The effect of the addition of silanated poly propylene fiber to polymethylmethacrylate denture base material on some of its mechanical properties

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ABSTRACT

Background: Poly propylene fibers with and without silane treatment have been used to reinforce heat cure denture base acrylic but, some mechanical properties like transverse strength, impact strength, tensile strength, hardness, wear resistance and wettability. Which are related to the clinical use of the prosthesis are not evaluated yet. The aim of the study is to identify the influence of incorporation of treated and untreated fibers on these properties

Materials and methods: Eighty four heat cure acrylic specimens were constructed by conventional flasking technique. They were divided into six groups according to the tests and each group was subdivided into two subgroups control and experimental groups (seven samples for each subgroup).Transversestrength and Tensile strength test were performed using Instron universal testing machine. The impact strength test was evaluated by the use of Impact testing device. 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. Descriptive statistics and independent t test were used for statistical significance.

Results:The results revealed that the addition of Silanated polypropylene fibers produced significant difference in transverse strength and highly significant difference in tensile strength, the impact strength,Wettability value compared with the control group also the results showed that the hardness test was not significant and different control group . Wear resistance was highly significant decreased in experimental groups

Conclusion: Incorporation of silanated treated Poly propylene fibers to heat cure Poly methyl methacrylate resin was beneficial regarding the tested properties to improve the mechanical properties of the resin.

Key words: Poly propylene fibers, transverse strength, impact strength, tensile strength, hardness, wear resistance, wettability. (J Bagh Coll Dentistry 2015; 27(1):40-47).

INTRODUCTION

Developments in dentistry have largely been investigated as a result of scientific research. Of particular note, are developments in the field of dental materials and a drive towards the practical dentistry. Many aspects of Prosthodontic treatment; clinical or laboratory based, may impact on overall patient satisfaction and the clinical success of treatment [(1)]. Denture base materials, especially the resin based poly (methymethacrylate) (PMMA) materials are the most widely used non-metallic denture base materials [(2)].

They do, however, have a number of well documented problems. Since its introduction in 1937, poly (methyl methacrylate) (PMMA) has become the most commonly used material for denture bases. It remains most popular of all the polymeric denture base materials. This is largely due to its favourable, although not ideal, characteristics [(3)].

Since PMMA was introduced, most dental material research has been focused upon developing materials with higher strength, lower levels of residual methacrylate monomer after processing, improved dimensional stability, increased radiopacity and improved resistance to candidal infiltration [(4)].

There are three potential avenues for investigation which have been studied in the field of denture base materials. These are: the development of a new alternative to PMMA, the chemical alteration of PMMA; or the reinforcement of PMMA with another material such as fibres of with more favourable fracture resistance properties [(5,6)] . Acrylic resin dentures are susceptible to fracture during wear or if dropped, Fractures in dentures occur principally due to two different types of forces, flexural fatigue or impact [(5)]. Flexural fatigue results from repeated application of a force.

Numerous techniques for reinforcement of PMMA with inclusion of other materials have been described [(7,8)]. Carbon and Kevlar fibre reinforcement techniques have also been investigated. These were found to be not satisfactory aesthetically. However the complicated etching process required to improve their incorporation into PMMA, and preparation

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and positioning of the fibre layers, was found to be both time consuming and technique sensitive [9]. Such factors have thereby, reduced the routine applicability of this method. The use of glass fibres to reinforce PMMA is probably the most common technique described in the literature [9-11], not only have these been found to improve the mechanical properties of PMMA, but they are also highly publishable, aesthetic and easy to manipulate [5,11].

The addition of treated and untreated silica to PMMA was investigated by McNally *et al*.2006 Silica is commonly used as a filler to improve strength and wear characteristics of dental materials. The data collected in the study did not support the hypothesis that silica would improve the material's impact or transverse strength. Therefore, the authors were unable to recommend the inclusion of silica fillers in PMMA materials [12].

Notable efforts have been conducted to solve this problem and to strengthen the dental polymer by incorporating various types of fibers [10, 13, 39–43] and fillers [19,24, 44–46], but it has not been solved [13].

Interfacial Phase and Coupling Agents

For a composite to have effective clinical performance, a good bond must form between the inorganic filler particles and the organic resin matrix during setting. This phenomenon
is achieved through the use of coupling agents, such as silane [14]. This interfacial bonding is important to transfer load from the polymer matrix to the reinforcing fillers.

The most common types of coupling agents are organ of unctionalsilanes and organotitanates, which are used to improve filler dispersion in matrix, prevent aggregation, and reinforce the interfacial coherence with resin [15].

Silane-Coupling Agents

Silanes are commonly used in dentistry in different applications to provide the opportunity for chemical bonding.

The Silane can function as mediators between dispersed and organic phases. The most common silane, 3-methacryloyloxypropyltrimethoxysilane (MPS, or 3-MPS [15] ,which chemically bond the silica, present in silica-based fillers such as porcelain, quartz, pyrogenic silicon dioxide, and silicate glasses because of the similarity in their ordered structure [16], to the organic matrix of resin by means of siloxane bonds and hydrogen bond [17].

Most recent investigations give increasing prominence to transverse strength, impact strength, tensile strength and hardness as these properties give an idea about the resistance of the material during use, in order to develop strong denture base material [17].

Wear resulted from removal and relocation of materials via the contact of two or more materials which cause the material to be loss. Wear resistance is deemed as one of the characteristics that be developed by composite [18].

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 [18].

In this study the attempt has been done to incorporate silanated polypropylene fibers to reinforce polymethyl methacrylate denture base material and examine the effect of this addition to some mechanical properties, which are: transverse strength, impact strength, tensile strength, hardness, wear resistance and wettability which are related to the clinical use of the prosthesis.

MATERIALS AND METHODS

Eighty four acrylic specimens were constructed from heat cure acrylic resin by conventional technique for flasking and curing using heat cure acrylic resin. They were divided into six groups according to the tests and each group was subdivided into two subgroups (control and experimental groups, seven samples for each subgroup).

The required amount of the powder of the polymer and polypropylene fibers was used by weighting using digital electronic balance 2.5 %(by weight 6mm length) poly propylene fibers (PP fibers) (Cracecemfiber®) as more recent study showed an incorporation of 2.5% percentage had the best effect regarding impact and transverse strengths as that considered to be important in measuring the strength of materials [19].

Polypropelene fibers were treated with 3-(methacryloyloxy)propyltrimethoxysilane (MPS) to improve the bonding between pp fibers and PMMA matrix to get PMMA/pp fibers composite with improved properties over pure PMMA .

One hundred milliliter of ethanol aqueous solution (70 vol%) was prepared using 99.8 vol% ethanol and deionized water, and adjusted to pH of 4.5 by titrating with 99.9% acetic acid using a pH meter. Then, 5wt%, g of MPS was added respectively into ethanol aqueous solution, and stirred. This MPS solution was stored in a 100 mL polyethylene cup with a cover, and allowed 5 min for hydrolysis and silanol formation. Then, 100 g pp fibers were added into MPS solution. The mixture was stirred with magnetic stirrer for 30

minutes, then the mixture was sonicated with probsonication apparatus for 30 minutes, then the solution left dried at room temperature for 14 days [20].

The (FTIR) spectrophotometer where done to determine whether or not functional group of the MPS have been attached to pp fibres by analyzing characteristic vibrations of functional groups [21]. When there was change in the vibration or absorption of functional group indicated to be used to complete the study,change which has been occurred indicated by the FTIR spectra as shown in figure 1and 2.

After silanization of pp fibers, the selection of the percentage of the fiber has been done. 2.5% were used as percentages of fiber to be added to the PMMA according to previous study [19], mixing of polymer powder and fibers was done by using mortar and Pestle to attained homogenous mixture for about 3.0 minutes, All the specimens for all the six tests were finished and polished. the addition of silanated pp fiber to the PMMA was done only to the experimental groups

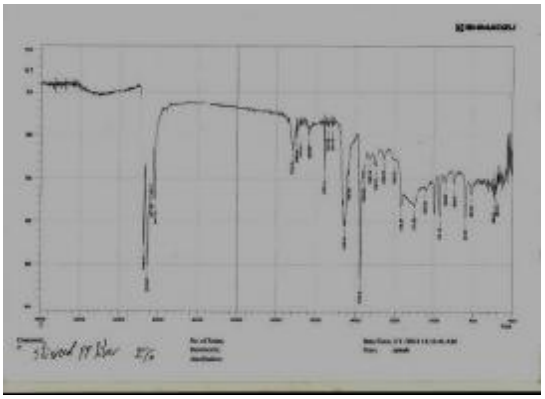

Fig. 1: FTIR of polypropylene fiber without silanation

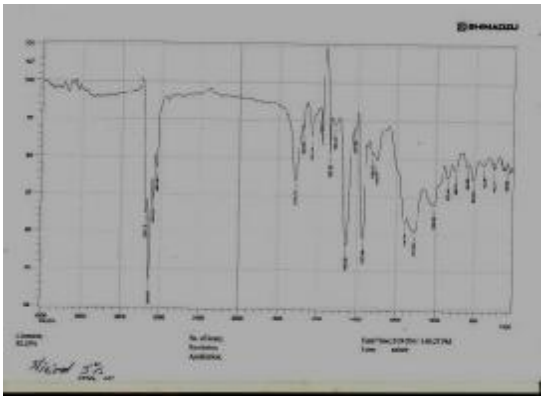

Fig. 2: FTIR of silanated polypropylene fiber

The tests used in this study were:

A- Transverse strength test

Specimens were prepared with dimensions (65mm x 10mm x 2.5 + 0.1mm). All the specimens were stored in distilled water at 37 0°C for 48 hours before performing the test [22]. The test was performed using Instron universal testing machine (WDW-200 E), each specimen was positioned on the bending fixture which consist of two parallel supports (50 mm apart), the full scale was 50 Kilo and the load was applied with across head speed of1mm/min until fracture occurs.

B- Impact strength test

Specimens have dimension of (80mm x 10mm x 4mm) (ISO 179, 2000) for unnotched specimens. Specimens were stored in distilled water at 370C for 48 hours before being tested [22].

The impact strength test was evaluated following the procedure recommended by the ISO 179 with Impact testing device. By supporting the specimen 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 =E/bd $X10^3$ (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

C- Tensile strength test

Dumbbell shaped specimens were fabricated according to ASTM specification D-638M(1986).Conditioning of the specimen was done by storing them in distilled water for two days at room temperature25ºC±2before testing according to ADA specifications. The testing procedure was accomplished using Instron universal testing machine (LARYEE, make test easy, china) with crosshead speed 1 mm/min and maximum loading of 20 Kg. Tensile strength calculations was performed are automatically done by the program of the testing machine software.

D-Surface hardness test

Specimens were prepared with a dimension of (65mm x 10mm x 2.5 + 0.1mm). All specimens were stored in distilled water at 370C for 48 hours before being tested [22].

(Shore D) durometer hardness tester Surface hardness were used for measuring the hardness which is suitable for acrylic resin material. It consists of spring – loaded indenter (0.8mm in

diameter), that attached to digital scale scored each specimen (one in the center and other at each end) then the mean of three readings was calculated.

E- Wear rate test

Special device was used to test the wear that was designed at University of Technology Material, engineering department, resistance laboratory-Iraq. called Pin on disk wear testing device ,it was recognized with high accuracy of results [23,24], The specimens were cylindrical in shape with dimensions of 20mm length and 10mm diameter according to the device requirement(figure 3). It consisted of pin which was the held specimen and disk made from stainless steel wheel which revolve 950 r/min, the specimen was weighed before and after the testing procedure, after that the specimen was secured to the holder and was put 10N load on the horizontal arm, the device switched on for 10 minutes (wear testing time)[25]. The distance between the center of the disk and center of the specimen was 65mm. the following equation to was used determine the wear resistance: wear resistance (grm/mm) =change in weight/slide distance (slide distance=2π×radius distance between centers of disk and specimen×number of cycles ×time of test)[25]. Cleaning of the disk must be done after each test All specimens were immersed in distilled water for 48 hours before testing.as shown in figure 3.

F- Wettability

Measurement of the contact angle was necessary 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 10µL at the center of the fabricated sample using pipette. The sample dimensions was 8×30×2mm width, length and thickness respectively [26].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× supplied with camera (high resolution 1024×768 pixel) and software (Dino capture2.0 Version 1.3.3.) granted a complete and precision analysis to the contact angle[27] (Figure 4).

Statistical analysis

The data collected of the tested specimens were translated to computerized statistical from 0 to 100 units. Three readings were done on analysis system by using (SPSS) statistical package for social sciences version 20. Descriptive statistics and independent t test where used for statistical significance.

Fig. 3: Pin on disk with the specimen

Fig. 4: Digital microscope with specimen.

RESULTS

Characterization of silanized pp fiber

The absorption bands of MPS can be assigned to the presence of the functional groups, which are two prominent peaks at 2945cm^{-1} and 2841cm^{-1} which can be attributed to the (C-H) stretching, and the characteristic (C=O) stretching occurs at 1720cm^{-1}, and the characteristic for (C=C) stretching occurs at 1637cm^{-1} for (CHR2R-andCHR3R) occurs at 1413cm-1,and groups of peaks between 1296cm^{-1} and 1166cm^{-1}can be attributed (C-O-C) stretching, the characteristic (Si-O-CHR3R) stretching occurs between (400 - 470)cm^{-1}figure (1,2) .

The descriptive statistics which include Means, Standard deviations, standard error of mean value, of the tests which are conducted to evaluate the effect of silanizedpp fibers addition on some properties of PMMA are shown in table 1.

By using independent samples t-test, the results revealed statistically significant difference between flexural strength of fiber reinforced PMMA and the control resin group.

Table 1: statistical analysis between control and experimental groups for all the tests

Tests performed	Studied groups	Descriptive statistics				Comparison		
		N	Mean	S.D.	S.E.	t-test	df	P-value
Transverse strength	Control	7	86.9187	7.2264	2.7313	2.632	12	.022
	Silanated pp fiber 2.5%	7	76.5621	7.4945	2.8327			
Impact strength	Control	7	12.2601	1.0234	.3868	-6.352	12	.000
	Silanatedpp fiber 2.5%	7	23.1549	4.4210	1.6710			
Tensile strength	Control	7	54.2143	1.5126	.5717	-3.421	12	.005
	Silanatedpp fiber 2.5%	7	57.1286	1.6710	.6316			
Hardness	Control	7	83.0000	.9018	.3409	.499	12	.627
	silanated pp fiber 2.5%	7	82.7143	1.2158	.4595			
Wear rate	Control	7	.000012	.000001	.000000	3.795	12	.003
	silanated pp fiber 2.5%	7	.000010	.000001	.000000			
Wettability	Control	7	45.8739	1.6865	.6374	21.015	12	.000
	silanated pp fiber 2.5%	7	28.2646	1.4391	.5439			

The addition of 2.5% silanized pp fiber lead to decrease in the mean value of transverse strength from (86.9187 MPa)to (76.5621 MPa) and when applying t. test the results revealed a significance difference P= 0.022 as shown in table 1 and the mean in figure 5.

The impact strength test results showed a highly significant difference, between the control which has been found (12.2601 Kj/m^2) mean value, with the silanated pp fiber group which had (23.1549 Kj/m2) mean value,p 0.000.as seen in table 1.fig 6,7.

The tensile test revealed highly significant difference between the control (54.2143 Mpa) and the silanated pp fiber group (57.1286 Mpa), p= .005,as shown in (table 1. and figure 7.)

No significant difference was found in the hardness test, decreased wear rate from (.000012 to 0.000010 gm/mm) and decreased wettability after addition of ppfiber 2.5 %, as shown in Table 1 and figure 8.

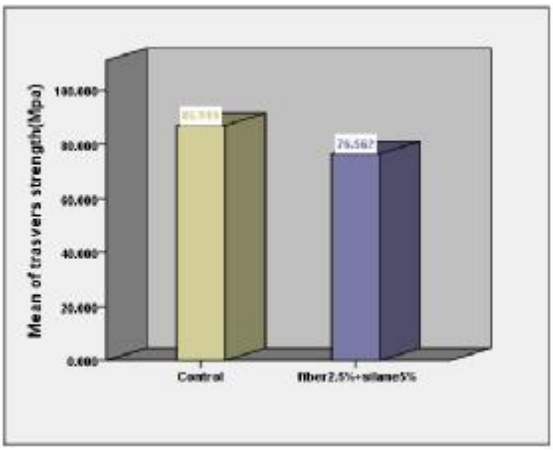

Fig. 5: Bar chart for the mean value of transverse strength of the control and ppf groups reinforced PMMA

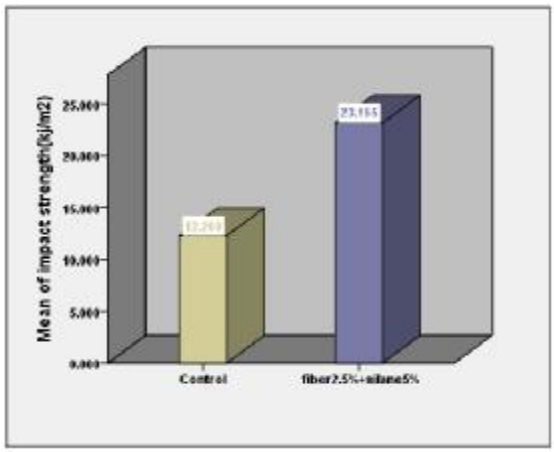

Fig.6: Graph showing mean impact strength of control and ppfiber-reinforced polymethyl methacrylate

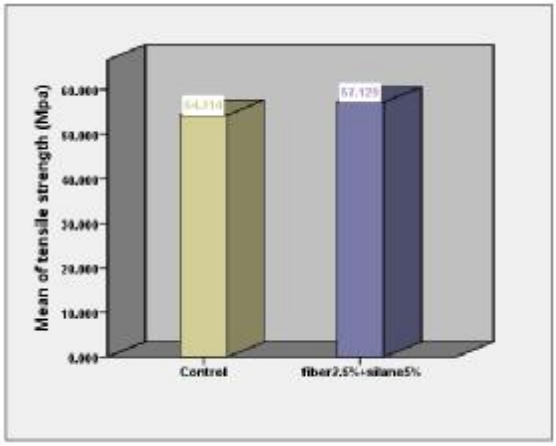

Fig. 7: Graph showing means tensile strength of control and fiber-reinforced PMMA

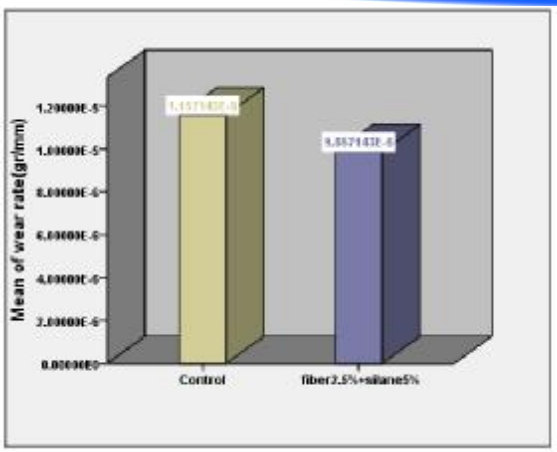

Fig. 8: Graph showing mean wear rate test of control and pp fiber-reinforced polymethyl methacrylate

DISCUSSION

The use of fibers to reinforce denture base material has an acceptable success rate mainly because of the advancements in the fiber-reinforcing materials. Reinforcement with fibers enhances the mechanical strength characteristics such as transverse strength, ultimate tensile strength, and impact strength [9].

In this study, using a type of olefin fibers named polypropylene (pp) as reinforcing filler to PMMA denture base resin has many properties like high strength, good surface finish and polish, low cost and excellent biocompatibility [28].

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 [29], surface chemistry and topography may be influenced to result in improved adhesion between the polymer matrix and fibers used. plasma treated polypropylene fiber had been used [19] , which results in improvement in the mechanical properties, but in this study the attempt has been done to use chemical surface treatment for the fibers, by treated with 3-(methacryloyloxy) propyltrimethoxysilane (MPS) to improve bonding between pp and PMMA matrix.

The results revealed that the addition of Silanated polypropylene fibers produced significant difference decrease 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. Also this may be 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 silane 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 and affect strength, these results were with no agreement with result of Waffae[19] who found increase in transverse strength after addition of plasma treated pp fiber but was not significant.

Highly significant increase in impact strength mean value (23.1549 Kj/m^2)compared with control group, this increase which could be related to the presence of silanated pp fibers which prevent the crack propagation and change in direction of cracks resulting in smaller flows between the fibers, These results are in agreement with results obtained by Waffae,2013 and Mowade et al 2012., this increase could be attributed to the fact that silane 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 therefore, enhance the impact strength.

The results also revealed highly significant difference in tensile test, for the 2.5 wt% silanated pp fibers group, these are unlike the study done by Alalwan [27] who used 2.5 wt% pp fibers plasma treated and found negative effect of plasma treatment to PP on the tensile strength, it could be inferred to the water absorption of PP fibers which was improved by plasma treatment, while silanated pp fiber may show reduced water absorption. Because the functional group which

cause stretching not include polar group-OH, many, studies reported that incorporation of oxygen plasma treated poly propylene fibers to PMMA had increased the water absorption significantly [19] and PMMA tensile strength is negatively affected by water absorption and ambient moisture, this is in agreement with Ishiyama et al [30], in addition to the acrylic used in this study was of cross linked type that might exaggerate the water absorption [31], in addition to that the orientation of the fibers and the bonding with the matrix of PMMA affect the mechanical properties. Plasma treatment for fibers can lessen the tensile strength of the fibers themselves due to their loss of weight and diameter reduction [32], this is not present with silanation,in contrast , there is addition of layer to the fibers. In this study there was slight decrease in the hardness number which statistically not significant, this decrease could be related to no presence of these fibers near or at the surface of the composite which extremely hard and stiff [33,34] .

Highly significant (p=0.003) decrease in the wear rate could be attributed to that high abrasion resistance of PP fiber[35] ;however, after silane 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 cross linking that decreased plastic flow which in turn increased the wear resistance and that was consistent with Kyomoto et al2007 and Drobny2013[36,37].

Wettability of acrylic reinforced with silanated treated PP fiber was highly significant, regarding the treated PP, it might be pp fibers used are insoluble in water and could reduce the overall volume of the absorbing polymer [38]. The second factor could be the silane coupling agent which was used to silanization of pp fibers The silane coupling agent had similar chemical nature to polymer matrix (methacrylate at one side). Thus, the pp fibers-resin interface provided by silane coupling agent could lead to a reduction in the amount of water that reached to the inner layers of the polymer matrix.This is in agreement with several researchers [39,40], that overwhelmed on PMMA-PP fiber composite wettability and occurrence of new functional groups as noticed guiding the change in a surface layer structure.

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الخلاصة

الخلفية:حديثا الياف البولي بروبيلين مع و بدون معالجة بالسيلان قد استخدمت في تقوية الراتنج الاكريلي الحراري لقاعدة الطقم لكن لحد الان لم يختبر تاثيرها على قوة الشد وقابلية الترطب ومقاومة البلى.

المواد و الاساليب:اربع وثمانونعينه استخدمت في البحث بلمرت بطريقة البلمرة الحرارية ومقسمة على ستة مجاميع ولكل مجموعة رئيسة تحوي مجموعتان فرعية سبع عينات لكل منها أجريت اختباز قوة الشد واختبار القوة االمستعرضةباستخدام شهاز الانسترون، تم تقييم مقاومة البلى عن طريق استخدام جهاز اختبار بطريقة المسمار على القرصو قياس الوقت،استخدم جهاز الديبورامیتر لقياس قوة الصلادة واستخدمت كاميرارقميةعالية الدقة لقياس زاوية الاتصال مع السطح لقياس قابلية صفة الترطيب .

النتائج:اظهرت النتائج تباينا واضحا عند فحص قوة الشدواختبار القوة االمستعرضةوقوة الصلادةو قابلية صفة الترطيبلصالح اضافة الياف البولي بروبيلين المعالجة بالسيلان بينما بينت تاثير ايجابي للالياف المعالجة في ما يخص صفة مقاومة البلى ولا يوجد فرق فيما يخص صلابة السطح .

الاستنتاج:مع محدودية البحث يستنتج بأن اضافة الياف البولي بروبيلين المعالجة اثر ايجابيا بالصفات المدروسة .