

# Preparation and Investigation of some Properties of Acrylic Resin Reinforced with Siwak Fiber Used for Denture Base Applications

**Sihama I. Salih**

Material engineering department  
University of technology  
Baghdad, Iraq  
130005@uotechnology.edu.iq.

**Jawad K. Oleiwi**

Material engineering department  
University of technology  
Baghdad, Iraq  
130041@uotechnology.edu.iq

**Hwazen S. Fadhil**

Material engineering department  
University of technology  
Baghdad, Iraq  
130143@uotechnology.edu.iq

**Abstract:** *Natural fibers have recently become attractive to researchers due to their low cost, fairly good mechanical properties, high specific strength, non-abrasive, eco-friendly, bio-degradability and biocompatibility characteristics, they are exploited as a replacement for the conventional fibers, such as glass, aramid and carbon. This study investigated the influence of fiber length and weight fraction of natural siwak fiber, with the selected length of (2, 6 and 12 mm) and weight fraction (3, 6 and 9 wt %), on some of mechanical and physical properties such as (tensile, impact and hardness), in addition to test the infrared spectroscopy FTIR of the prepared denture base resin and all of these tests were carried out at laboratory temperature.*

*The properties of PMMA reinforced by natural fiber are mainly affected by the interfacial adhesion strength between the matrix and the fiber, and in order to improve interconnection between the siwak fiber and PMMA matrix, so the siwak fibers were treated with alkali (sodium hydroxide) solution prior to use as reinforcement materials. The results illustrated that the tensile strength, young modulus, fracture toughness and hardness tended to be improved with length and concentration ratios of siwak fiber, while the impact strength and elongation percentage at break decrease with fiber content in composite samples.*

**Keywords:** *Acrylic Resin, Natural fibers, Siwak, Denture.*

## 1. INTRODUCTION

Tooth loss is one of the manifestations of aging. Dentures and dental implants are the major prosthetic devices given to restore physiological and esthetic functions of oral tissues of patients. An ideal denture base material is the one that possesses biocompatibility with the oral tissues, excellent esthetics, and superior mechanical properties especially modulus of elasticity, impact strength, hardness, sufficient bond strength with artificial teeth and lining materials. Dentures made from resin based polymeric systems were popular because of their ability to be molded easy with excellent esthetic appearance and suitable mechanical characteristics in most clinical condition and low cost compared with metal-base denture. Among the polymers, (PMMA) is the most commonly used material for this purpose. One

of the main drawbacks of this material is considered to be its poor mechanical performance [1]. Fracture of acrylic resin removable dentures occurs frequently during service through heavy occlusal force or accidental damage [2]. Therefore, improvements in the mechanical performances of denture base structures have been sought by adding reinforcing compounds to the PMMA matrix, thus creating a reinforced denture base resins [3].

**The aim of this study is to prepare PMMA composites reinforced by the natural siwak fiber, with the different lengths and different weight fraction, and their characterization study.**

## 2. METHODS AND MATERIALS

In this research the DURACRYL PLUS self-curing base resin, manufactured by Spofa Dental Company used as a matrix. This type of material characterized by many properties compared with other kind of PMMA polymer, such as: softer feel, low molecular weight, color stable in the long run, minimized shrinkage, stable polymerization cycle with a perfect end result, the acrylic is long pourable and modulate for a long period of time [4]. Siwak fibers in three different length (2, 6 & 12 mm) and three concentration (3, 6 & 9 wt. %) used as reinforcing materials in acrylic resin. Figure (1) shows the methyl methacrylate powder and monomer and the siwak fiber that are used to fabricate composite specimens in this study.

### Methods

According to required selection ratio of weight fractions of the reinforcement materials weighing the amount of reinforced material (siwak fibers) by using electronic balance with accuracy (0.0001) digits depending on total weight of the matrix material PMMA required for filling the mould cavities by using theory of rule of mixtures. The fibers treated with 5% (w/v) alkali (sodium hydroxide) solution at 25 °C for 24 h, maintaining a fiber-to-liquor ratio of 1:30 (w/v). The fibers which alkali treated washed several times with distilled water to remove excess alkali sticking on their surface, then neutralized (PH-7) with distilled water containing a few drop of acetic acid and finally washed with distilled water, then the treated fibers were dried at room temperature for 5 days and finally kept in hot air oven at (50-60 °C) until dry. The liquid monomer (MMA) with

one type of reinforcement fibers (siwak) should be mixed together at room temperature homogeneous and continuously, so, it must be sure of homogeneity of the mixture, before added powder to it to produce composite materials. The powder then added to the mixture and gradually mixed, then cured in same manner which indicated above.

The acrylic resin samples were de-molded to remove from the metallic mould cavities with smooth surfaces, followed by heat treatment at 55°C for 3hr to remove residual stresses as a result of de-molding of the specimens from the metallic mold cavity.

### 3. RESULTS

#### Fourier transforms infrared spectroscopy (FTIR)

Fourier transforms infrared spectroscopy (FTIR) was used, to fully characteristic band of PMMA composite specimens. Figure (2) illustrated the infrared spectrum of neat PMMA and shows that the asymmetric and symmetric correspond to the (C–H) stretching of methyl group (CH<sub>3</sub>) assigned to peaks at 2990.53 and 2853.28 cm<sup>-1</sup> respectively. Furthermore C=O and C-O bands appear at range (1500-2000 cm<sup>-1</sup>) and (1000-1400) respectively [5]. So, the medium strength of C=O stretching assigned at 1722 cm<sup>-1</sup> and the bands at (1447.03 and 1385) cm<sup>-1</sup> are associated with (C–H) symmetric and asymmetric stretching modes respectively. The 1239.54 cm<sup>-1</sup> band is assigned to torsion of the methylene group CH<sub>2</sub> and the peak at 1189.98cm<sup>-1</sup> for the band corresponds to vibration of the ester group (C–O), while (C–C) stretching band are at (984.77 and 840.98) cm<sup>-1</sup> [5 and 6]. In this spectrum medium strength of C=O bending assigned at 748.98 cm<sup>-1</sup>. The infrared spectrum of PMMA composites reinforced with different ratios of siwak fiber (0, 3, 6 and 9%) for different lengths (2, 6 and 12mm) is shown in figures (3, 4 and 5). It can be seen from the infrared spectrum of these group composite specimens, no other new peak or peak shifts were observed for the PMMA composite specimens with the addition of siwak fiber. This is due to the find physical bond and absence of any cross linking in these specimens. Furthermore, there is a clear increase in peaks intensity for all of characteristic peaks of PMMA with increasing siwak fiber ratio, and it reaches a maximum at ratio of 3% siwak fiber. Then the peaks intensity of PMMA composites decrease to lower than the peaks intensity of neat PMMA, when the ratio of siwak fiber increases to higher than 3% ratio.

From FTIR test we can observed that there is no other peak or peak shift of FTIR Spectra for PMMA composite specimens with addition of siwak fibers, also increase in ratio of fibers led to increase in peak intensity and reach maximum at 3%, then peaks intensity of PMMA composites decrease to lower than the peaks intensity of neat PMMA, when the ratio of fibers increases to higher than 3% ratio.

#### Tensile test results

The composite specimens with siwak fiber have the highest tensile strength than the neat acrylic (PMMA). This is due to the presence of fibers in resin thus ensuring transmission of loads from matrix to fiber. Mostly PMMA matrix is much weaker in strength than samples with strengthening fibers, since the matrix alone is unable to resist the tensile force applied on it and fails with strength lower than specimens with strengthening fibers that withstand the tensile load [7]. The relationship between the tensile strength and weight fraction of siwak fibers is shown in figure (6). Figure shows the increasing in the mechanical properties with the fiber length. However, the optimal condition is at a fiber length of 12 mm. The fiber length of (2, 6 mm) has a lower tensile strength and the reason behind this is that shorter fiber may not be compatible composites due to the improper bonding between the matrix and fibers [8]. It can be noted that the tensile strength increases with increasing weight fraction of reinforcing fibers. The reasons behind such behavior is that the strengthening mechanism of reinforcing fibers in which, the amount of these fibers plays an important role impedes increasing the slipping of PMMA resin chains.

Figure (7) shows the relation between modulus of elasticity of PMMA composites as a function of weight fraction and fiber length of siwak fibers in composite. It can be noted that the modulus of elasticity increases with increasing fiber length and weight fraction in composite samples. So, the weight fraction of (9 wt.%) and (12 mm) fiber length represent the greatest value for the modulus of elasticity for PMMA reinforced with siwak fibers. The reason behind increasing young's modulus value with increasing content of fibers, may be related to good interfacial adhesion strength between the matrix (t will be explained later by SEM morphology), in addition to that the siwak fibers have good stiffness than acrylic resin because they have young modulus higher than matrix and that leads to improving the stiffness of the composite. Also as the fiber weight fraction increased, there is possibility of fiber-matrix interaction leads to an increase in efficiency of stress transfer from the matrix phase to the fiber phase.

The percentage of elongation determined at fracture for PMMA composites as a function of weight fraction and fiber length of siwak fibers in composite, and is shown in figure (8). Increasing the fiber length and weight fraction of siwak fibers leads to reduce the percentage of elongation of composite specimens. This is because the presence of reinforcement imparts the stiffening effect within the resin and thus imposes a mechanical restraint on the composite. Also decrease of elongation percentage depend on the interface that is between fiber and matrix [9 &10].

From tensile test we observed that pure PMMA had the lower value of tensile strength and modulus of elasticity which equal (47.6 MPa &1.7 GPa.), respectively. Tensile strength and young modulus, increase with the increase in the weight fraction and fiber length of the siwak fibers in PMMA resin. Elongation percentage at

break decrease with increasing the weight fraction of siwak fibers.

The largest values of tensile strength and young's modulus for specimens reinforced with siwak fibers are (71 MPa. & 4.9GPa.) at optimum condition of weight fraction (9%) and fiber length (12 mm).

### Hardness test results

A figure (9) shows the relationship between the hardness and the weight fraction of the reinforcing fibers (siwak), as a function to fiber length, which were added to the Poly Methyl Methacrylate resin. Figure illustrate that, the hardness increases with increasing weight fraction and fiber length of siwak fibers, and reaches its maximum amount at (9 wt.%) and 12mm length. This is because of the increase of fiber content, the composite becomes stiffer and harder, as compared to the matrix polymer. The fiber has superior mechanical properties such has hardness, modulus, strength etc. Therefore the additional high modulus fiber increases the hardness of the composite [11]. Also the increase in hardness may be attributed to increasing wettability or bonding (interaction) between the matrix and fibers due to alkali treatment of fibers [12]. Also, the results for specimens increase with increasing fiber length of fibers.

Hardness increase with the increase in the weight fraction and fiber length of the siwak fibers in PMMA resin.

### Impact test results

A figure (10) shows the relationship between the impact strength and the weight fraction as well as, the fiber length of the reinforcement (siwak fiber), which were added to PMMA resin. Figures illustrate that impact strength for neat PMMA resin is equal to (8.75 kJ/m<sup>2</sup>), and higher than a composite specimen with reinforcing fibers. The impact strength decreases with increasing weight fraction and fiber length of reinforcing fibers of composite materials. This is because the presence of reinforcement such as siwak fibers it will increased the stiffness of PMMA composite (as mentioned earlier) and thus imposed restraint the movement of polymeric chain, and this lead to decreased in impact strength of the composite samples with increased the fiber length and fiber content in composite. Furthermore, due to the stress concentration sites in the composite samples, which result from clustered fibers especially with the high ratios of fiber content in composite and that may be reduction in bonding between fiber and PMMA resin [13].

It can be also noted from this figure that specimen which have higher length about (12mm) have higher impact strength than specimen with a lower length.

The results of fracture toughness for samples with siwak fibers in PMMA resin is illustrated in figure (11). Which, illustrates that the fracture toughness increase with increasing weight fraction and fiber length for composite specimens reinforced by siwak fibers. The maximum values of fracture toughness were observed at (12mm) fiber length and (9%) weight fraction and reach (6.03 MPa.m<sup>1/2</sup>) for siwak specimens.

Impact strength decrease with increasing the weight fraction of siwak fibers. Fracture toughness increase with the increase in the weight fraction and fiber length of the siwak fibers in PMMA resin.

Pure PMMA have impact strength value higher than composite specimens reinforced by siwak fibers and equal to (8.75 KJ/m<sup>2</sup>).

### Morphological analysis

Scanning electron microscopy SEM micrographs were done to the tensile tested fractured surface for the neat PMMA polymeric material and PMMA composite samples with different composition at different magnification (1000x and 3000x). The fractured surface morphology of the neat PMMA sample, as shown from figure (12 a and b), in the most case, it appeared as a homogeneous morphology.

SEM photography of the fracture surface morphology of PMMA composites reinforced by 3% ratio of siwak fiber with 2mm fiber length, and PMMA composites reinforced by 9% ratio of siwak fiber with 12mm fiber length are shown in figure (13 a and b) and figure (14 a and b) respectively at different magnifications (a:1000X and b: 3000X). It is noticed from the fracture surface morphology of SEM photography. This microscopic imaging exhibited a heterogeneous morphology. As well as, figure (14 a) shows a continuous long fiber morphology embedded in the matrix material and these fibers remained coated with a matrix material, even after the failure which occurred in the sample, as designated by the red coloured arrows in figure (14 a).

The fracture surface morphology of neat PMMA appeared homogeneous morphology in (SEM) test, while the PMMA composite reinforced by siwak fibers the fracture surface appeared sem-continuous.

## 4. Figures and Tables

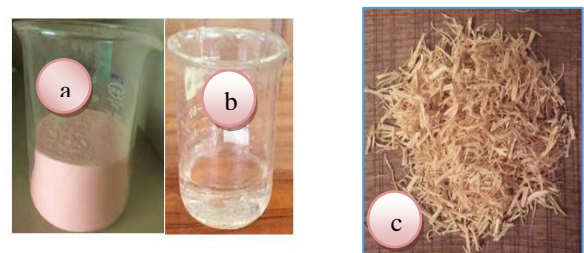


Figure 1: where (a): (PMMA) powder, (b): monomer and (c): siwak fibers.

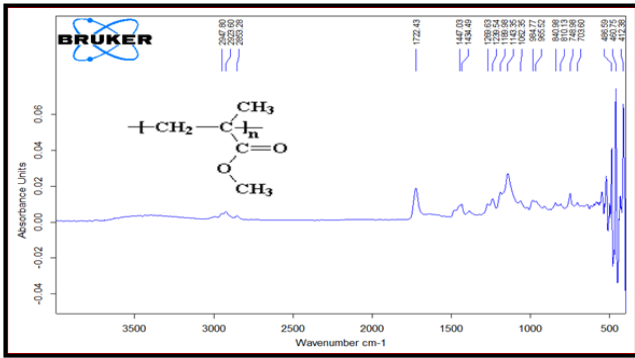


Figure 2: FTIR Spectrum that Obtained for the Cold Cure PMMA Specimens.

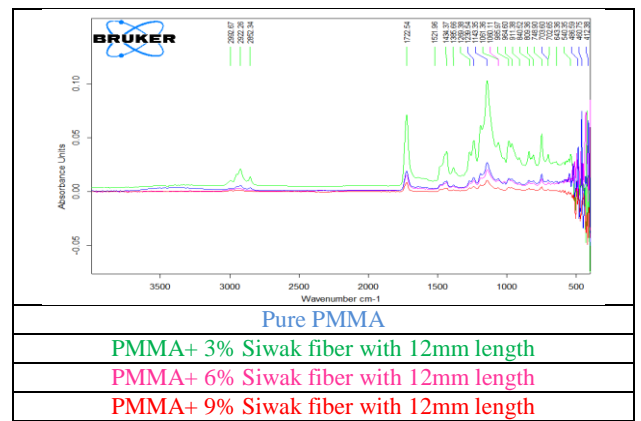


Figure 5: FTIR Spectra for PMMA Composites as a Function of Siwak Fiber Content in Composite Samples at Constant Length (12mm) of Fiber.

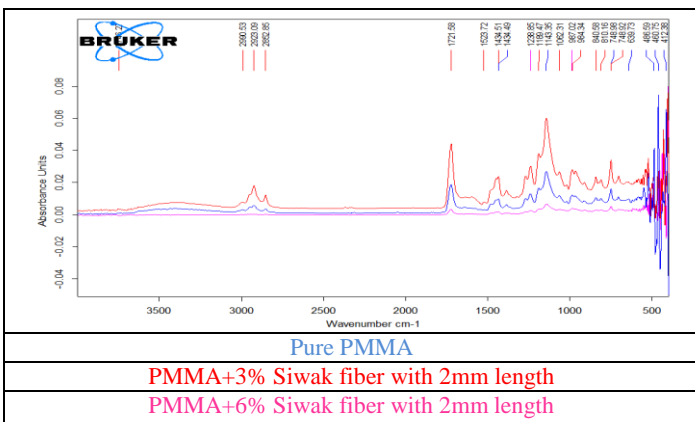


Figure 3: FTIR Spectra for PMMA Composites as a Function of Siwak Fiber Content in Composite Samples at Constant Length (2mm) of Fiber.

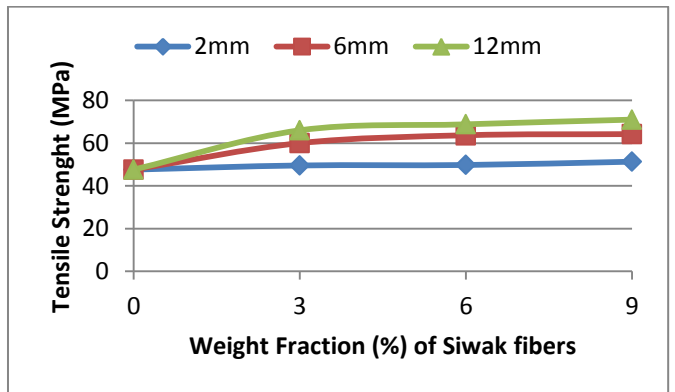


Figure 6: Tensile Strength of PMMA composites as a function of weight fraction and fiber length of siwak fibers in composite.

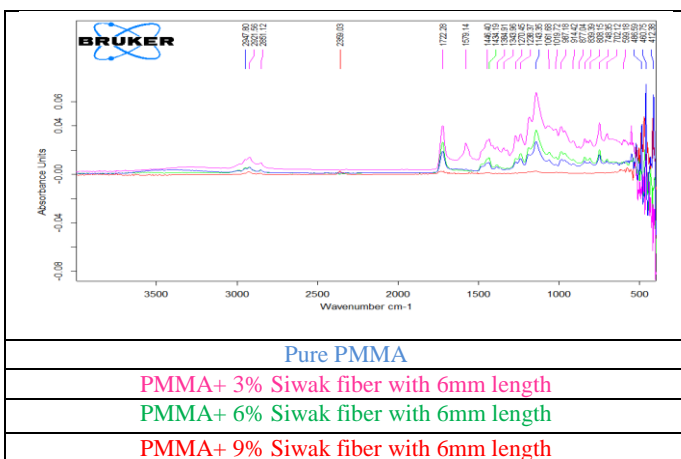


Figure 4: FTIR Spectra for PMMA Composites as a Function of Siwak Fiber Content in Composite Samples at Constant Length (6mm) of Fiber.

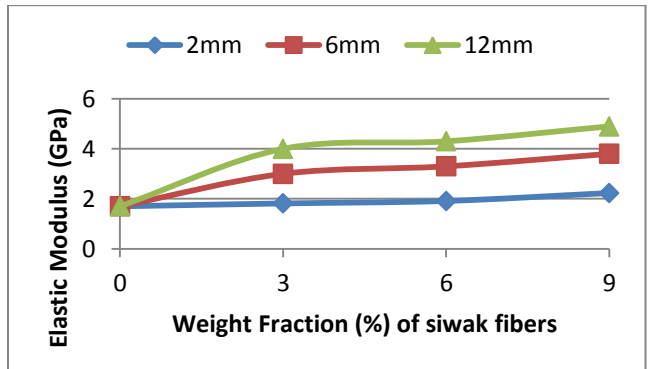


Figure 7: Young's Modulus of PMMA composites as a function of weight fraction and fiber length of siwak fibers in composite.

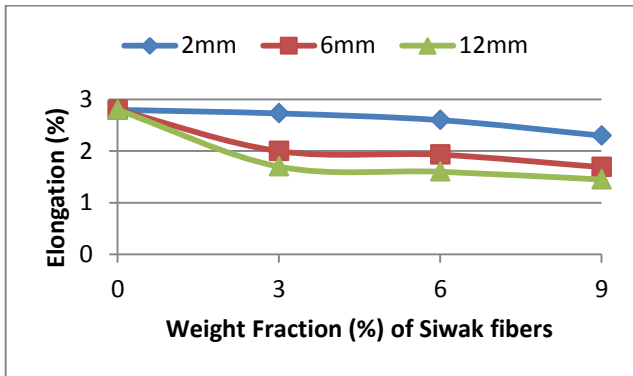


Figure 8: Elongation Percentage at Break of PMMA composites as a function of weight fraction and fiber length of siwak fibers in composite.

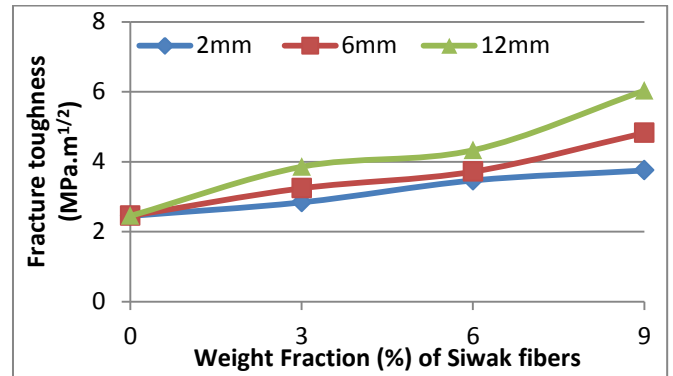


Figure 11: Fracture toughness of PMMA composites as a function of weight fraction and fiber length of siwak fibers in composite.

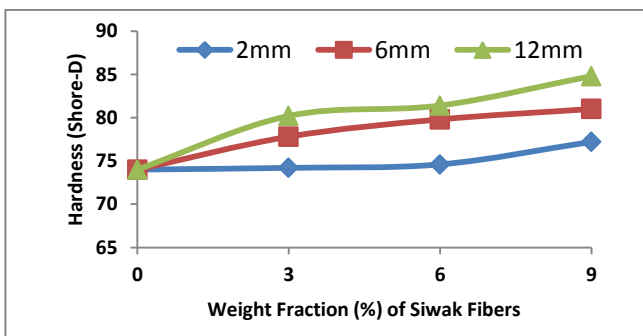


Figure 9: Hardness (Shore-D) of PMMA composites as a function of weight fraction and fiber length of siwak fibers in composite.

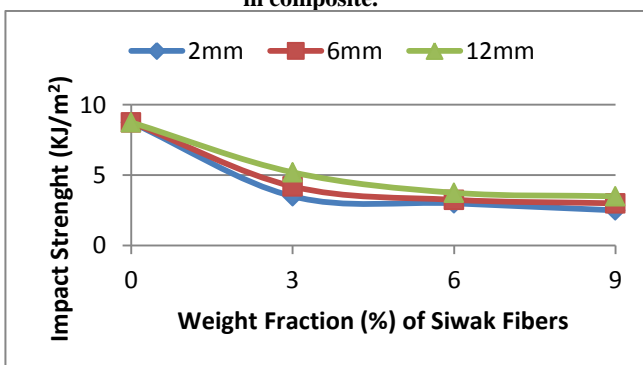


Figure 10: Impact Strength of PMMA composites as a function of weight fraction and fiber length of siwak fibers in composite.

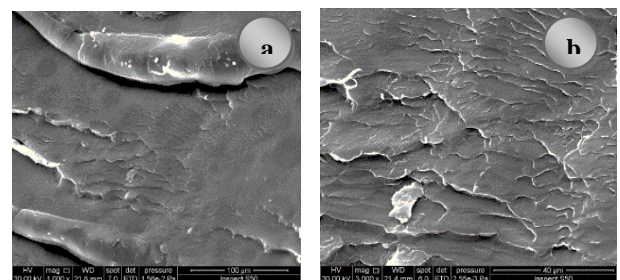


Figure 12: SEM Image of Fractured Surface Morphology of Neat PMMA Different Magnifications ((a) 1000x and (b) 3000x).

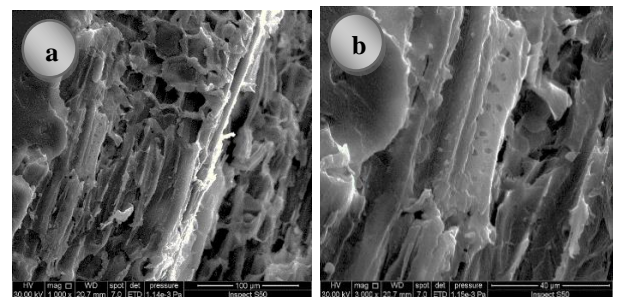


Figure 13: SEM Image of Fractured Surface Morphology of PMMA Composites Reinforced by 3% Ratio of Siwak Fiber with 2mm Fiber Length at Different Magnifications ((a) 1000x and (b) 3000x).

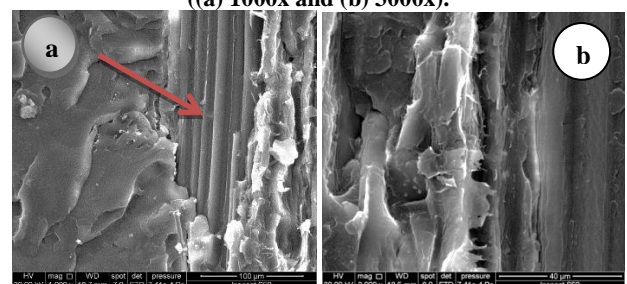


Figure 14: SEM Image of Fractured Surface Morphology of PMMA Composites Reinforced by 9% Ratio of Siwak Fiber with 12mm Fiber Length at Different Magnifications ((a) 1000x and (b) 3000x).

## 5. CONCLUSION

On the basis of the results arrived at this study, it was concluded that the incorporation of siwak fibers into the acrylic resin improved the tensile strength, modulus of elasticity and hardness, and the largest values of tensile strength and young modulus for specimens reinforced with siwak fibers are (71 MPa) and (4.9GPa) respectively at optimum condition of weight fraction (9%) and fiber length (12 mm). The SEM morphology indicates to good interfacial adhesion between the PMMA matrix and siwak fibers. As well as, no any new peak or peak shift was appear for FTIR Spectra of PMMA composite specimens with addition of siwak fibers as compared with FTIR Spectrum of neat PMMA, and this indicates there are no chemical reaction has occurred between the composite constituents. And this it may be qualifies the PMMA which reinforced with siwak fibers for use in denture base applications.

## 6. REFERENCE

- [1] R. K. Alla, S. Sajjan, V. R. Alluri, K. Gijupalli, and N. Upadhyaya, "Influence of fiber reinforcement on the properties of denture base resins, " *Journal of Biomaterials and Nano biotechnology*, Vol.4, No.1, pp.(91), (2013).
- [2] A. M. El-Sheikh and S. B. Al-Zahrani, "Causes of denture fracture: A survey", *Saudi Dental Journal*, Vol.18, No.3, (149-154), (2006).
- [3] J. Xu, L.Cong, and Y. Li, "Fabrication and mechanical properties of short sisal fiber reinforced composites used for dental application", 18 the international conference on composite materials.
- [4] O'Brien WJ., "Dental materials and their selection", 3rd ed. Chicago:Quintessence Int, pp.(216-20), (2002).
- [5] F. chen, Z. Cheng Wang and C. Jian Lin "Preparation and Characterization of nanosized Hydroxyapatite particles and Hydroxyapatite/Chitosan nanocomposite for use in Biomedical Materials", *Materials Letters* Vol.57, PP.(858-861), (2002).
- [6] D. E. Baciú, J. Simitzis, and D. Glannak Opowlos, "Synthesis and Characterization of Acrylic Bone Cement Reinforced with Zirconia Bio ceramic", *Digest Journal of Nanomaterials and Bio structures*, Vol.7, No.40, PP.(1779-1786), (2012).
- [7] J. K. Oleiwi, and S. J. Ahmed, "Studying the Tensile and Buckling for PMMA Reinforced by Jute Fibers for Prosthetic Pylon", *Engineering &Technology Journal*, Vol.32, Part (A), No.1, (2016).
- [8] P. Amuthakkannan<sup>1</sup>, V. Manikandan, J.T. Winowlin Jappes and M. Uthayakumar, "Effect of fiber length and fiber content on mechanical properties of short basalt fiber reinforced polymer matrix composites", *Materials Physics and Mechanics*, Vol.16, pp.(107-110), (2013).
- [9] M. S. Sreekanth, V. A. Bambole, S. T. Mhaske, P. A. Mahanwar, "Effect of concentration of Mica on Properties of Polyester Thermoplastic Elastomer Composites" , *Journal of Min. and Mat. Character. and Eng.*, Vol.8, No.4, PP.(271-282), (2009).
- [10] S. H. Sawalha, A. Mosleh, A. D. Manasrah, "Tensile Properties of Extruded Short Glass Fiber/Low Density Polyethylene Composites", *Iranian Polymer Journal*, Vol.16, No.10, PP.(719-726), (2007).
- [11] W. D. Callister, Jr., and D. G. Rethwisch, "Fundamentals of Materials Science and Engineering", 5<sup>th</sup> edition, John Wiley & Sons Inc., (2001).
- [12] S. C. George<sup>1</sup>, T. Jose, et.al, "Mechanical Properties of Short Sisal Fiber Reinforced Pheno Formaldehyde Eco-Friendly Composites", *Polymers from Renewable Resources*, Vol.8, No.1, PP.(27), (2017).
- [13] Ö. Karacaer, T. Polat, et.al, "The effect of length and concentration of glass fibers on the mechanical properties of an injection- and a compression- molded denture base polymer", *Journal of Prosthetic Dentistry*, Vol.90, No.4, pp.(385-9),(2003).