


**Fig. 6. Durometer hardness tester (shore D).**

**Surface roughness:**

The rectangular acrylic specimens with the dimensions (65×10×2. 5mm ± 0. 2mm) length, width and thickness were prepared for the surface roughness test by using an analyzing surface roughness tester (TR220 portable roughness tester, Beijing, time high technology. Ltd, China).

The mean value for the four readings of the sensible needle on the surface of the tested material was dependent, figure-7.

**Fig.7. Surface roughness tester**.

**Water sorption:**

A Preformed stainless steel disc was prepared to obtain the acrylic samples in form of a disc (50mm±1mm in diameter and 0. 5mm ± 0. 05mm in thickness) according to ADA specification No, 12, 1999 [8], as shown in figure-8.

Seven specimens for the control group and seven for experimental group make a total of 14 specimens for measuring of water sorption. The resin specimens were dried in desiccators containing silica gel. The specimens were weighted with a digital balance. This process was repeated until a constant mass (M1) conditioned mass was obtained, then all discs involving the control and experimental groups were kept in distilled water for 7 days at 37°C ± 2°C. The discs were picked up from the water dried with a towel till they appear without moisture and weight one minute after removal from the water, then water sorption was measured according to the following formula:

WSP=M2-M1\S

(ADA specification No. 12, 1999)

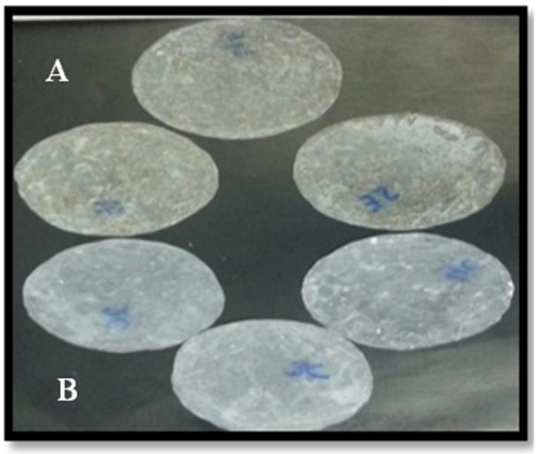


**Fig.8.A: Experimental acrylic resin specimens.**
**B: Control specimens for water sorption test**

Independent t- test was used to investigate the effect of adding silanized mixture of siwak and poly ethylene fibers on certain mechanical and physical properties of heat siwak and poly ethylene fibers on certain mechanical and physical proper-

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ties of heat cure acrylic resin, the signify-cant level was α=0. 05.

## Results:

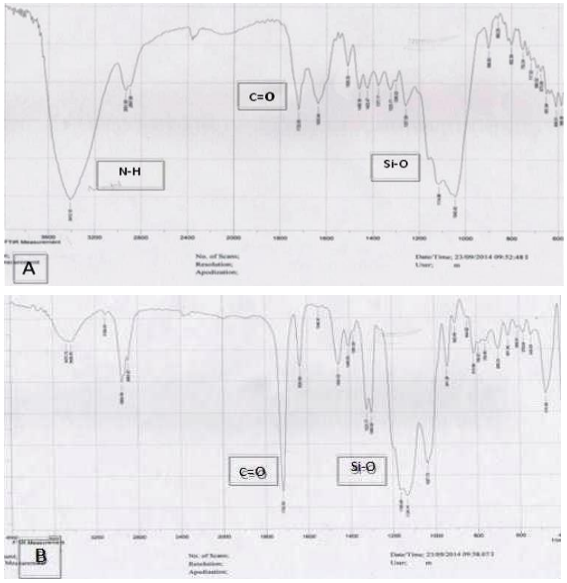


**Fig.9. FTIR spectra of: A. silanized siwak fibers; B. silanized poly propylene fibers**

**Spectroscopy:**

FTIR for silanized siwak and poly propylene fibers exhibited the charac-teristic stretching bands of the ester carbo-nyl C=o stretching peaks at 1720 cm-1 and Si-o peaks at 1114,1134 $cm^{-1}$, which represent a clear indication for silanization process, N-H peak refers to siwak fibers, figure 9 (A, B).

**Impact strength:**

Acrylic resin samples with silanized siwak and PP (poly propylene) fibers demonstrated significant increase in impact strength mean value than the control samples, table-2, figure-10.

**Transverse strength**

Acrylic resin specimens with fiber mixture demonstrate highly significant increase in transverse strength mean values in comparison to specimens with no fiber content, table-2, figure-11.

**Shear bond strength:**

Independent t- test exhibited that addition of 2% silanized siwak and PP fibers improve the shear bond strength between heat cure acrylic resin and the mollosil soft lining material significantly in comparison the control group, table-2, figure-12.

**Table-2: Descriptive statistics and independent t-test for impact strength (Kj\m$^2$), transverse strength (N\mm2) and shear bond strength (N\mm2).**

| Tests | Groups | N | Mean | SD | SE | t- value | df | Pvalue |
|---|---|---|---|---|---|---|---|---|
| impact strength | Control(A) | 7 | 8. 37 | 0. 899 | 0. 340 | -2. 398 | 12 | 0. 034 (S) |
| | Experimental(B) | 7 | 9. 27 | 0. 427 | 0. 161 | | | |
| Transverse strength | Control(A) | 7 | 108.6 | 17.410 | 6.580 | -4. 15 | 12 | 0. 001 (HS) |
| | Experimental(B) | 7 | 143.8 | 14.200 | 5.370 | | | |
| Shear bond strength | Control(A) | 7 | 0. 271 | 0. 025 | 0. 009 | -4. 053 | 12 | 0. 002 (HS) |
| | Experimental(B) | 7 | 0. 318 | 0. 007 | 0. 002 | | | |

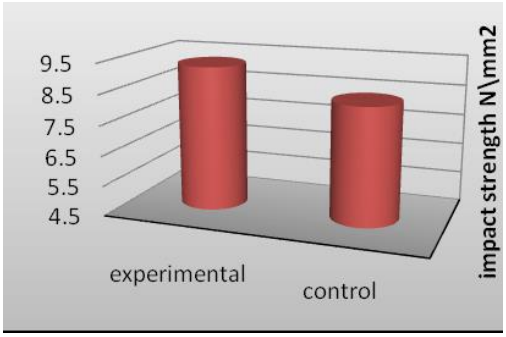


**Fig. 10: Bar chart of impact strength.**

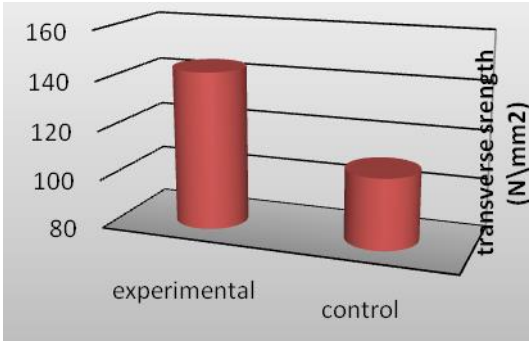


**Fig.11: Bar chart of transverse strength.**

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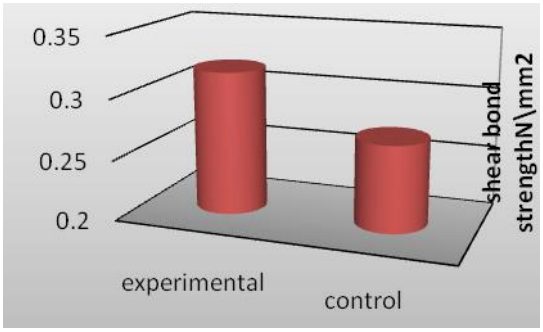


**Fig.12: Bar chart of shear bond strength**

**Thermal conductivity:**

Independent t-test revealed a significant increase in thermal conductivity with p- value 0.002 after incorporation of 2% silanized mixture of siwak and poly propylene fibers, table-3, figure-13.

**Shore D hardness:**

From table-3, the results of the test indicate a highly significant increase in surface hardness of heat cure acrylic resin after that addition of 2% silanized mixture of siwak and PP fibers, figure-14.

**Surface roughness:**

From table-3, the results of the test indicate anon significant decrease in surface roughness of heat cure acrylic resin after that addition of 2% silanized mixture of siwak and PP fibers, figure-15.

**Water sorption:**

Independent t-test showed anon significant reduction in water sorption mean value after addition of 2% silanized mixture of siwak and PP fibers, table-3, figure-16.

**Table-3: Descriptive statistics and independent t-test for thermal conductivity (W\m˚C), shore D hardness, surface roughness (µ m) and water sorption (mg\cm2)**

| Tests | Groups | N | Mean | SD | SE | t- value | df | sig |
|---|---|---|---|---|---|---|---|---|
| Thermal conductivity | **Control(A)** | **7** | **0. 222** | **0. 009** | **0. 003** | **-3. 851** | **12** | **0. 002 (HS)** |
| | **Experimental(B)** | **7** | **0. 243** | **0. 011** | **0. 004** | | | |
| Shore D hardness | **Control(A)** | **7** | **84. 21** | **1. 214** | **0. 459** | **-3. 484** | **12** | **0. 005 (HS)** |
| | **Experimental(B)** | **7** | **86. 11** | **0. 349** | **0. 132** | | | |
| Surface roughness | **Control(A)** | **7** | **0. 495** | **0. 096** | **0. 036** | **0. 535** | **12** | **0. 603 (NS)** |
| | **Experimental(B)** | **7** | **0. 468** | **0. 093** | **0. 035** | | | |
| Water sorption | **Control(A)** | **7** | **0. 308** | **0. 066** | **0. 025** | **1. 236** | **12** | **0. 240 (NS)** |
| | **Experimental(B)** | **7** | **0. 239** | **0. 131** | **0. 049** | | | |

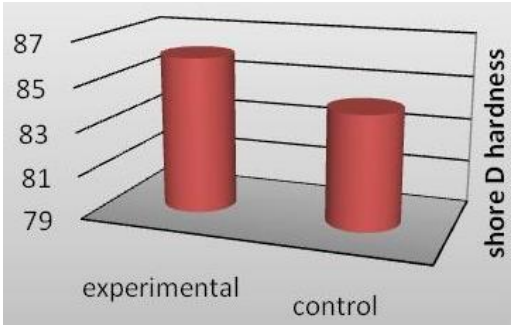


**Fig.13: Bar chart of thermal conductivity**

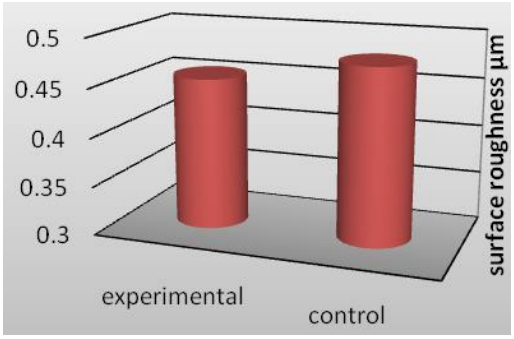


**Fig.15: Bar chart of surface roughness**

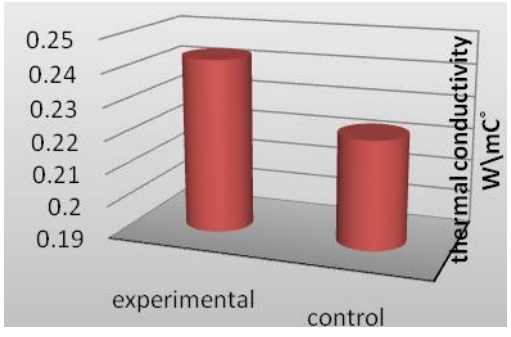


**Fig.14: Bar chart of shore D hardness.**

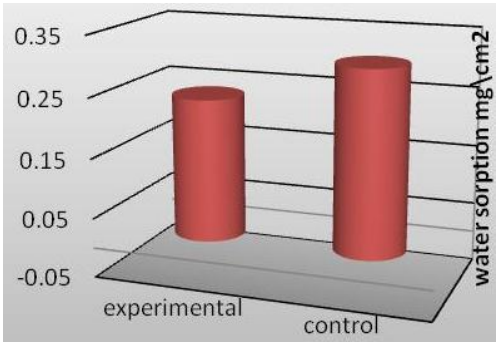


**Fig.16: Bar chart of water sorption.**

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## Discussion:

### Impact strength:

The reinforcement of acrylic resin specimens with fibers mixture demonstrates a significant increase in impact strength in comparison the control group, this finding could be explained that the reinforced fibers (Poly propylene fibers) are quite light in weight, have high abrasion resistance, resilient and not brittle, these fibers hold the load along their length and supply strength to the acrylic resin specimens [7].

The result findings exhibit coincidence with the results produced by Ladizesky et al, 1993 and Gutteridge, 1992, who proved that poly propylene fibers enhance the impact strength of acrylic resin [9, 10].

Another explanation for the improved impact strength of fiber resin composite is related to the action of silane agent which provides good chemical bonding of fibers to the resin matrix. The usage of silanized fibers in random order could stop the crack due to transferring action of stress from polymer to the fibers which might be attributed to the covalent bonding between the silanized fibers and the polymer chains [26].

### Transverse strength:

The experimental acrylic resin specimen showed highly significant increase in transverse strength mean values than the control group, this finding agree with Vojdani et al and Zbingniew et al [2, 5].

The transverse strength improvement could be attributed to the rough surface reinforced fibers produced by silanization process which provides better bonding between the fibers and the matrix of acrylic resin [6].

Several factors are responsible for increasing the strength of fiber reinforced resin including; the amount of fibers, the orientation of fibers and the bonding of fibers to the resin matrix [2, 6].

### Shear bond strength:

This study evaluate the bond strength of silicone soft liner to silanized fiber resin composite, the significant increase in bond strength could be related to surface modification of fibers incorporated into the resin and the type of acrylic resin used in this research.

The surface of acrylic resin specimen that is attached to the silicone soft liner is not polished so the silanized fibers that are randomly oriented within acrylic specimen become in contact with the adhesive containing polymeric substance dissolved in a solvent which accelerates bonding to the reactive groups of silane coupling agent. The non hydrolysable organo functional group of silane (methacrylate) can react with soft liner containing vinyl group and with adhesive containing polymeric groups (reactive groups)and by such mechanism the debonding of silicone lining material to fiber reinforced resin denture base is reduced [30].

### Thermal conductivity:

Thermal conductivity is an important property for the denture base material, several additives were added to resin denture base to improve this property.

Thermal conductivity mean values of fiber resin composite are higher than the control group (in significant increase) , this could be attributed to overlapping of the randomly oriented fiber mixture in some areas within the resin specimen that form pathways and facilitate transmission of heat therefore increase the thermal conductivity, another reason could be related to the presence of silanized fibers acting as thermal conductors as a result of cross linking that allows heat transmission through atoms in covalent bonds [34].

### Surface hardness:

Dental restorations with low hardness value will be affected by brushing and increased surface roughness with subsequent plaque accumulation and

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increased staining of the prosthesis therefore the hardness of the resin prosthesis should be adequate to prolong their shelf life.

The enhancement of hardness after addition of silanized fiber mixture in this study could be related to the random dispersion of fibers into the acrylic matrix and also the increase of hardness indicates that monomer to polymer conversion has been completed, because the hardness property affected by monomer which has plasticizing effect and decrease inter chain forces for this reason deformation can occur under force [31, 32].

**Surface roughness:**

The results of the study exhibited non significant reduction in mean values of surface roughness for the fiber reinforced resin in comparison to the control group ,this finding could be related to the smooth surface of silanized PP and siwak fibers after silanation process and also to good dispersion of silanated fibers in the polymer matrix. This finding disagree with Waltimo et al,1999, who found significant increase in surface roughness with glass fibers reinforcement.

Surface roughness of auto polymerized acrylic was significantly improved by the addition of poly vinyl pyrrolidone [36].

**Water sorption:**

It is mandatory to maintain the values of water sorption low for dental restorations because water molecules will produce certain stress strain areas and will adversely affect the mechanical properties and strength of the acrylic resin restoration[33].

Pole propylene fibers have hydrophobic nature and they exhibit reduction in water sorption and dimensional changes, the silanation process are not affecting the hydrophobic nature of poly propylene fibers.

The reduced water sorption of reinforced specimens could be related to the absence of micro-voids in between PMMA and silanized fibers due to the similarity in the chemical structure of PMMA and silane coupling agent. Another explanation for the deceased water sorption is attributed to good bonding and strong adhesion between the hydrophobic silanized fiber and the hydrophobic PMMA. Thus, water molecules could not enter at the filler-matrix interface. The presence of the –MPS formed a hydrophobic layer on the fiber surfaces, therefore the amount of water sorption was reduced [1, 33].

## Conclusions:

Within the limits of this research, the following conclusion was obtained:
Fiber resin composite (poly propylene and natural siwak fibers) has positive effect on the tested physical and mechanical properties of heat cure acrylic resin denture base material.

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