



Influence of Fish Scale Powder Filler on Physico-Mechanical Properties of Jute Fabric Reinforced Epoxy Resin Composites

Zakiya Yasmin^{*1}, G.M.Shafiur Rahman², Md. Asadul Hoque², Hrithita Aftab² and Md. Rezaul Karim Sheikh³

¹Institute of Environmental Science, University of Rajshahi, Rajshahi - 6205, Bangladesh.

²Dept. of Materials Science & Engineering, University of Rajshahi, Rajshahi - 6205, Bangladesh.

³Dept. of Applied Chemistry & Chemical Engineering, University of Rajshahi, Rajshahi - 6205, Bangladesh

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ABSTRACT

The aim of this work is to characterize and analyze the influence of fish scale powder on the physical, mechanical and morphological properties of jute fabric reinforced epoxy resin composites. Composites were fabricated by hand lay-up method for six different wt.% (0, 5, 10, 15, 20, 25 wt. %) of the fish scale powder (FSP) in jute epoxy composites (JEC) at normal temperature and pressure. The physico-mechanical properties of the “jute fabric reinforced epoxy composites” (JEC) and “fish scale powder filler added jute fabric reinforced epoxy composites” (FSP-JEC) were compared. The addition of the fish scale as filler enhanced the mechanical and thermal properties. The bulk density, water absorption and degradation of composites under soil were increased with the increase of filler loading. The tensile and flexural strength of the composite were increased with the increasing filler loading up to 15 wt.% and after that it decreased due to irregular distribution and agglomeration of filler in epoxy matrix. Surface morphology and the filler/ matrix debonding, poor interfacial adhesion, internal cracks and fibre pull-out were identified using scanning electron microscopy (SEM).

1. Introduction

Environmental pollution is one of the most focusing issues facing the modern world. With the increasing all over the world, environment pollution occurs rapidly. Every year hundreds millions tons of plastics and non- biodegradable products are produced from petroleum resources. So that with the increasing waste disposal, various problems create in environment day by day. As a result, the environment posing significant health risks to people, wild animal and the person's lifestyle would be impractical without them (Hornsby *et al.*, 1997; Oksman *et al.*, 2002; Oksman 2001). Researchers have recently become interested in natural fibre composites as a possible answer to these problems because they have advantages over other well-known materials. They are cheap, available, completely biodegradable, non-toxic, renewable, and

environmentally friendly (Mieck 1994). Jute is the most inexpensive natural material of all the natural fibres. It can be used instead of regular strands in many situations because it is stronger and has a higher modulus than plastic (Gowda *et al.* 1999).

Epoxy resins are frequently composed of low or high molecular weight prepolymers and at least two epoxide groups (Saba *et al.*, 2015). It is a thermosetting resin that can be cured with a variety of curing procedures and chemicals. Epoxy resins are frequently used as general purpose adhesives, particle - reinforced materials, encapsulating materials and high-performance coatings due to their superior mechanical properties, high adhesion to different substrates and excellent chemical and heat resistances (Jin Fan- Long, *et al.*, 2015).

Corresponding author.

E-mail address: zakiyayasmin65@gmail.com (Zakiya Yasmin)

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The outside extensions of a fish's body are its scales. Despite the fact that they can be turned into useful items, they are typically categorized as garbage. The skeletal elements known as scales cover and safeguard the fish skin. It is made up of a surface layer made of hydroxyapatite and calcium carbonate and a subsurface layer largely made of type I collagen. Ca, Mg, P, Na, and S are also present in trace amounts (Troncoso *et al.* 2013; Pati *et al.*, 2010). Fish consumption is rising globally on a daily basis. Therefore, a substantial amount of solid wastes, including as fish skin, bones, scales, and viscera, are produced in households, fish markets, and processing enterprises, which collectively account for between 50 and 70% (Kittiphattanabawon *et al.*, 2005). The environment will get more and more polluted if these wastes are not appropriately handled.

Bangladesh, which ranks fifth in the world in terms of aquaculture production, produced 4.38 million metric tons of fish annually from 2018 to 2019 (DoF. 2019).

In previous study, Gopi *et al.* (2016) developed fish scale reinforced vinyl ester composites using various reinforcing compositions. Thammahiwes *et al.*, (2017) produced wheat gluten-based bioplastics with fish scale through compression moulding. Abed *et al.*, (2021) used fish scales as a reinforcing component in polyester/polystyrene (PS) with a constant 1N load and temperatures of 20°C and 26°C to produce composite materials with varying weight fractions of 0, 6, 7, and 11%.

To the best of my knowledge, few literature studies have reported the improved mechanical performance of epoxy jute mat composites and no investigation has highlighted the enhance of mechanical properties of bio-composites by adding different loads of agricultural wastes especially fish scale. The aim of this research was to utilize the agricultural wastes available in Bangladesh and reduce the environmental pollution by these wastes.

2 Experimental

2.1 Materials and Methods

In this work, epoxy resin was used as a matrix purchased from Lucky acrylic and fibre, Dhaka, Bangladesh. Jute mat was used as a reinforcement and was purchased from local market, Rajshahi. Fish scale powder was used as a filler and was collected from local fish market, Rajshahi. Sodium hydroxide (NaOH) was used for the treatment of jute mat.

2.2 Treatment of Jute Mat

Jute mat was treated with 5% NaOH solution and was carried out for an hour at room temperature. After that, the jute mat was repeatedly rinsed with distilled water

to remove the NaOH solution and dried at 105°C for 24 hours (Neher *et al.*, 2020; Hai *et al.*, 2009).

2.3 Preparation of Fish Scale Powder

Fish scales were collected from local market, Rajshahi. The collected fish scales were cleaned by distilled water to remove dirty components and then it was dried in an oven at 50°C for 2 days. It was then broken into powder using an electric blender and sieved through a stainless steel sieve after being crushed and homogenized. The fine powders were finally placed in a plastic bag for additional analysis.

2.4 Fabrication of Composites

To prepare the composites hand lay-up method was used (Robel *et al.*, 2014; Stephano *et al.*, 2017). Before applying matrix material to the jute mat, epoxy resin, hardener and filler were thoroughly mixed up to obtain a homogeneous solution. Six types of composites were prepared in which the weight ratio of filler varied between 0%, 5%, 10%, 15%, 20% and 25% where the weight ratio of Jute fabric remained fixed at 10%. Composites were prepared by sandwiching one layer of JM between two layers of mix matrix solution. Two open molds were used for fabrication. A melot paper was placed on the bottom part of the mold. The mix material solution applied on the melot paper, then kept the jute mat on the mix matrix and used a roller to roll properly on the jute fabric. Again, applied the mix matrix on the layer of jute fabric. After that the mold was closed and kept for 24 hours at normal temperature and pressure. Finally, the composite was separated by removing of two steel plates, cut into desired size according to ASTM requirements and kept in the desiccators for future analyses.

2.5 Characterization

The bulk density of the composites was calculated using the formula below in accordance with ASTM C-134-76:

$D = W/V$, where D is the bulk density, W is weight of the sample and V is volume of the sample (<https://www.arpro.com>).

According to ASTM C- 67- 91, water absorption test was performed (Neher *et al.*, 2020). The sample's dimensions are 50 mm × 14.50 mm × 2.50 mm. After drying in an oven at 105°C for an hour, the samples were promptly weighed. The weighed samples were submerged in a 25°C static water bath for 24 hours. After that the samples were removed from the bath, cleansed with tissue paper and weighed. Let the initial weight be W_i and final weight be W_f . Percentage of

water absorption was calculated by the following formula:

$$\text{Water absorption (\%)} = \left[\frac{(W_f - W_i)}{W_i} \times 100\% \right]$$

According to ASTM D 5988-18, the biodegradability of the composites was measured using soil burial test in the laboratory (Goudar *et al.*, 2020). Fresh soil was provided by the garden on the Rajshahi University Campus in Rajshahi, Bangladesh. The dimensions of the sample are 50 mm × 14.50 mm 2.50 mm. To determine the initial dry weight (W_i), the samples were heated to approximately 40 °C. The soil samples were interred approximately 8 to 10 cm below the soil's surface. By sprinkling water onto the earth's surface, the soil's moisture content was maintained. After 20 days, the samples were extracted from the soil, cleansed, and heated in an oven prior to obtaining the weight (W_f). This was done to determine the sample weight loss. Soil decomposition was calculated using the following formula:

$$\text{Soil degradation (\%)} = \left[\frac{(W_i - W_f)}{W_i} \times 100\% \right]$$

According to ASTM D638, the tensile and flexural strength of the composites were measured using Universal Testing Machine (WDW-50, Serial No.-180067, China). The maximum speed, load range, and gauge length to be used in the machine are 10 mm/ mm, load limit of 500N and gauge length of 50 mm. Impact strength of the composites was measured using Universal Impact Tester (Model No. 7408, Hung Ta, Taiwan), according to ASTM D 6110-97(Khanam *et al.*, 2015). According to ASTM D 785, the hardness of the composites was measured using Rockwell Hardness Tester (HR- 150DT, China).

Melting and degradation rates of the composites were examined using a TGA thermal analyzer. Using nitrogen gas flow, the experiments were run from 30 to 700°C.

Scanning Electron Microscope (JEOL USER 7610 F, Japan) was used to observe the surface morphology of the composites.

3. Results and Discussion

3.1 Physical Properties of the Composites

3.1.1 Bulk Density of Jute Epoxy Composites at Different wt% of Fish Scale Powder Filler.

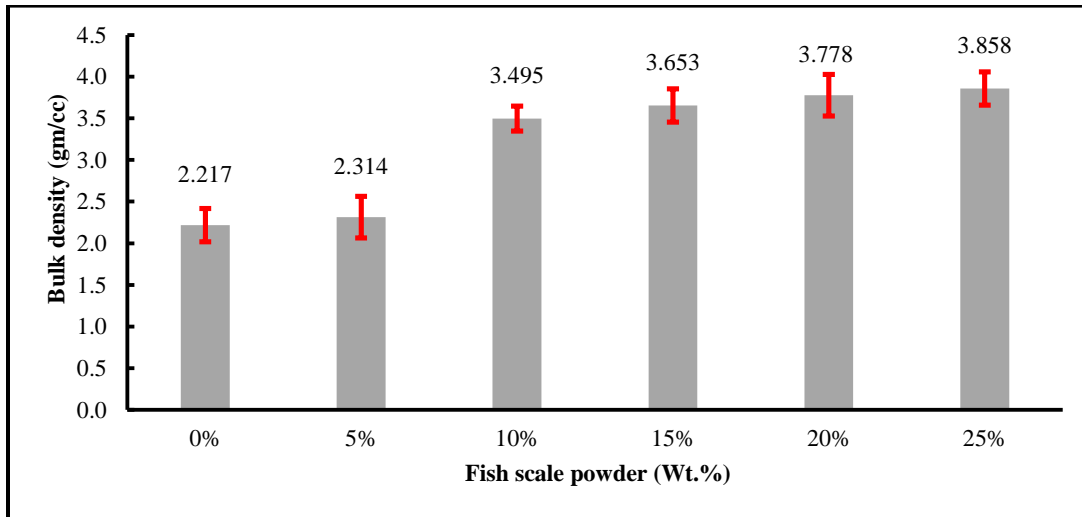


Figure 1. Impact of filler content on bulk density of fish scale powder- jute epoxy composites at different wt.%.

The bulk density of fish scale powder- jute epoxy composite is shown in figure 1. The figure showed that the bulk density increases with the increase of filler percentage and it increased up to 25 wt.% of filler content. It is seen that bulk density ranged from 2.217 to 3.858 gm/cc. The composite made with 25 wt.% fish scale powder filler has

the highest bulk density and the composite made without filler (only jute-epoxy) has the lowest bulk density. With the increase of fish scale powder filler percentage, the microscopic gap between matrix and filler become small, which is responsible for the increase of bulk density of the composites (Jahan. *et al.*, 2012).

3.1.2 Water Absorption of Fish Scale Powder– Jute Epoxy Composites at Various Soaking Time

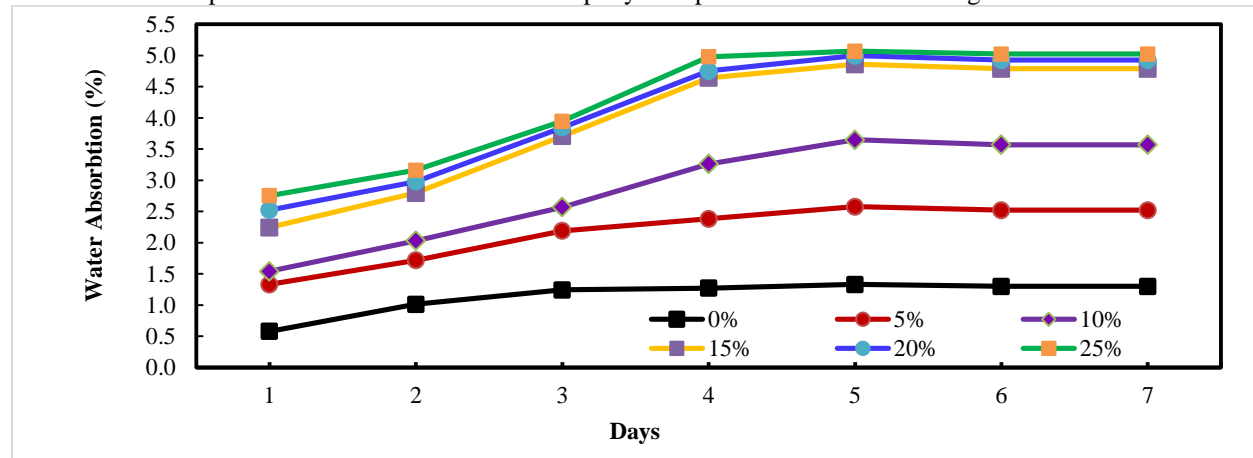


Figure 2. Water absorption of different composition of fish scale powder- jute epoxy composites as a function of time.

Water absorption of different composition of fish scale powder- jute epoxy composites as a function of time are shown in figure 2. According to the figure above, the percentage of water absorption increases as the filler loading increases. It is also observed that the water absorption of FSP- JEC become maximum within 5 days and after that it decreases or remain constant. The jute epoxy composite with 25 wt.% of filler has the

highest value which is 5.023% and the lowest value is found in the reference sample (0 wt.% filler) which is 1.30%. Due to the hydrophilic nature of jute fibre and fish scale powder, the value of water absorption was increased. On the other hand, epoxy resin is hydrophobic in nature, so JEC has the lower water absorption (Samson.*et. al.*, 2013; Abdel *et. al.*, 2011).

3.1.3 Degradation of Fish Scale Powder- Jute Epoxy Composites under Wet Soil

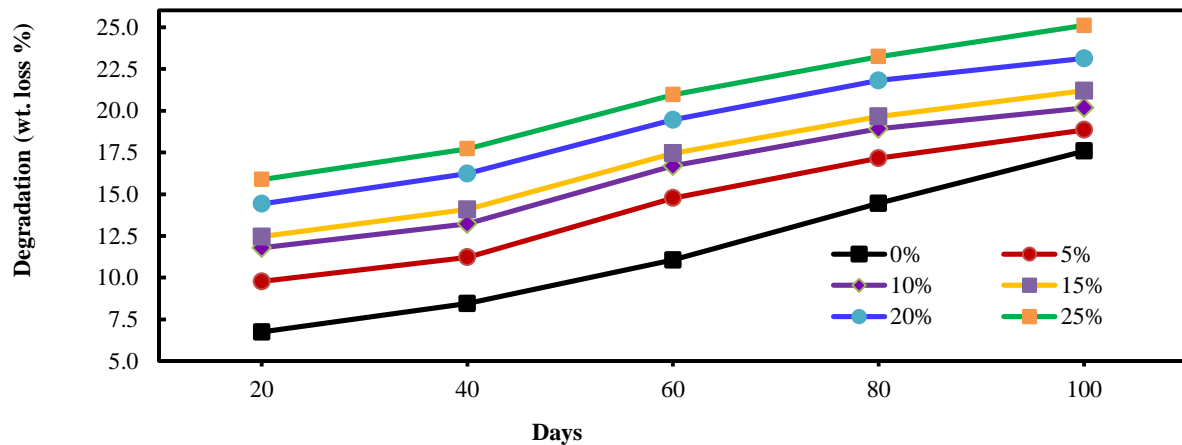


Figure 3. Degradation of different composition of fish scale powder- jute epoxy composites as a function of time under wet soil.

Degradation of JEC with different composition of fish scale powder under wet soil are shown in figure 3. The percentage of degradation (wt. loss) under wet soil increases up to 100 days. As shown in the figure, weight loss increases as the percentage of fish scale filler increases up to 100 days. The highest weight loss is 25.10 % for 25 wt. % fish scale filler for 100 days

and the lowest weight loss is 17.58% at 0 wt.% of filler for 100 days. We know that the jute fibre and fish scale powder are hydrophilic in nature and epoxy resin is hydrophobic. As a consequence of being submerged in the moist soil medium, water has infiltrated the fibre and filler edges of the composites, resulting in the composites' gradual degradation. In addition, because

microbial activity frequently assaults biodegradable fibres and fillers, bacterial action may also be

responsible for the loss of mechanical properties (Khan *et. al.*, 2010).

3.2 Mechanical Properties of the Composites

3.2.1 Rockwell Hardness of Fish Scale Powder- Jute Epoxy Composites

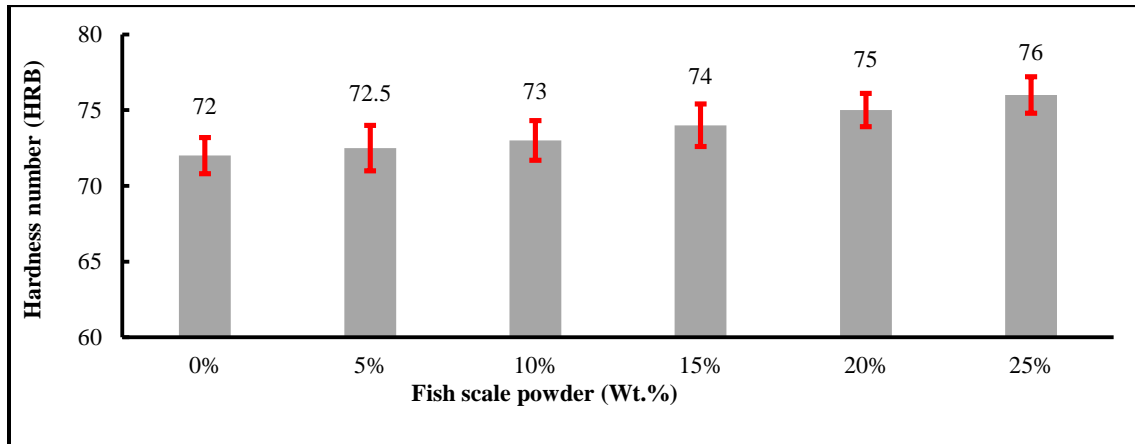


Figure 4. Impact of filler content on Rockwell hardness of fish scale powder- jute epoxy composites.

The Rockwell hardness of fish scale powder-jute epoxy composites at different wt.% are shown in figure .4. From the figure, it is clearly observed that the hardness of prepared composites slightly increases with the increase of filler loading. The highest value of hardness

is 76 (hardness number) at 25 wt.% of filler and the lowest value is 72 at 0 wt. % of filler. This happens when more filler is added, the composite becomes harder than pure ones (Subramonian *et. al.*, 2016; Cao *et. al.*, 2006).

3.2.2 Flexural and Tensile Strength of Fish Scale Powder- Jute Epoxy Composites

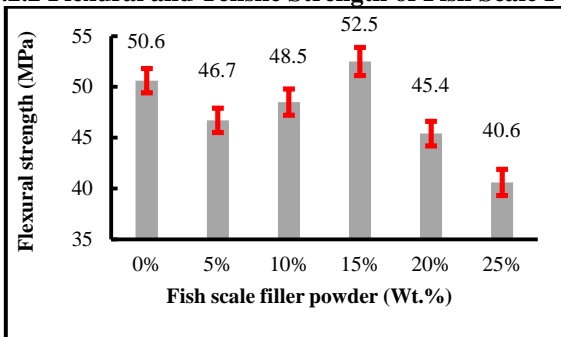


Figure 5(a). Impact of filler content on flexural strength of fish scale powder- jute epoxy composites.

Flexural and Tensile strength of jute epoxy composites with different wt. % of fish scale powder filler are shown in figures 5 (a) and 5 (b). From the figures, it is seen that the value of flexural and tensile strength increases with the increase of fish scale powder content from 5 to 15 wt.% and then decreases the flexural and tensile strength until further increase of filler content (20-25 wt.%). The maximum values of flexural and tensile strength are 52.5 MPa and 26.78 MPa at 15% filler content and the minimum values flexural and tensile strength are 40.6 MPa and 16.2 MPa at 25 wt. %

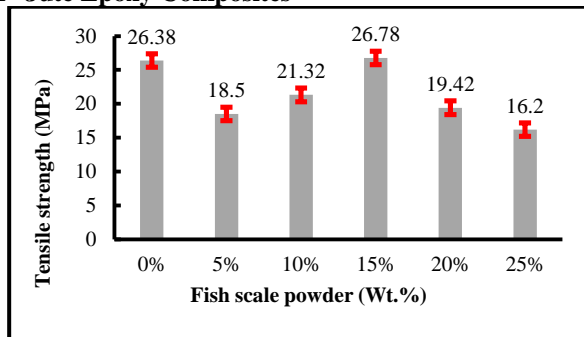


Figure 5(b). Impact of filler content on the tensile strength of Fish scale powder-jute epoxy composites.

filler content. The increase of flexural and tensile strength is due to the good interface and strong bonding between the filler and resin matrix until 15 wt. % of filler content, which is optimum. However, further increase of filler content, the value of flexural and tensile strength decreases. This is because at higher percentage of filler (20-25 wt.%), lower the amount of matrix and it also increases the void and imbalance the filler & matrix composition, which make the poor adhesion between filler and matrix in the composites

3.2.3 Elongation at Break of Fish Scale Powder-Jute Epoxy Composites.

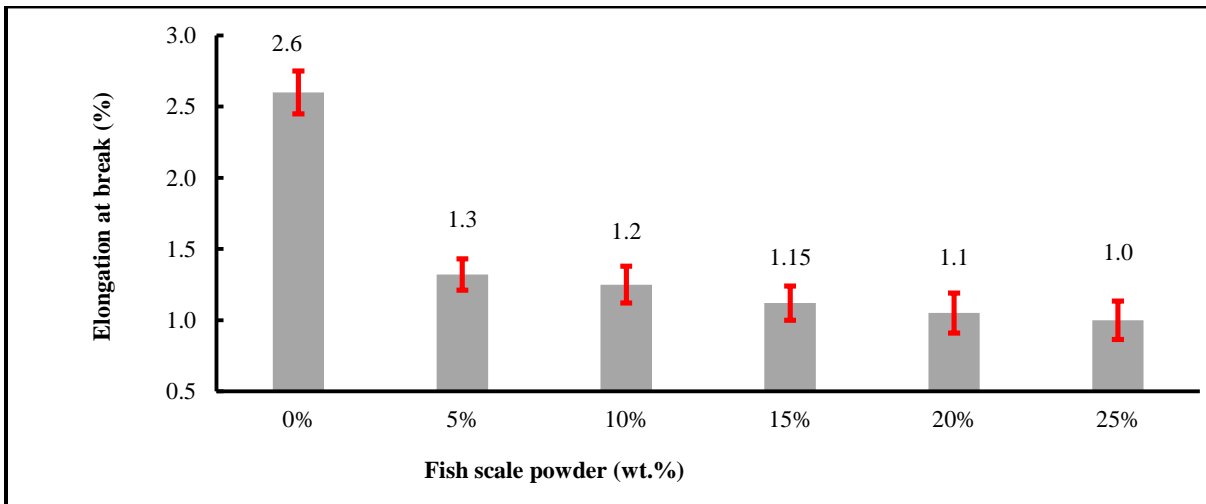


Figure 6. Impact of filler content on elongation at break of fish scale powder- jute epoxy composites.

The effect of the fish scale powder filler loading on the elongation at break of the prepared jute epoxy composites are shown in figure 6. From the graph, we see that the jute epoxy composite without filler reveals higher elongation at break than the corresponding value of fish scale powder- jute epoxy composites at any fish scale powder filler content. It is clearly seen that with the increase of filler content from 5 to 25 wt.% in the composite's composition, the elongation at break gradually decreases. The elongation at break at 5 wt.%,

10 wt.%, 15 wt.%, 20 wt.% and 25 wt.% of filler contents are 1.3%, 1.2%, 1.15%, 1.1% and 1.0% respectively. The elongation at break of jute epoxy composite without filler is 2.6%. The filler's adherence to the composite matrix, which results in the stiffening of the composite materials and, as a result, resistance to stretching when the strain is applied, is one explanation for a decrease in elongation at break (Ekebafe *et. al.*, 2010).

3.2.4 Tensile Modulus of Fish Scale Powder-Jute Epoxy Composites

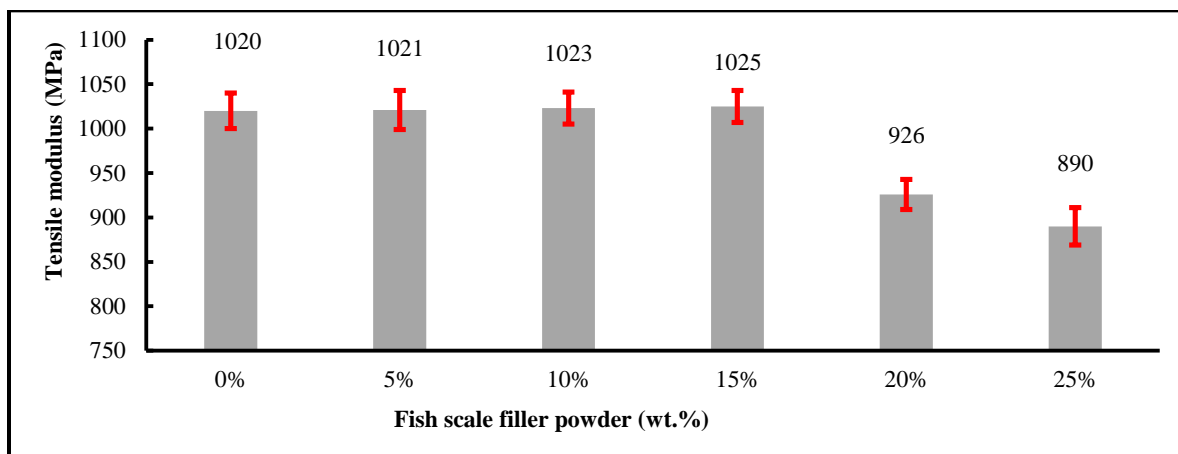


Figure 7. Impact of filler content on tensile modulus of fish scale powder-jute epoxy composites.

Tensile modulus of jute epoxy composites at different wt.% fish scale powder filler are shown in figure 7.

From the graph it is seen that with the increase of fish scale powder filler loading from 5 wt.% to 15 wt.%, the

tensile modulus of jute epoxy composites increases very slowly. And after that with the increase of fish scale filler loading from 20 wt.% to 25 wt.% the value of tensile modulus decreases rapidly. The highest tensile modulus is 1025 MPa for composites containing 15 wt.% fish scale powder filler. This disclosed the fact that up to 15 wt.% filler content, the filler and the

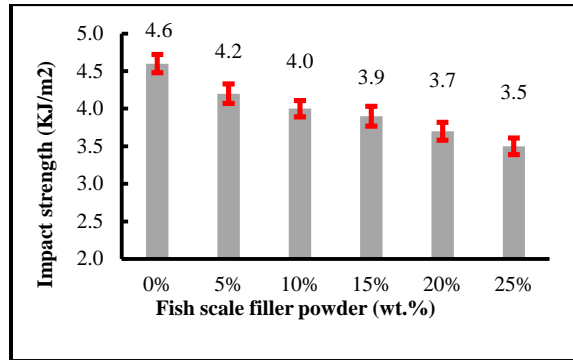


Figure 8. Effect of filler content on the impact strength of fish scale powder-jute epoxy composites.

3.2.5 Impact Strength of Fish Scale Powder-Jute Epoxy Composites

The impact strength of jute epoxy composites with different percentage of fish scale powder filler are shown in above figure 8. From the figure it is seen that the impact strength of composites slightly decreases with the increase of fish scale powder filler content. This is primarily the result of the composite material's decreased elasticity (Bose *et al.*, 2004) due to the inclusion of filler, which also reduces the matrix's deformability. As infill concentration increases, impact strength decreases because the matrix becomes less able to absorb energy and consequently less robust. (Raju *et al.*, 2012) also reported that the poor interfacial adhesion between hydrophobic matrix and hydrophilic nature of filler usually results in decrease toughness.

3.2.7 Morphological Observation of Jute Epoxy Composite (JEC) and Fish Scale Powder- Jute Epoxy Composite (FSP-JEC) by Scanning Electron Microscope (SEM)

polymer are well distributed in the composites. So that, the stiffness of the FS-JEC is increased with the increase in filler content percentage until 15 wt.%. At higher filler content (20-25 wt.%), the filler and matrix become irregular distribution in composites, which may be the cause of reduction of tensile modulus of composites (Jahan *et al.*, 2012).

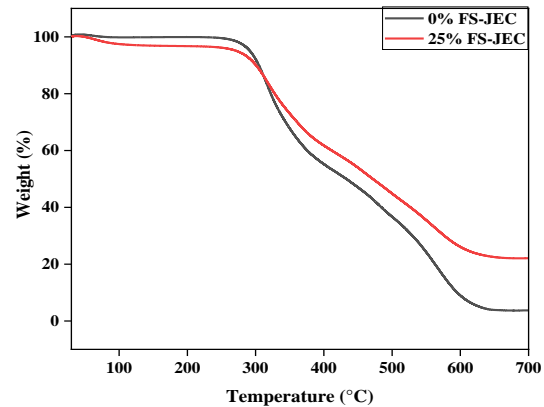


Figure 9. Thermogravimetric Analysis of JEC and FSP- JEC.

3.2.6 Thermogravimetric Analysis of Jute Epoxy Composite (JEC) and Fish Scale Powder - Jute Epoxy Composite (FSP- JEC)

From the above figure 9, it is observed that primarily weight losses of the composites are due to the removal of moisture at the temperatures of around 100°C.

The jute epoxy composites showed the first set of degradation at around 275°C and continued up to 600°C and then after weight loss remained constant with the residual weight of 5wt.% at the temperature of 630°C and above. On the other hand, fish scale powder - jute epoxy composites showed the first set of degradation at around 300°C and continued until at around 610°C and then after weight loss remained constant with the residual weight of 18 wt.% at the temperature of 680°C and above (Polat *et al.*, 2013).

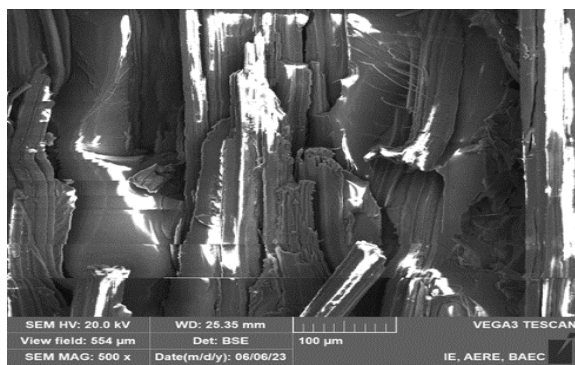


Figure 10(a). SEM image of JEC magnification at 500X magnification

The magnifying images by SEM of jute epoxy composite and 15 wt.% of fish scale powder - jute epoxy composite are shown in figure 10 (a) and 10 (b). From the figure 10 (a), fibre fracture, fibre pullout and voids on the fractured surface of the composites were observed. The plausible reason may be due to the weak interfacial adhesion between jute and polymer matrix. Fibre pullout, voids and fibre agglomeration are the main reason to decrease the mechanical strength of JEC. On the other hand, fibre pullout was also observed at figure 10 (b). But in this case, fish scale powder-jute epoxy composite pullout was observed as individual fibre. This major alteration in morphology improved the mechanical bonds between the fibre, filler, and polymer matrix. According to figures 10(a) and 10(b), jute fabric, fish scale powder, and epoxy matrix had stronger reinforcement matrix adhesion than jute epoxy composites. This could be the cause of the composites made from fish scale powder and jute epoxy's superior mechanical characteristics and smoother surface (Shah *et. al.*, 2012; Majid *et. al.*, 2018).

4. Conclusion

Jute fabric-reinforced epoxy composites (JEC) and fish scale powder (FSP) filled jute fabric-reinforced epoxy composites (FSP-JEC) were fabricated. The physical, mechanical, thermal, and morphological properties of fish scale powder-jute epoxy composites (FSP- JEC) were studied. The highest tensile strength, flexural strength, and tensile modulus of the composites were found at the 15 wt.% filler loading. The percentage of elongation and impact strength of the composites decreased with the increase of filler loading. In both cases the highest values were observed at 0wt.% loading of filler in FSP-JEC. The Rockwell hardness of the composites increased with the increase of filler loading and the highest value was observed at 25 wt.% of the composites. The bulk density, water absorption, and percentage of degradation under wet soil of the

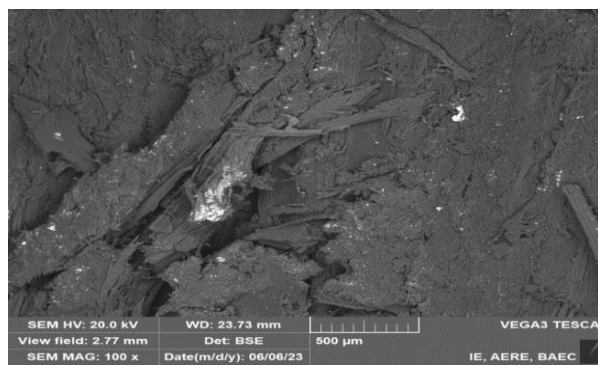


Figure 10 (b). SEM image of FSP-JEC at 100X

composites increased with the increase of filler loading, and the highest value was observed at 25 wt.% of the composites. The thermal stability of the composites was measured with TGA analysis and FSP- JEC showed higher thermal stability than JEC. The cross-sectional views of the fractured surface of prepared composites were analyzed by SEM. The microscopic analysis supports the homogeneous and smooth formation of the composites.

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