

Tensile and Fracture Properties of Coir Fiber Green Composites Bone Plate Fixation

Ahmed M. Hashim

Department of Materials Engineering
College of Engineering
Al-Qadisiyah University
Diwaniyah, Iraq

Email: ahmedmudhafarhashim@yahoo.co.uk

K.E. Tanner

Biomedical Engineering Division
School of Engineering
University of Glasgow
Glasgow, Scotland

Jawad .K. Ulaiwe

Department of Materials Engineering
University of Technology
Baghdad, Iraq

Abstract— Plates for long bone fractures are a major element of internal fixation technique includes various important developments in designs, materials, and implementation process during the last century. The main purpose of this paper was to introduce a new series of green composites as a plate for bone fracture applications. These green composites offer many advantages over traditional composites and biocomposites based on synthetic polymers. In current work, the new green composites bone plate based on two types of biopolymers involves biopolymethylmethacrylate thermoplastic material and bioepoxy thermosetting material reinforced with randomly oriented coir natural fiber at different weight fractions (5, 10, 15, and 20%) were fabricated by hand lay-up technique. Four forms of green composites; alkali treated with 1% NaOH and untreated coir biopolymethylmethacrylate groups A and B respectively, alkali treated with 1% NaOH and untreated corn bioepoxy groups C and D respectively were investigated. The mechanical properties were tested (tensile, and compact tension) under a flow of phosphate buffered saline PBS at 37 °C has been reported. The experimental results showed that the chemical treated coir green composites group A have the best mechanical properties than other groups under PBS condition while the untreated coir green composites group D have the weak mechanical properties due to plastization by soaked in PBS solution.

Keywords-green composites; biopolymethylmethacrylate resin; bioepoxy resin; coir natural fibers; biomechanical properties; biodegradation properties.

I. INTRODUCTION

Internal fixation is a surgical procedure in orthopedics that stabilizes and joints the ends of fractured (broken) bone by mechanical devices such as plates, pins, wires, and screws positioned entirely inside the skin. Plates for long bone fractures are a major element of internal fixation [1] and must be bear external loads for 1.5 to 2 years [2]. Various materials can be used as bone plates including metals, ceramics, and polymer based composites [3]. Metallic materials such as stainless steel, cobalt alloys, pure titanium, and titanium alloys are most widely used as internal fixation for long bone fractures because of their outstanding mechanical properties [4-6]. However, these devices are not suitable for use as internal fixation plates inside the skin for long time due to numerous limitations. The large difference in elasticity

between metals and bone causes stress shielding results in osteopenia, osteoporosis, plate loosening, and delay the healing period [7-9] as well as metallic bone plates are incompatibility with living tissue, bioactive, magnates effect [10], lower fabricability and high cost [11]. Although ceramic materials such as Al₂O₃ and ZrO₂ have been considered bioinert, but the higher degree of brittleness, hardness, and high elastic modulus lead to stress shield effect and thus making its unsuitable for use as bone plates [3]. Composites based on polymers derived from petrochemical materials results in production of harmful by degradation products when used in orthopedic surgery [12]. To eliminate these serious limitations, friendly material consist of natural fiber and biodegradable polymers are becoming highly attractive for alternative material use as bone plates which should satisfy the conditions of fully biodegradability and biocompatibility.

Biocomposites made from natural fibers as the matrix material and biodegradable polymers as the reinforcing material are called "green composites" [13]. The word "green" affixed with materials which are "biodegradable" and "renewable" in same time [12]. Frequently, they are referred as biomedical materials [14].

Natural fibers are renewable materials which extracted from natural sources [15-16] They are derived from plants, animal, and minerals but the most used are the plants ones because of their high availability and renewability in short time comparison to others [15]. In general, natural plant fiber have several advantages than synthetic fiber such as glass and carbon. They are environmental friendly material, complete biodegradable, non toxic, inexpensive, light weight, and have adequate specific strength properties [17]. Fully biodegradability of natural plant fiber can be contribute to a healthy eco-design whereas their low cost and high performance fulfils the economic attractive of industry [18].

Coconut is the tropical plant and a member of the Areceae (palm) family [19-20]. Coir is the scientific name of coconut fiber [21]. Coir is a coarse fruit fiber extracted from the tissues surrounding the seed of coconut palm (husk of a coconut) [19]. Cellulose, hemi-cellulose, and lignin are the three main content of coir fiber as shown in Table 1 [22]. Due to their high lignin content, coir fiber is consider one of the hardest natural fibers [23]. The individual coir fiber cells are narrow

and hollow as shown in “Fig. 1” with thick walls of cellulose. The length of coir fivers are varying from 100mm to 200mm [24].

Natural Fiber	Cellulose	Hemicellulose	Lignin
Coir	43-53	14.7	38-40

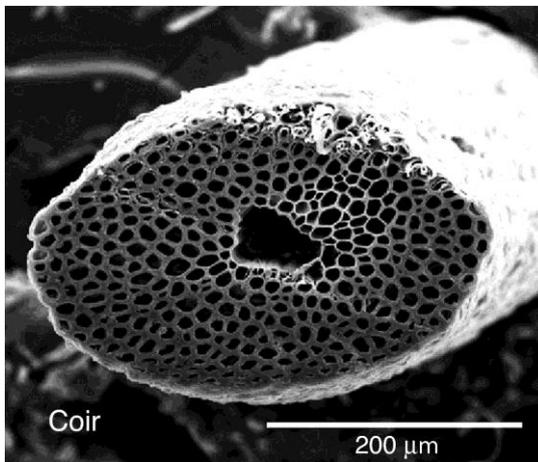


Figure 1 SEM structure of coir fiber [26].

II. MATERIALS AND METHODS

A. Matrix Materials

Two matrix materials were investigated in this study. Polymethylmethacrylate (PMMA) was produced using 617H55 resin and 617P37 hardener (Otto Bock Healthcare plc, Egham, Surrey, UK). SUPER SAP® 100/1000 high bio-epoxy system composed of SUPER SAP® 100 resin and SUPER SAP® 1000 hardener (Professional Epoxy Coating, Kendal, Cumbria, UK).

B. Reinforcing Fibers

Coconut fruit were obtained from local shops (Glasgow), the fibers were extracted from the husk and then dried at 37 °C for 240 hours in an incubator (Sanyo MIR262, Japan) before surface treatment. All the natural fiber types were cut in length of 10 mm. Alkaline treatment is one of the most commonly used chemical treatments for natural fibers used to reinforce thermoplastics and thermosets. The coir fibers were soaked in 1% NaOH solution at room temperature for 2 hours. The fibers were then washed several times with distilled water to remove any NaOH remaining on the fiber surface. The fibers were then dried at 37 °C for 10 days in an incubator. “Fig. 2” show the short coir fiber before and after alkali treatment.



Figure 2 Coir natural fiber: before (left) and after (right) alkali treatment.

C. Composite Preparation

A PTFE mold of dimensions 240 × 110 × 4 mm was used to cast the composite sheets for tensile test while a PTFE mold of dimensions 220 × 110 × 3 mm was used for casting the sheets for compact tension testing. A hand lay-up technique was used to prepare the samples. The coir fibers were then distributed uniformly over the mixture, and the remainder of the mixture was then poured into the mold which was immediately, before reaching hardening, pressurized to 2 tons for 30 minutes, giving a pressure of 27.58 MPa. After compression molding the material was allowed to cure at room temperature for 24 hours. Finally, the samples were taken out of the mold and machined into the required sizes for ASTM standard mechanical tests.

Group	Green composite	Composition % wt	Chemical Treatment
Group A	Coir/PMMA	5 Coir/95 PMMA	Yes
		10 Coir/90 PMMA	
		15 Coir/85 PMMA	
		20 Coir/80 PMMA	
Group B	Coir/PMMA	5 Coir/95 PMMA	No
		10 Coir/90 PMMA	
		15 Coir/85 PMMA	
		20 Coir/80 PMMA	
Group C	Coir/Epoxy	5 Coir/95 PMMA	Yes
		10 Coir/90 PMMA	
		15 Coir/85 PMMA	
		20 Coir/80 PMMA	
Group D	Coir/Epoxy	5 Coir/95 PMMA	No
		10 Coir/90 PMMA	
		15 Coir/85 PMMA	
		20 Coir/80 PMMA	

The bio PMMA matrix and bio epoxy matrix green composites were manufactured with 5, 10, 15, and 20% weight content of natural corn fibers with or without alkali treatment. Non-filled PMMA and epoxy were manufactured in the same manner to provide 0% filler samples.

Before testing all samples were immersion in phosphate buffered saline (PBS) solution (Sigma-Aldrich, United Kingdom) at 37 °C for 7 days. The composite systems were prepared are divided into 8 groups as shown in Table 2.

D. Mechanical Testing

Tensile test

Tensile testing was performed according to ASTM D638-02 standard at materials testing laboratory – School of science and engineering – University of Glasgow – Scotland using an MTS 850 Mini Bionix® II universal testing machine (MTS, Minneapolis, USA) as shown in “Fig. 3”. The dimensions of the gauge section were 64 x 10 x 4 mm. The cross-head speed was 1 mmmin⁻¹ at 37 °C with a flow of saline over the sample during testing to ensure constant temperature as seen in “Fig. 4”. Five specimens for each sample were tested and the tensile strength and tensile modulus calculated from the following expressions [27]:

$$\text{Tensile Strength } (\sigma) = F/b.h \quad (1)$$

$$\text{Strain } (\varepsilon) = \Delta l/l \quad (2)$$

$$\text{Tensile Modulus } (E) = \sigma/\varepsilon \quad (3)$$

Where:

F = force (N), b = width of specimen (mm), h = thickness of specimen (mm), Δl = change in length (mm), l = original length of specimen (mm), σ = stress (MPa), and ε = strain.

The average values and standard deviations were calculated.

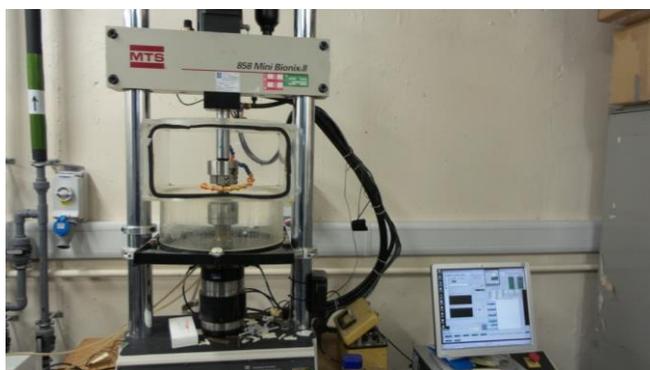


Figure 3 MTS 850 Mini Bionix® II universal testing machine (MTS, Minneapolis, USA).



Figure 4 A 5coir/95epoxy green composite sample during the tension test under a flow of PBS at 37 °C.

Fracture toughness

Samples of each material were machined into compact tension specimens according to ASTM E647-13a with dimensions of 38 mm x 40 mm x 3 mm. The samples were tested at a cross head speed of 1 mm min⁻¹ at 37 °C with and without a flow of saline over the sample during the test as seen in “Fig. 5”. The load – displacement data were recorded during the test. Again five specimens for each sample were tested.

From the maximum load in the load – displacement curve, the fracture toughness K_{IC} was calculated using the following equation [28]:

$$\Delta K = P/B(W)^{1/2} \cdot ((2+\alpha) / (1-\alpha)^{3/2}) [0.886 + 4.64\alpha - 13.32\alpha^2 + 14.72\alpha^3 + 5.6\alpha^4] \quad (4)$$

Where:

$\alpha = a/W$; expression valid for $a/W \geq 0.2$, a = is the crack length, B = is the thickness of the specimen, and W = is the

dimension from the load line to the right hand edge of the specimen.



Figure 5 A 10coir/90PMMA green composite sample during the compact tension test under a flow of PBS at 37 °C.

Degradation Test

The degradation test of the green composite samples were performed according to the standard BS ISO 10993-1 [29]. Five specimens of each groups of green composites were placed into 170 mm polypropylene cups and then filled with phosphate buffered saline PBS as degradation medium. All cups puts in an incubator at 37 °C for 40 days. Waterproof pH and temperature tester (AD12-Romania) was used to measurements of pH of all samples immersed in phosphate buffered saline PBS at 37 °C. The data was plotted pH change as against time.

E. Results and Discussion

Tensile Test

“Fig. 16” and “Fig. 7” shows the stress-strain curves for pure biopolymethylmethacrylate with and without immersion in phosphate buffered saline (PBS) at 37°C for 7 days respectively. It has been seen that biomethylmethacrylate thermoplastic material exhibited brittle failure in dry and wet conditions. As shown in Table 3, the experimental results

showed that there is a little effect of degradation medium on tensile test results (tensile strength, percentage elongation and tensile modulus) for pure bioPMMA. The drop in tensile strength, elongation, and tensile modulus are very small about 5.74%, 12.33%, and, 3.17% respectively of the biomaterial in dry condition.

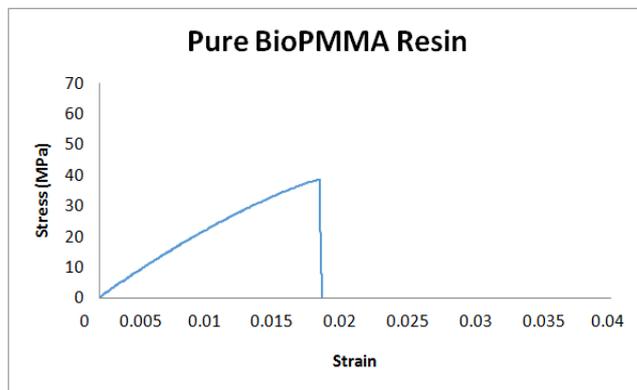


Figure 6 Stress-strain curve for pure bioPMMA resin without immersion in PBS at 37°C.

Matrix Resin	Immersion in PBS at 37 °C	Average tensile strength MPa	Average Young’s modulus GPa	Average Strain to failure %
PMMA	No	38.16	2.93	1.46
PMMA	Yes	36.09	2.84	1.64

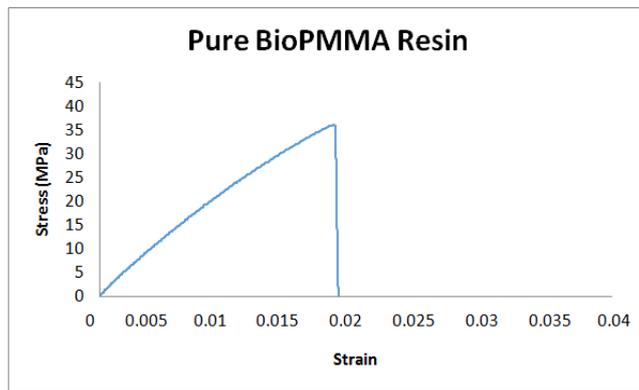


Figure 7 Stress-strain curve for pure bioPMMA resin with immersion in PBS at 37°C.

In other side, the stress-strain curve for dry pure bioepoxy thermosetting resin is shown in “Fig. 8” while for the same biomaterial immersion in PBS at 37°C after 7 days is shown in “Fig. 9”. In case of degradation condition, the larger drops in tensile strength of 69% and tensile modulus of 74.34% and meanwhile increases in strain to failure of % compared to that

of dry condition are related to the plastization of the bioepoxy resin so the immersion in PBS at 37°C led to change in fracture mode of pure bioepoxy resin from brittle failure to more ductile failure after 7 days of immersion. Table 4 show the major changes in tensile test properties of pure bioepoxy resin before and after immersion in PBS at 37°C for 7 days.

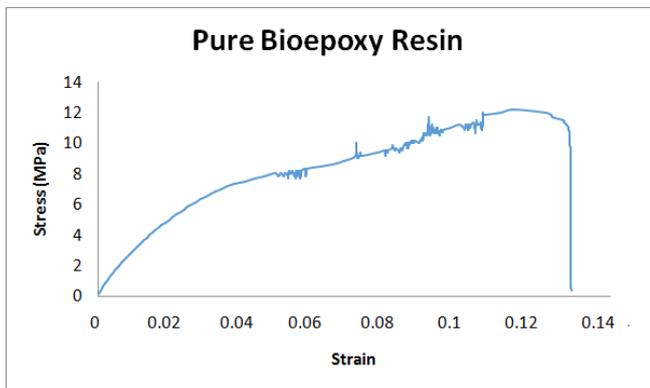


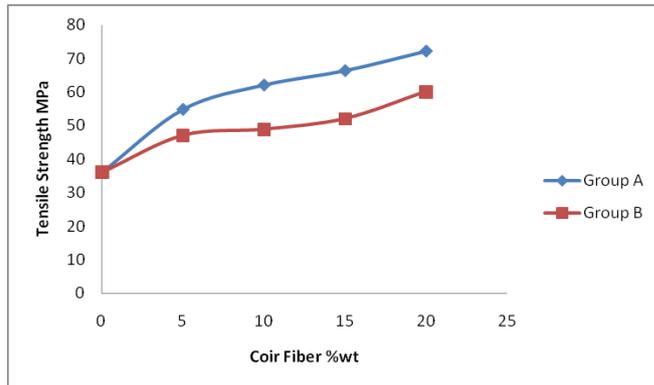
Figure 8 Stress-strain curve for pure bioepoxy resin without immersion in PBS at 37°C.

Matrix Resin	Immersion in PBS at 37 °C	Average tensile strength MPa	Average Young's modulus GPa	Average Strain to failure %
Epoxy	No	55.29	3.17	3.2
Epoxy	Yes	17.15	0.75	14.33

The 1% NaOH alkali coir fiber treated have the major effect on both tensile strength and tensile modulus of both PMMA and epoxy based green composites at degradation medium (groups A and C) as shown in “Fig. 10”, “Fig. 11”, “Fig. 12”, and “Fig. 13” respectively. It was found that both tensile strength and tensile modulus increased as fiber concentrations increased. The tensile strength and tensile modulus were enhanced by 20% and 13.4% of 20coir/80PMMA green composites from 60.12 MPa (group B) to 72.17 MPa (group A), and from 3.59 GPa (group B) to 4.07 GPa (group A) respectively. For coir epoxy green composites (groups C and D), the largest results were found in group C of 68.37 MPa and 3.83 GPa respectively, while the lowest results in group D of 38.62 MPa and 3.08 GPa respectively.

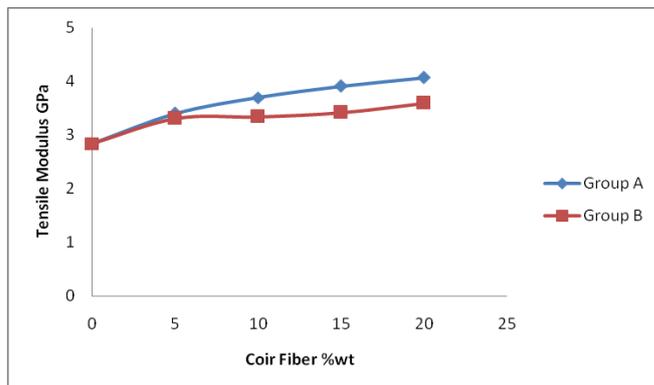
The tensile test results for natural fiber green composites (groups A, B, C, and D) showed that the bioepoxy resin are degraded more level than biopolymethylmethacrylate resin when immersion in PBS at 37°C for 7 days due to higher degree of plasticity for bioepoxy based resin. As a result, tensile strength and tensile modulus decrease significantly compared to those for composites based on bioPMMA resin. It

has been observed that the percentage decrease in tensile modulus at degradation condition for untreated and treated corn epoxy green composites (groups C and D) is larger than the percentage decrease in tensile strength for the same



composites.

Figure 10 Effect of immersion in PBS at 37°C for 7 days on tensile strength



of treated and untreated coir/PMMA green composites (Groups A and B).

Figure 11 Effect of immersion in PBS at 37°C for 7 days on tensile modulus of treated and untreated coir/PMMA green composites (Groups A and B).

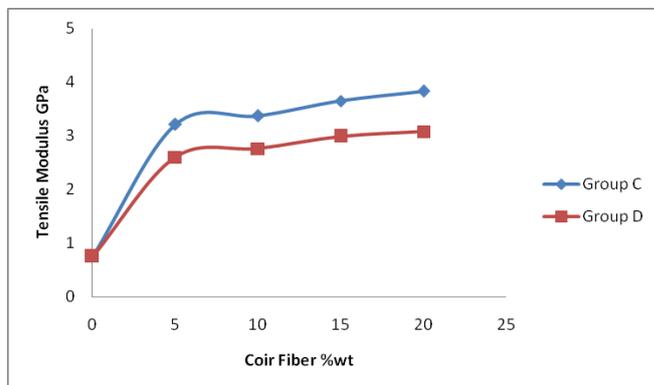


Figure 12 Effect of immersion in PBS at 37°C for 7 days on tensile strength of treated and untreated coir/ epoxy green composites (Groups C and D).

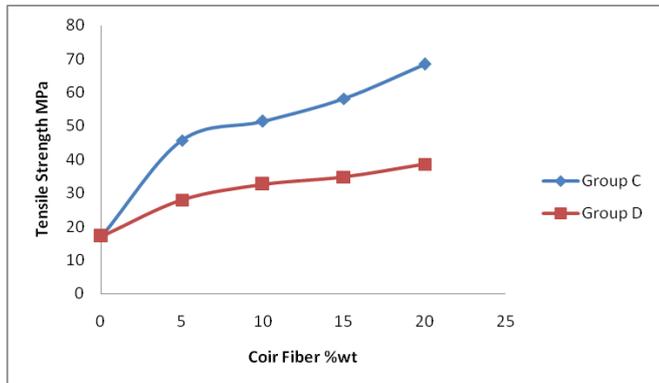


Figure 13 Effect of immersion in PBS at 37°C for 7 days on tensile modulus of treated and untreated corn/ epoxy green composites (Groups C and D).

It has been shown that the moisture absorption results in reduction of tensile properties of natural fiber polymer composites due to plasticization of the matrix and presence of abundant polar hydroxide groups in the structure of natural fibers [30]. These reasons result in high level of moisture absorption for natural fiber composites based on polymer resins. Another reason that increases the rate of moisture absorption in natural fiber composites is the presence of voids and cracks at the surface of composites and then allowed for water molecules to penetrate inside the matrix material along the fiber-matrix interface by capillary action caused reduction in tensile strength and tensile modulus due to the weakness of the fiber-matrix interface [31].

One of the important significant effect of the alkali treatment is the disruption of hydrogen bonding in the natural fiber surface by ionization of the hydroxyl group to the alkoxide according to the following reaction which takes place during the treatment [32-33]:



The reaction above led to increase the surface roughness of the natural fiber due to remove some amounts of lignin, oil, and was formed the external layer of the fiber surface, meanwhile increase the amount of cellulose exposed on the fiber surface and then increasing the bonding between hydrophilic fiber and hydrophobic matrix resulting in better tensile properties of the composites [34-35].

Also, it has been shown that the chemical treatment improved tensile properties of PBS immersed corn fibers green composites (groups A and C). This improvement is due to the partial dissolution of hemicellulose compounds such as wax and gum. Hemicellulose is considered the main responsible for the moisture absorption of the natural fiber [36].

Compact Tension Test

The effect of alkali treatment and immersion in PBS solution at 37 °C on fracture toughness K_{IC} of both

corn/PMMA and corn/epoxy biocomposites are shown in “Fig. 14” and “Fig. 15” respectively. In all conditions, the fracture toughness K_{IC} increased as increased in fiber concentrations especially in groups C and D. The experimental results showed that there is a little enhancement in fracture toughness K_{IC} by chemical treatment of coir fiber PMMA green composites (group A). The maximum average values of K_{IC} of samples in groups A and B were 1.71 MPa (mm)^{1/2} and 1.6 MPa (mm)^{1/2} respectively. The significant effects of PBS immersion was occurred in treated and untreated coir epoxy green composites (groups C and D) respectively due to the higher plasticization of the matrix material where the values of maximum fracture toughness K_{IC} enhancement by 67% from 2.33 GPa of pure epoxy resin to 3.88 MPa (mm)^{1/2} of epoxy composites containing 20% weight fraction of untreated coir fiber (group D) and by 72% of 4 MPa (mm)^{1/2} of composites containing treated corn fiber of the same weight fraction.

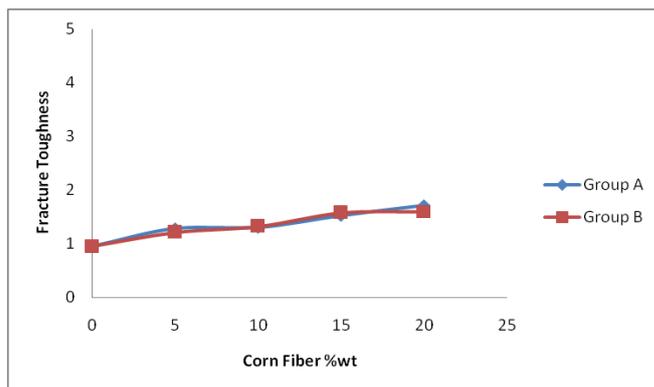


Figure 14 Effect on immersion in PBS at 37°C for 7 days on fracture toughness of treated and untreated coir/PMMA green composites (Groups A and B).

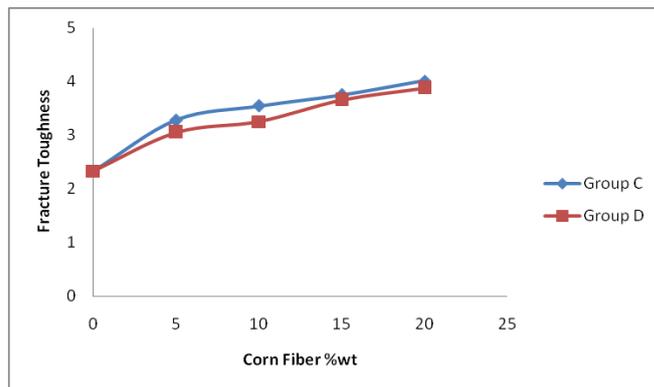


Figure 15 Effect on immersion in PBS at 37°C for 7 days on fracture toughness of treated and untreated coir/epoxy green composites (Groups C and D).

Degradation Test

“Fig. 16” and “Fig. 17” shows the change in pH values against degradation time in phosphate buffered saline (PBS) at

37°C for pure bioPMMA and bioepoxy respectively. For pure bioPMMA, the Ph value slowly degraded from 7.6 (the pH value of fresh PBS) to reach 7.01 after 23 days and then increased to maximum value of 7.26 after 30 days and remained in the range of 7.25-7.26 up to end of the degradation period (40 days).

In contrast, the pH value for pure bioepoxy is rapidly increased in short time (7 days) from 7.6 to reach the maximum value of 9.11 and then decreased to 7.37 after 38 days and remained at 7.27 in two last days of the degradation period. The results indicated that the pure bioepoxy have more plasticity than pure bioPMMA.

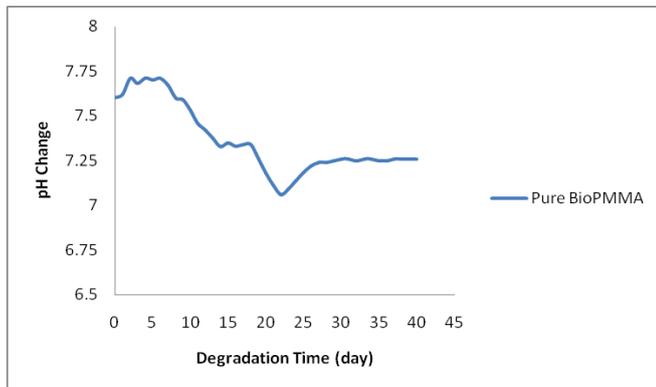


Figure 16 pH change of PBS against degradation time for pure bioPMMA during immersion in PBS at 37°C.

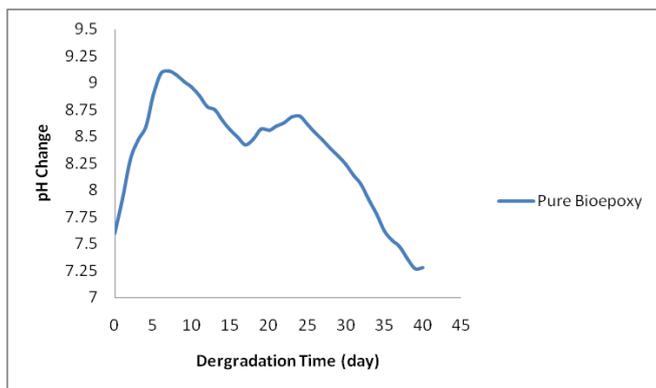


Figure 17 pH change of PBS against degradation time for pure bioepoxy during immersion in PBS at 37°C.

The degradation behavior of treated and untreated corn/PMMA green composites (groups A and B) are the same where the pH values are increased after 2 days and then degraded to the minimum values after 21-25 days excepting the composites have the composition 20corn-80PMMA in each group after 14 and 15 days respectively, Then the pH values increased gradually and maintained in the range of 7.21-7.24 at the last five of the degradation period in all

compositions as shown in “Fig. 18” and “Fig. 19” respectively.

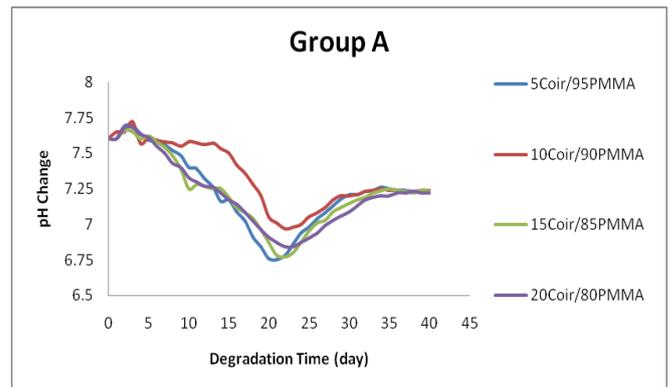


Figure 18 pH change of PBS against degradation time for treated coir/PMMA green composites (group F) during immersion in PBS at 37°C.

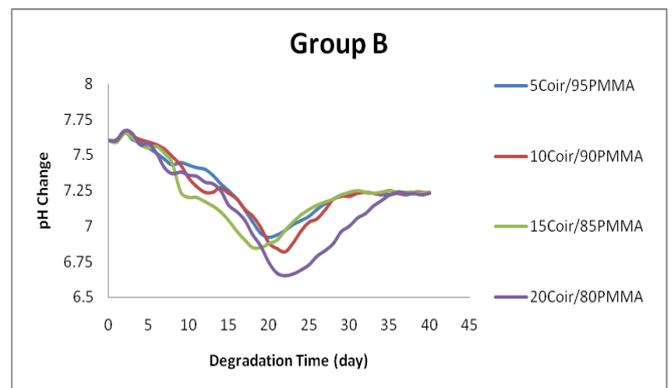


Figure 19 pH change of PBS against degradation time for untreated coir/PMMA green composites (group H) during immersion in PBS at 37°C.

The degradation behavior of treated and untreated coir/epoxy green composites in PBS at 37°C are differs than for those composites based on biopolymethylmethacrylate reinforced with both treated and untreated corn and coir natural fibers (groups C, and D) as shown in “Fig. 20” and “Fig. 21” respectively. In groups C and D, the change in pH values are increasing drastically in first 12-13 days from 7.6 to exceeding 9 especially in group D where the maximum values of ph are 9.04 and 9.01 for 5corn-95epoxy and 10corn-90epoxy green composites respectively. Then the values of pH in both groups are slowly decreased and maintained at the end of degradation period in the range of (7.28-7.29) and (7.30-7.32) for groups C and D respectively.

The experimental results showed that the alkali treatment of corn natural fiber with 1% NaOH increased the values of pH of green composites of both groups in all compositions.

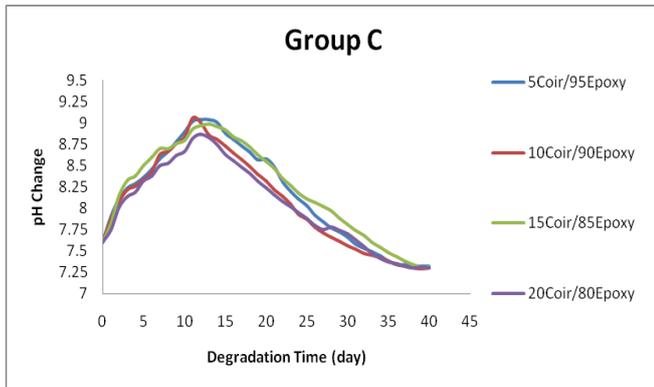


Figure 20 pH change of PBS against degradation time for treated corn/epoxy green composites (group C) during immersion in PBS at 37°C.

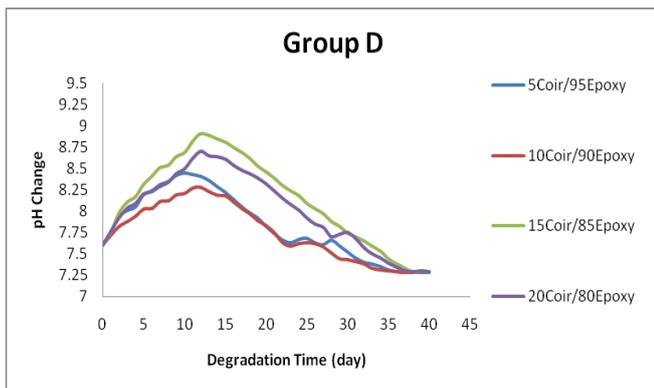


Figure 21 pH change of PBS against degradation time for untreated corn/epoxy green composites (group D) during immersion in PBS at 37°C.

F. Conclusions

- The mechanical properties of biopolymethylmethacrylate resin are not affected by immersion in PBS at 37°C, whereas in case of bioepoxy resin larger drops were occurred due to plastization of the material.
- The alkali treatment by 1% NaOH of corn natural fibers reinforced polymethylmethacrylate green composites give better tensile properties in PBS conditions than in case of bioepoxy based resin. The treated corn polymethylmethacrylate green composites (group A) have the best mechanical properties, whereas the untreated corn epoxy green composites (group D) have the weakness properties.
- The most significant of PBS immersion at 37°C fracture toughness properties were occurred in coir epoxy green composites due to higher plastization of the matrix resin.
- The experimental results showed that the alkali treatment of corn natural fiber with 1% NaOH increased the values of pH of green composites of both groups in all compositions.

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