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Preparation and Characterization of Pineapple Leaf Fiber Reinforced Epoxy Composite: Effect of Gamma Radiation

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Abstract: Natural fiber reinforced composite materials have been gaining popularity because, natural fibers are cheap and biodegradable. Pineapple leaf fiber (PALF) is a natural fiber and an agro waste. This PALF was treated with sodium hydroxide to clean the surface. 10%, 15%, 20%, 25% and 30% PALF based epoxy composites were fabricated by hand-lay-up technique. The mechanical properties such as tensile strength (TS), percentage of elongation at break (%Eb) and impact strength (IM) for composites were found to increase with increasing fiber content. The highest TS, %Eb and IM for 30% PALF based epoxy composite were 51.6 MPa, 15.882% and 14.189 Kg-cm respectively but tensile modulus (TM) was decreased with increasing fiber content. Degradation both in water and soil were done and the mechanical properties during degradation decreased a little bit over time because of the hydrophobic epoxy (matrix) material. 2.5 KGy, 5.0 KGy, 7.5KGy and 10 KGy gamma radiation doses were applied to composites and the effect of these doses were analyzed. For PALF based epoxy composite TS, TM, %Eb and IS values increased for certain value of gamma doses and for 7.5 KGy, these values were the highest while the values decreased in the increasing gamma radiation. Thermogravimetric analyses of PALF, epoxy and PALF based epoxy composite of non-radiated and irradiated composites materials were investigated. The thermogram of composite material was different than matrix materials as well as reinforced materials. The surface morphology of the reinforced, matrix and composite materials were investigated using scanning electron microscope (SEM).

Keywords: Mechanical Properties, Thermo Gravimetric Analysis, SEM, Radiation Effect, Degradation

1. Introduction

Natural fiber has been of great interest in research in recent years, because it is not harmful for environment. Artificial fibers make environmental pollution and are hazardous for human health. The global yearly production of the lignocellulosic materials is approximately 2×10^{11} tons. In which approximately 3×10^{7} tons of natural fibers are produced and this fibers are utilized for a wide range of applications, such as clothing, paper pulp, packaging, automotive field, and building and construction materials and sports equipment [1]. For this reason 2009 was declared as the international year of natural fibers by the UN Food and Agriculture Organization (FAO) [2]. Pineapple leaf fiber (PALF) is waste materials and natural fiber in agricultural field, which mainly grows in Asia. Pineapple leaf fiber can be used in various fields like, packaging, biomedical, furniture and automotive etc. that way the farmer who cultivate pineapple get additional money and it helps to make balance in ecological system [3]. The cellulose content in PALF is the highest among various natural fibers extracted from plant leaves. The fiber from PALF was obtained by manual scrapping method [4]. PALF has a high crystalline cellulose compare to other plant fibers, so PALF has better mechanical properties compared to other plant fiber [5-7]. The density, softening point, tensile strength, tensile modulus, specific modulus, elongation at break and moisture regain of PALF are 1.526 gm/cm^3, 104°C, 170 MPa, 6260MPa, 4070MPa, 3% and 12% respectively [8]. Since PALF has good mechanical properties such as tensile strength, tensile modulus etc. It is possible to make fiber reinforced polymer composite, low density polyethylene composite and biodegradable plastic composite [9]. Natural fiber composite showed excellent mechanical properties, electrical properties and environmental properties such as biodegradability, recyclability and renewability [10-12]. The mechanical properties of the composite depend upon the finer length, matrix ratio and rearrangement of fiber [13, 14]. The orientation of fiber for example transverse, longitudinal and random orientation have effect on mechanical properties of fiber reinforced polymer composite [15]. In composite having longitudinal orientation has better mechanical properties than those of transverse orientation of fiber [16]. The mechanical and other properties of fiber as well as composite depend upon the surface modification of the fiber, like alkaline treatment, silane treatment and grafting with malic anhydride copolymer [17-19]. Alkaline treated fiber based polyamide-6 composite shows better mechanical properties than silane treated polyamide-6 composite [20]. Epoxy is either any of the basic components or the cured end product of epoxy resins, as well as a colloquial name for the epoxide functional group [21]. PALF is hydrophilic in nature and can easily degrade under general environmental condition. On the other hand, epoxy is hydrophobic and does not degrade under adverse environmental condition (in water and soil). So PALF based epoxy composite may have good mechanical as well as thermal properties. The aim of the research is to make composites which have good mechanical and thermal properties. In addition to find the most suitable gamma radiation dose.

2. Experimental

2.1. Materials

The following materials were used for the fabrication of pineapple leaf fiber reinforced epoxy composite: Pineapple leaf fiber collected from Madhubon, Tangail, a local market of Bangladesh, Epoxy resin and Hardener were collected from our laboratory.

2.2. Preparation of Pineapple Leaf Fiber

Before the PALF can be used as a filler, an alkaline treatment was carried out. The raw pineapple leaf fiber was first cut into pieces of 15 cm length. After that these pieces were immersed in 2% concentrated (w/v) solution of NaOH and stirred continuously for 2 h. Then the fiber was washed several times to remove alkali and finally the solution was washed with 1% acetic acid solution to neutralize. After washing several times the fiber was oven dried at 60°C for 12 hours. The purpose of chemical treatments are to remove all the impurities, treat the fiber surface and to stabilize the molecular orientation.

2.3. Fabrication of PALF Based Epoxy Composite

For the preparation of pineapple leaf fiber reinforced epoxy composite, the fiber was cut into 12 cm in length and the fiber was placed in 15 cm bed. The epoxy resin with 1% hardener was uniformly circulated over the fiber bed in all direction. Then it was taken for 12 hour for making hard solid form. Finally the composites were cut into desired size for different type of characterizations.

3. Mechanical Test

3.1. Tensile Properties

Tensile Strength (TS), Tensile Modulus (TM), and Percentage of Elongation at break (%Eb) epoxy and both irradiated and nonradiated composites were measured according to ASTM D638 having efficiency of 1% in a universal testing machine (model: H50KS, Hounsfield, UK) with a crosshead speed of 20 mm/min. Composites were cut into 20 mm in length and the applied force of the machine was 500 N. We took the average value of 5 similar samples at ambient temperature and the relative humidity of the room was 60 ± 5%.

3.2. Impact Test

Impact strength of the samples were determined according to ASTM D-256 on a Hung Ta^{TM}Izod Impact tester, model HT8041B IZOD.

3.3. Degradation

The Composites were cut into a specific measurement of 60mm×20 mm×2 mm sized. Then the samples were kept under soil at depth of 12cm from the surface of earth. In addition other samples were kept under water for degradation. After a definite time period the samples were taken out from soil and water and washed several times to removed dirt and soil. Then the samples were dried at oven at 105°C for 2 hours. After cooling at room temperature, the samples were ready for testing.

3.4. Gamma Radiation (γ Radiation)

The Composites were cut into a specific measurement of 60mm×20 mm×2 mm sized and packed in polyethylene bag to maintain air tight. Cobalt 60 was the radiation source. 2.5 kGy, 5.0 KGy, 7.5KGY and 10 KGy gamma radiation doses were applied to composites and the effect of these doses were analyzed.

3.5. Thermo Gravimetric Analysis (TGA)

TGA analysis measures the amount of weight change of a material, either as a function of increasing temperature. The TGA value of Pineapple Leaf Fiber, Epoxy and PALF based epoxy composite both irradiated and nonradiated were measured. The thermograms were recorded with TGA-50, Shimadzu, Japan. The initial temperature was room temperature and final temperature was 800°C. The temperature rate was 10°C/min. Samples were kept in Alumina cell at inert (nitrogen) atmosphere.

3.6. Scanning Electron Microscopic (SEM) Analysis

The surface morphologies of the matrix, reinforced and composite material were investigated by scanning electron microscope (JEOL JSM-840 SEM).

4. Result and Discussion

4.1. Mechanical Properties

The TS, TM, %Eb and IS of PALF based epoxy composites were measured for the 10%, 15%, 20%, 25% and 30% (weight %) of PALF. With the increased percentage of PALF in composite increased the TS, %Eb and IS value in a linear fashion. The reason behind this improvement of these values is that the TS value is much higher in PALF than the epoxy. On the other hand with the increased the percentage of PALF in composite decreased the TM in a linear fashion. The reason behind this TM was that the elongation of the composite was much higher in PALF than the epoxy.

The tensile strength of epoxy was 27.112 MPa. The minimum TS value among the composites, 36.75 MPa was obtained at 10% PALF and 90% epoxy based composite. For 30% PALF and 70% epoxy based composite the highest TS was obtained 51.6 MPa and this value was 40% higher than the 10% PALF/Ep based composite and 90% higher than epoxy. The tensile modulus of epoxy was 444.1 MPa. The minimum TM value among the composites, 318.94 MPa was obtained for 30% PALF and 70% epoxy based composite. For 10% PALF and 90% epoxy based composite the highest TM value was obtained 392 MPa and this value was 19% higher than the 30% PALF/Ep based composite and 28% lower than epoxy.

The Percentage of Elongation of epoxy was 6.27%. The minimum percentage of elongation value among the composites 9.226% was obtained for 10% PALF and 90% epoxy based composite. For 30% PALF and 70% epoxy based composite the Percentage of Elongation was obtained 15.882% and this value was 72% higher than the 10% PALF/Ep based composite and 153% higher than epoxy. The impact strength of epoxy was 4.334 Kg-cm. The minimum impact strength among the composites, 9.619 Kg-cm was obtained at 10% PALF and 90% epoxy based composite. At the 30% PALF and 70% epoxy based composite the height impact strength was obtained 14.189 Kg-cm and this value was 47.5% higher than the 10% PALF/Ep based composite and 227.38% higher than epoxy.

These properties were shown in Figure 1.

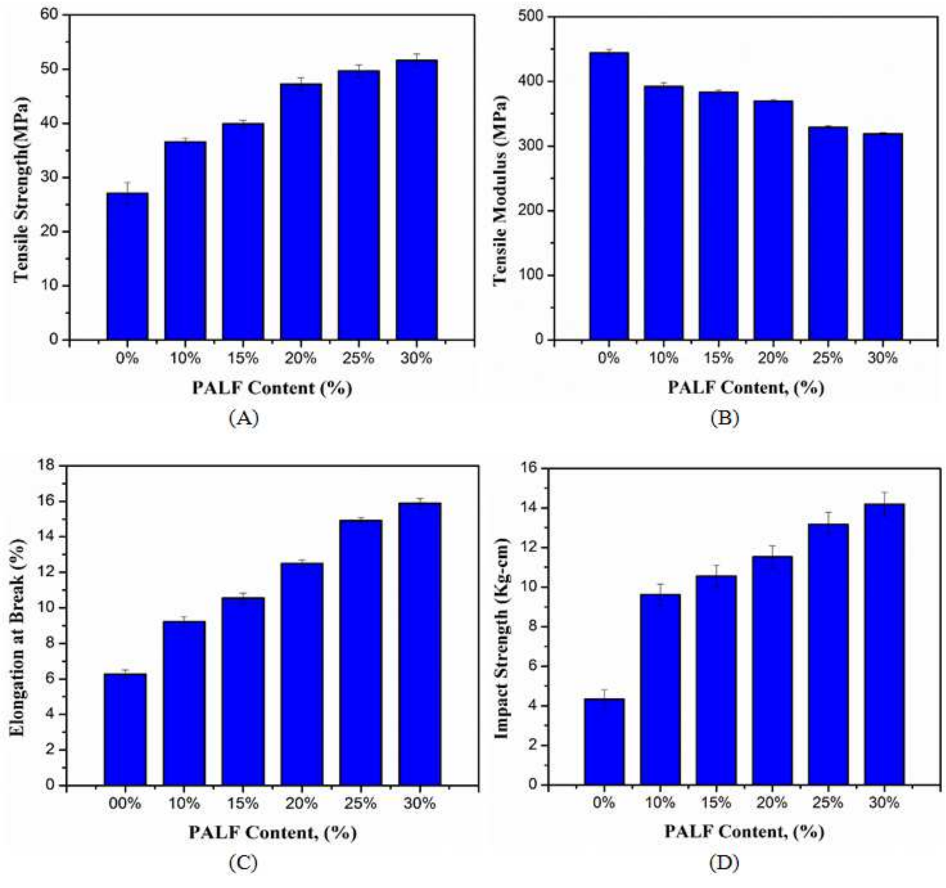

Figure 1. *Mechanical properties of the samples (A) Tensile Strength, (B) Tensile Modulus (C) Percentage of Elongation at break and (D) Impact Strength of the composites.*

4.2. Thermogravimetric Analysis (TGA)

4.2.1. Thermogravimetric Analysis of PALF, Epoxy and Composite

The TGA result of PALF was illustrated in figure 2. At initial temperature weight loss occurred at 91.88°C and 6.69% weight was lost at this temperature and this weight was the weight of water content in PALF. The cellulose of PALF was converted to gaseous compounds at 369°C and that was the major weight loss. The residues that formed after degradation required a higher temperature for subsequent degradation. The degradation of final residues occurred at 545°C.

The TGA result of Epoxy was illustrated in figure 4. At initial temperature weight loss occurred at 328°C and 7.81% weight was lost at this temperature and this weight was lost because of the presence of some volatile compounds. Hydrocarbons in epoxy were converted into gaseous compounds at 436°C and that was major weight loss (77.9%). The residue that formed after degradation required higher temperature for subsequent degradation. The degradation of final residues occurred at 660°C.

The TGA result of PALF/Ep composite was illustrated in figure 4. At initial temperature weight loss occurred at 286°C and 8.13% weight was lost at this temperature and this weight was lost because of the presence of some solvents in the composite. The major weight loss was occurred at temperature 440°C due to degradation and volatilization of epoxy along with fiber containing composite. The residue that formed after degradation required a higher temperature for the subsequent degradation. The degradation of final residues occurred at 710°C.

4.2.2. Thermogravimetric Analysis of Irradiated PALF Based Epoxy Composite

The TGA results of irradiated PALF/Ep composites were illustrated in figure 4. For 2.5 KGy irradiated composite initial weight loss occurred at 275°C and 6.56% weight was lost at this temperature and this weight was lost because of some solvents present in the composite. The major weight loss occurred at 433°C due to degradation and volatilization of epoxy along with fiber present in the respective composite. The residue that formed after degradation required a higher temperature for the subsequent degradation. The degradation of final residues occurred at 651°C.

For 5.0 KGy, irradiated composite initial weight loss occurred at 295°C and 7.98% weight was lost at this temperature and this weight was lost because of some solvents present in the composite. The major weight loss occurred at 448°C due to degradation and volatilization of epoxy along with fiber present in the respective composite. The residue that formed after degradation required a higher temperature for the subsequent degradation. The degradation of final residues occurred at 697°C.

For 7.5 KGy irradiated composite initial weight loss occurred at 296.5°C and 6.69% weight was lost at this temperature and this weight was lost because of the presence of some solvents in the composite. The major weight loss occurred at 445°C due to degradation and volatilization of epoxy along with fiber present in the respective composite. The residue that formed after degradation required a higher temperature for the subsequent degradation. The degradation of final residues occurred at 718°C.

For 10 KGy irradiated composite initial weight loss occurred at 285°C and 6.36% weight was lost at this temperature and this weight was lost because of the presence of some solvents in the composite. The major weight loss occurred at 442°C due to degradation and volatilization of epoxy along with fiber present in the respective composite. The residue that formed after degradation required a higher temperature for the subsequent degradation. The degradation of final residues occurred at 654°C.

From these results, it can be concluded that foe Gamma dose of 7.5KGy, the irradiated composite showed the best TGA result.

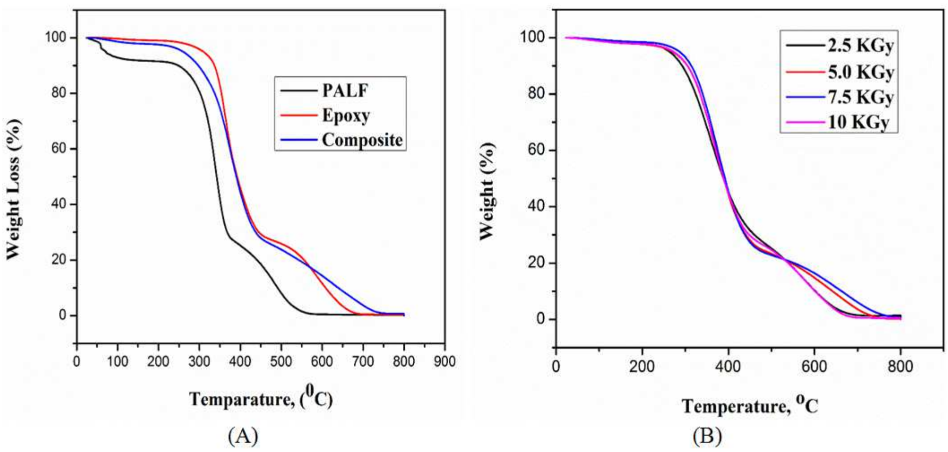

Figure 2. *(A) Thermogram of PALF, epoxy, composite and (B) Thermogram of irradiated composite.*

4.3. Degradation

Degradation tests of the composites of PALF/Ep were performed in soil and water at ambient conditions for up to 6 weeks. TS were plotted against degradation time as shown in figure 3. It was found that for PALF/Ep composites TS decreased slowly over time in water and for soil degradation it decreased very slowly over time but faster decreasing TS than water degradation. After 6 weeks of soil degradation PALF/Ep composites lost almost 9.58% of TS. On the other hand, in water degradation the composites lost only 2.26% of TS. From this it is clear that PALF/Ep based composites retained much of their TS during water degradation than soil degradation. Pineapple leaf fiber is biodegradable in nature but epoxy is not biodegradable. For this reason the composites showed small degradation rate both in water and soil. The main part of PALF is cellulose which easily degrades at ambient condition, because cellulose absorbs water within short time. Since cellulose are hydrophilic in nature can easily degrade and thus its mechanical properties decreases. In soil water and microorganism break down the cellulosic part of the composites and thus by two ways the composites were degraded. On the other hand in water degradation process, only water can degrade the composite, because we used distilled water which contained no microorganism. As a result, the mechanical properties of the composites in soil decreased significantly compared to water. But this value of degradation rate was very slow compared to the normal PALF fiber because PALF/Ep based composites are strongly hydrophobic and repel water thus retaining much of their integrity during exposure to the soil and water.

After 6 weeks of soil degradation PALF/Ep composites lost almost 2.37% of TM. On the other hand, in water degradation the composites lost only 0.81% of TM. From this it is clear that PALF/Ep based composites retained much of their TM during water degradation than soil degradation.

It was found that for PALF/Ep composites %Eb decreased slowly over time for water degradation but for soil degradation it decreased slowly over time. After 6 weeks of soil degradation PALF/Ep composites lost almost 9.93% of %Eb. On the other hand, in water degradation the composites lost only 2.18% of %Eb. From this it is clear that PALF/Ep based composites retained much of their %Eb during water degradation than soil degradation.

PALF/Ep composites impact strength increased slowly over time for water and soil degradation. After 6 weeks of soil degradation PALF/Ep composites gained almost 13.76% of impact. On the other hand, in water degradation the composites gained only 11.37% of impact. From this it is clear that PALF/Ep based composites retained much of their impact during water degradation than soil degradation.

PALF/Ep composites %wt loss increased slowly over time for water degradation but for soil degradation it was also increasing over time and this property was shown in figure 4. After 6 weeks of soil degradation PALF/Ep composites lost almost 1.03% of wt loss. Whereas in water degradation the composites lost only 0.54% of wt. From this it is clear that PALF/Ep based composites retained much of their %wt during water than soil degradation.

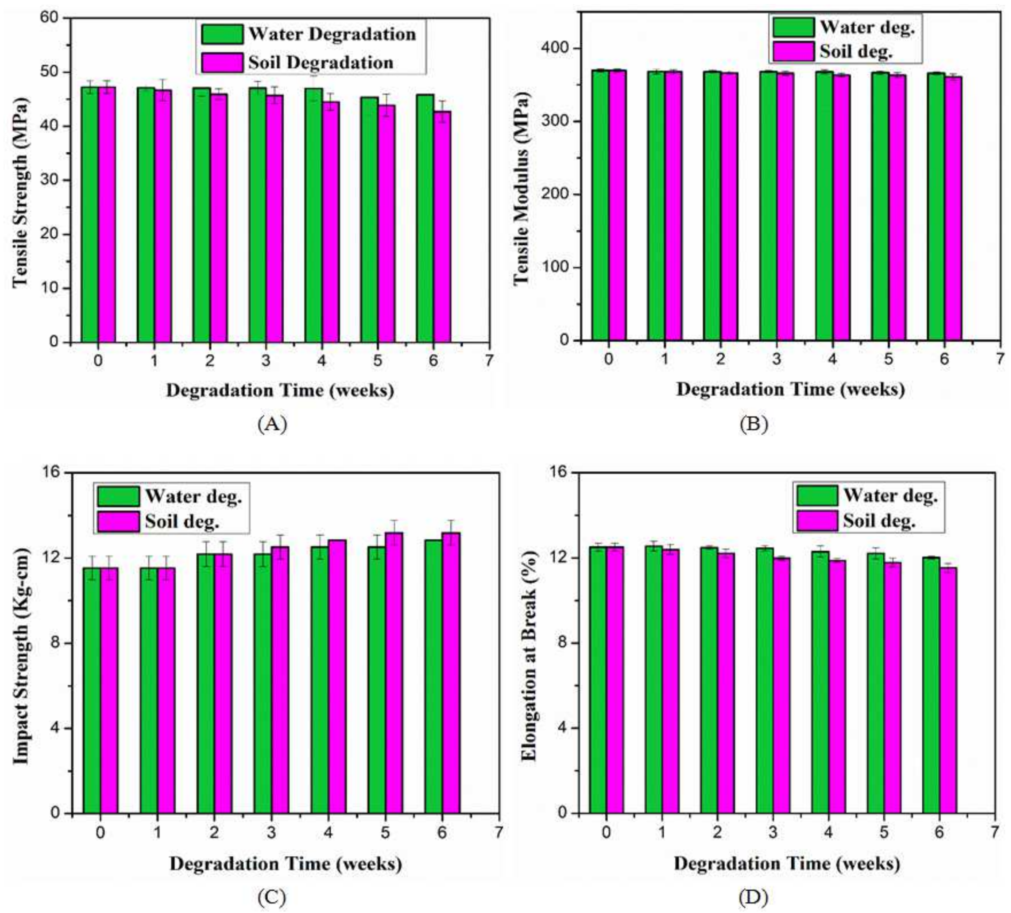

Figure 3. *(A) Change of tensile strength during degradation (B) Change of tensile modulus during degradation (C) Change of impact strength during degradation and (B) Change of percentage of elongation at break during degradation.*

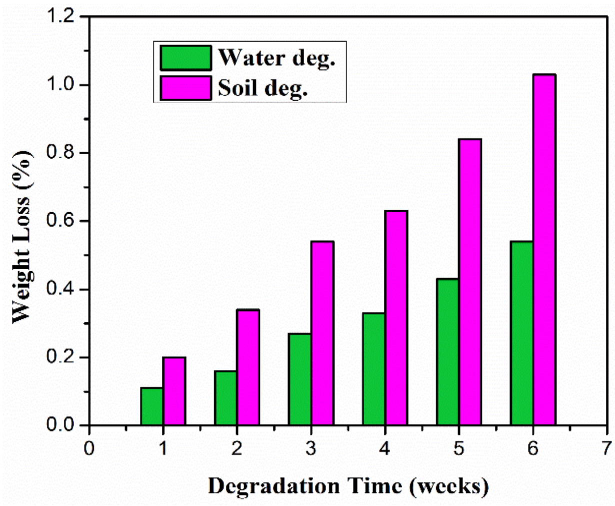

Figure 4. *Change of weight loss during degradation.*

4.4. Scanning Electron Microscopy (SEM) of PALF, Epoxy and PALF/Ep Composite

SEM image of PALF, Epoxy, PALF/Ep composite are illustrated in figure 5. Scanning electron microscopic images were taken to the fractured surface of the materials. From figure 5A it was evident that the fiber was straight and there were gap among the fiber but from figure 5B it could be said that there were some bubbles in the epoxy resin because during stirring with stirrer by hand air bubbles were formed in the epoxy. Scanning electron microscopy (SEM) investigation of the fractured surface of the PALF/Ep based composite was performed to study interfacial properties between fiber and epoxy matrix. From the SEM image of the factured sides of the composite was clear that there were considerable adhesion between the fiber and matrix. The image revealed that the fiber retains its structure inside the composites. This might be the reason for slightly better mechanical properties of PALF/Ep based composites.

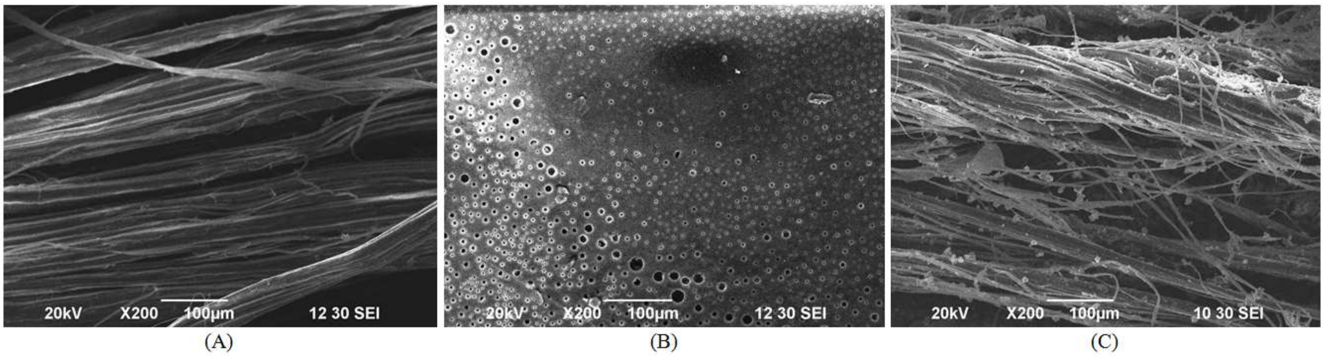

Figure 5. *SEM image of (A) PALF, (B) Epoxy and (C) PALF based epoxy composite.*

4.5. Mechanical Properties of Gamma Radiated Composites

The mechanical properties improved when the composites were irradiated with gamma radiation to some extent. In this research 2.5, 5.0, 7.5 and 10 KGy radiation dose were applied. PALF/Ep based composite up to certain doses of gamma radiation the TS increased and then decreased. The highest TS were obtained for 7.5 KGy radiation dose and this value was 28.5% higher than the normal composite materials. Up to certain doses of gamma radiation the TM values increased and then decreased. The highest TM value was obtained for 7.5 KGy radiation dose and this value was 41.92% higher than the normal composite materials. In case of PALF/Ep based composites for 2.5 KGy dose of radiation the %Eb values decreased. The %Eb values of 5.0KGy and 7.5KGy radiated samples were increased than 2.5 KGy radiated samples. The value of %Eb for 10 KGy further decreased. The tensile strength, tensile modulus, impact strength and elongation at break of irradiated composites were shown in figure 6.

Ionizing radiation such as gamma radiation is known to transfer energy to solid cellulose by Compton scattering and the rapid localization of energy within the molecules produced trapped macro-cellulosic radicals. This radical thus formed is responsible for the changed properties of the cellulose as well as the composite materials. The mechanism of the gamma radiation in the PALF fiber as well as the composite materials does not need any photo-initiator as gamma radiation itself is a high energy radiation. The proposed gamma radiation mechanism is illustrated below.

Initiation reaction:

$$RH \xrightarrow{\gamma-\text{radiation}} R^{\cdot} + H^{\cdot}$$

$$ROH \xrightarrow{\gamma-\text{radiation}} R^{\cdot} + OH^{\cdot}$$

Propagation:

$$R + O_2 \rightarrow ROO^{\cdot}$$

$$ROO^{\cdot} + RH \rightarrow ROOH + R^{\cdot}$$

$$ROOH \rightarrow RO^{\cdot} + OH^{\cdot}$$

$$2ROOH \rightarrow ROO^{\cdot} + RO^{\cdot} + H_2O$$

$$RO^{\cdot} + RH \rightarrow ROH + R^{\cdot}$$

$$OH^{\cdot} + RH \rightarrow R^{\cdot} + H_2O$$

Termination:

$$R^{\cdot} + R^{\cdot} \rightarrow R2$$

$$R^{\cdot} + ROO^{\cdot} \rightarrow ROOR$$

$$ROO^{\cdot} + ROO^{\cdot} \rightarrow ROOR + O_2$$

Here RH= PALF cellulose, $R^{\cdot}$= possible free radical formed by the abstraction of H or OH from PALF cellulose cleavage of C2-C3 bonds and chain scission of the cellulose backbone.

In case of PALF/Ep based composites up to certain doses of gamma radiation the impact strength increased and then decreased. The highest impact strength was obtained for 7.5 KGy radiation dose and this value was 26% higher than the non-radiated PALF/Ep based composites. The reasons of the changes of the impact were discussed in the previous section.

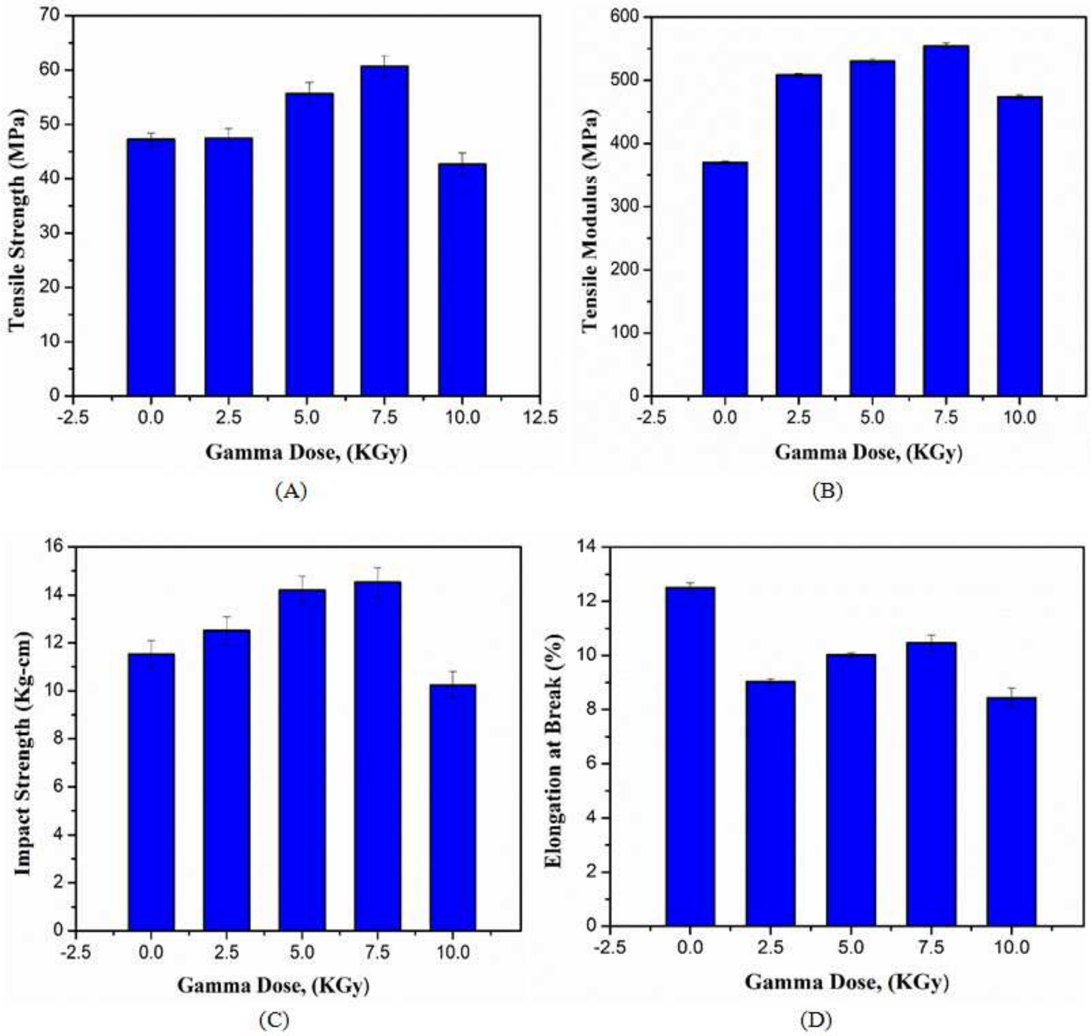

Figure 6. *(A) Change of tensile strength during irradiation, (B) Change of tensile modulus during irradiation, (C) Change of impact strength during irradiation and (D) Change of percentage of elongation at break during irradiation.*

5. Conclusion

The results of the present research work had evidenced that the composites fabricated by hand lay-up technique showed improved mechanical properties. That means this types of composites can be used for the construction materials. Based on this work the following conclusions may be drawn, for 30% PALF reinforced epoxy composite showed the highest tensile strength and elongation at break but the lowest tensile modulus. In water degradation test, PALF based epoxy composite retained much of its properties than soil degradation. When treated with gamma radiation, the PALF based epoxy composite showed better mechanical properties than the non-irradiated composites up to certain gamma dose but 7.5 KGy showed the best result. The thermogravimetric analysis showed the composites had a better thermal stability than the fiber. The surface morphology of these materials showed better adhesion of matrix and reinforced materials in the composites.

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