

**EFFECT OF HEAT AND CHEMICAL TREATMENT ON  
MECHANICAL, THERMAL AND WATER ABSORPTION  
BEHAVIOR OF JUTE FIBER REINFORCED HIGH  
DENSITY POLYETHYLENE COMPOSITES**

A DISSERTATION

Submitted in partial fulfillment of the requirements for the award of

**INTEGRATED DUAL DEGREE**

(Bachelor of Technology and Master of Technology)

In

**CHEMICAL ENGINEERING**

(With specialization in Hydrocarbon Engineering)

By

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**May 2016**



**DEPARTMENT OF CHEMICAL ENGINEERING  
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**CANDIDATE'S DECLARATION**

I hereby declare that the report titled “**Effect of Heat and Chemical treatment on mechanical, thermal and water absorption behavior of Jute Fiber Reinforced High Density Polyethylene Composites**” in partial fulfillment of the requirements for the award of degree Integrated Dual Degree (Bachelor of Technology and Master of Technology) with specialization in Hydrocarbon Engineering and submitted to the Department of Chemical Engineering of Indian Institute of Technology, Roorkee is an authentic record of my work under the supervision of **Professor Shishir Sinha**.

The matter presented in this report has not been submitted by me for the award of any other degree of this institution or any other institution.

Date: May 6, 2016

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**CERTIFICATE**

This is to certify that the above statement made by the candidate is correct to the best of my knowledge.

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## ABSTRACT

Jute fiber/High density polyethylene (HDPE) composites were prepared as sandwiches of woven Jute mats and HDPE sheets. Composites fabricated using two Jute mats yielded best results: Tensile strength, Flexural Strength, Impact strength, and Water resistance. Furthermore, the effect of heat, alkali, and silane treatment on fiber was investigated. The results showed that treatments enhance mechanical properties and thermal stability considerably. Alkali and Silane treatment led to improved water resistance. Crystallite size was found to increase with treatment. Results obtained were found to concur with Fourier Transform Infrared Spectroscopy (FTIR) analysis of Jute fiber both untreated and treated (various treatments). Results were further confirmed by the morphological study of the fractured surface of composite after tensile tests.

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## NOMENCLATURE

NFBC	Natural Fiber reinforced Polymer Composite
FTIR	Fourier Transform Infrared Spectroscopy
XRD	X-ray Diffraction
SEM	Scanning Electron Microscopy
HDPE	High Density Polyethylene
TGA	Thermal Gravimetric Analysis
DTG	Derivative Thermogravimetric Analysis
DTA	Differential Thermal Analysis



# CHAPTER 1. INTRODUCTION

---

## 1.1 Brief description

An appreciable amount of attention is being given to produce composites that are eco-friendly and economically produced. There is no denying that artificial fibers boast of relatively much better mechanical and thermal properties, however, owing to the environmental concerns associated with these fibers, research interest is getting diverted to natural fibers. Artificial fibers have various problems associated with them: as they are difficult to decompose, reclamation processing leads to a myriad amount of environmental load; it's extremely difficult to conduct appropriate disposal processing; for example incineration of discarded glass fibers leads to release of lot of smoke and in many cases, causes damage to incinerator because of the fusion by glass fibers. In order to alleviate these environmental issues while maximizing the desirable properties, natural fibers are being employed increasingly to prepare composites that have numerous advantages over artificial fibers like high availability, renewability, low density, biodegradability, less abrasiveness and low cost [1].

## 1.2 Literature review

Various natural fibers like jute, sisal, hemp, banana, bamboo, oil palm fibers, etc are used as reinforcement for polymer matrix composite. Jute is particularly interesting as it's grown in plenty in India and the composite made using Jute fiber have moderate tensile and flexural properties in comparison to those of others.

Hassan et al [2] reported that the demerit with jute fiber is in that, it's extremely hydrophilic and exhibits very poor tendency for associating with the hydrophobic polymer matrix. This makes it necessary to carry out treatments in order to improve the desired properties in the final composite.

Heat treatment has been seen to give good improvement in certain cases, Rong et al reported 37% improvement in properties of sisal fibre strength [3]. Three types of changes, we can expect in fibre because of heat treatment: surface energy increase, removal of moisture and changes in microstructure of the fibres.

Prasad et al. [4] showed that non-polar surface energies of hemp fibre which were heat-treated at 60 and 80°C were higher in comparison to those of the untreated ones. This implies a decrease in hydroxyl groups on fiber surface, thus decreased hydrophilicity and polarity. Removal of moisture is also very effective in improvement of the interfacial bonding matrix as water on the fiber surface tends to separate fiber and matrix [5].

Just opposite to this school of thought, removal of moisture may be seen decreasing the effectiveness of fiber as reinforcement by making it brittle, so the net effect is result of cumulative action of these abovementioned behaviors [6]. At high temperatures, natural fibres tend to undergo degradation leading to an adverse impact on the properties. High moisture absorption and lack of strong adhesion to polymer matrix are the major drawbacks of natural fibers. Fiber matrix adhesion is severely influenced on water absorption, acting as a plasticizer which results in very poor transfer efficiency, thus reduced mechanical properties.

Sudha et al [7] found From Box and Benkhen experimental design, the optimum conditions for improving water resistance through alkali treatment for Jute fibers is 4 h, 30 °C and 5% concentration.

Dash et al [8] reported that delignification results in much better adhesion between polymer and fiber matrix. Treatment of natural fibers with alkali results in the disruption of hydrogen bonding and removal of impurities, lignin, hemicelluloses etc; yielding better adhesion in composite [9,10].

Silanes are known for their sound efficiency as coupling agents. The bifunctional structures of silanes makes find their applications in natural fiber/polymer composites (NFPC), as natural fibers have reactive hydroxyl groups, and considerable work has been done to study the various silanes for NFPC production [11].

### 1.3 Objectives

- To prepare composite using HDPE and Jute fiber, both untreated and after continual treatments of Heat, Alkali and Silane.
- To investigate the water absorption behavior of composite.
- To study the mechanical properties of composite before and after water absorption.
- To carry out Thermogravimetric analysis of untreated and treated jute fiber in order to check the thermal behavior of composite.

## CHAPTER 2. Materials used and Methodology

---

### 2.1 Materials

The Jute fiber was procured from shops in nearby local market. It comprises of 60-63% Alphacellulose, 12-13% lignin, 21-24% hemicellulose, 0.2-1.5% pectin, 0.7-1.2% ash, and remaining other constituents like fats, waxes, nitrogenous material etc [12]. The thermoplastic High density polyethylene (HDPE), serves the role of matrix, and was collected from M/s Rapid Engineering Company Pvt Ltd, India. It had a melt flow index (MFI) of 19-21g/10 min (190°C/2.16 kg) and specific gravity of 0.923. The coupling agent Silane A174, [3-(Methacryloyloxy)propyl]trimethoxysilane, was obtained from M/s TCI Chemicals (India) Pvt Ltd. Sodium hydroxide (NaOH) and Ethanol used for treatment of fiber were bought from Merck (India).

## 2.2 Treatment of Jute Fiber

### 2.2.1. Untreated Jute Fiber

Chopped Jute fibers were cleaned using distilled water for removal of dirt and impurities. Jute fiber mats of deemed sizes were then heated in an oven at 50°C for 5 hours to remove moisture.

### 2.2.2. Heat Treatment

Untreated Jute fibre mats of deemed sizes were heated in an oven at 80°C for 6 hours to remove moisture and other impurities. Precautions were taken to ensure minimum moisture absorption by keeping them in zip pouches.

### 2.3.3 Alkali Treatment

From Box and Benkhen experimental design, the optimum conditions for alkali treatment for Jute fibers is 4 h, 30 °C and 5% concentration [7]. The liquor to fiber ratio was maintained as 10. After treatment as per the conditions, heat treated fibers were washed with tap water and later on using distilled water till the pH level came close to 7. This was followed by oven drying at 50°C for 5 hours.

### 2.2.4. Silane Treatment

3-methacryloxy-propyl-trimethoxy-silane was employed to treat jute fibers obtained after heat and alkali treatment. Jute Fabric was kept in the solution of ethanol and water (50% volumewise) and having 1% silane concentration for 2 hours and later on washed with ethanol and dried using hot air oven at 50 °C for 5 hours.

### 2.3 Preparation of Composites

Composite was prepared using Jute fiber woven mats and HDPE sheets. HDPE sheets were prepared as per the calculations maintaining net weight of composite as 110g. Jute Fiber mat and Polymer HDPE sheet have been mentioned as J and P respectively in Figure 2.3.1 which shows different composite sandwiches.

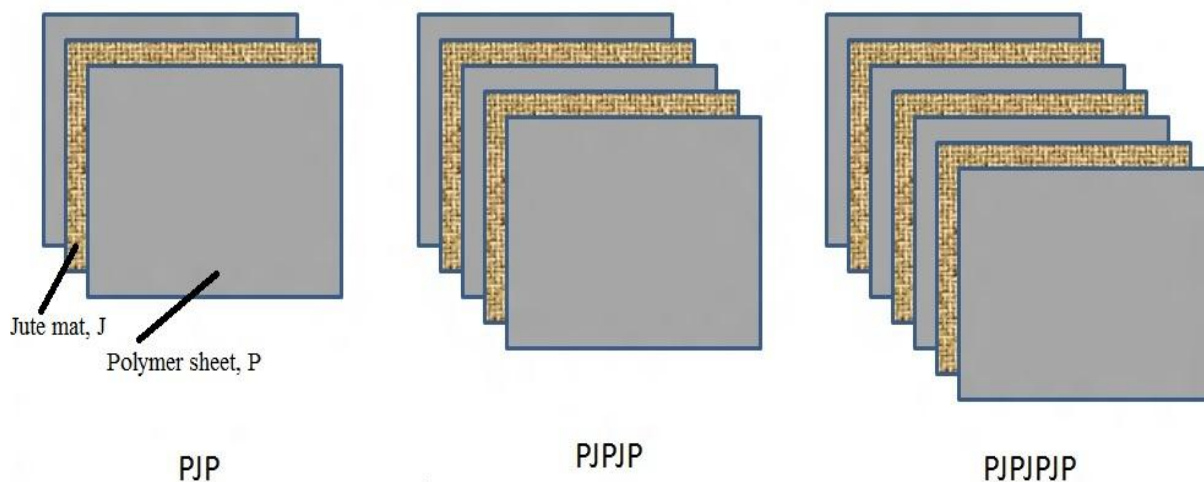


Fig. 2.3.1. Different Composite Sandwiches

After various treatments of Jute fibers, weight loss was observed as tabulated in Table 2.3.1. Appreciable amount of weight loss was observed after alkali treatment.

Table.2.3.1. Weight loss after various treatments of Jute fiber.

Treatment	Weight Loss
Heat	1.5%
Heat+Alkali	16%
Heat+Alkali+Silane	1%

Corresponding to different weight losses after the respective treatment, HDPE sheets of appropriate weight as shown in Table 2.3.2 were prepared to maintain the net weight as 110g of whole sandwich setup.

Table.2.3.2. Weight of single HDPE sheet used in the composite

	Untreated	Heat Treated	Alkali Treated	Silane Treated
PJP	46	46.25	47.625	48.09
PJPJP	24.67	25.00	26.83	27.45
PJPJPJP	14.00	14.38	16.44	17.14

Compression molding machine was employed at 170-180°C and 20 MPa for 20 minutes. Later on, the sample was left to cool at room conditions.



## 2.4 Characterization

### 2.4.1. Fourier Transform Infrared (FTIR) Spectroscopy

FTIR spectrometer (Nicolet 6700 series) was used to examine FTIR spectra for Jute fiber over 4,000-600  $\text{cm}^{-1}$  having a resolution of 4  $\text{cm}^{-1}$  for employing Potassium bromide (KBr) as the base material.

### 2.4.2. Scanning Electron Microscopy (SEM)

Surface morphology was investigated using scanning electron microscope (Model LEO-435VP) having the acceleration voltage of 0-30 kV was employed. Stretched out surfaces of composites after carrying out the tensile test were also examined. A thin layer of gold was coated over the sample for SEM.

### 2.4.3. Mechanical Testing

Tensile test was done according to the ASTM D3039 [15] employing universal testing machine (Instron Model 5982) with crosshead speed as 2 mm/min. For testing Flexural strength, ASTM D790 [16] was used at same crosshead speed. Impact testing machine (TINIUS OLSEN Model impact 104); the hammer weighing 4 kg was used. The Izod impact test was done in accordance with ASTM D256 [17]. All these tests were also conducted for composite sheets after water absorption keeping them submerged in water for 15 days.

### 2.4.4. Water Absorption

The water absorption for composite materials was done as per ASTM D570-98 [13]. Oven drying of samples was done at 50 °C, followed by dipping in distilled water. Samples were weighed after wiping in regular intervals. The percentage of water absorbed was obtained as [14].

$$\text{Water absorption (\%)} = \frac{\text{Weight of wet sample} - \text{Weight of dry sample}}{\text{Weight of dry sample}} \times 100$$

### 2.4.5. Thermogravimetric analysis (TGA)

TGA using EXSTAR TG/DTA 6300 equipment was done for jute fiber. Samples weighing 10 mg were heated steadily at 10°C/min from ambient temperature to 800°C under the nitrogen atmosphere.

#### 2.4.6. X-ray diffraction (XRD)

X-ray diffraction (XRD) was employed to examine crystallinity of the composites using Brucker AXS D8 diffractometer operating with Cu-K $\alpha$  radiation ( $\lambda = 1.5406 \text{ \AA}$ ) at 40 kV and 30 mA in the range of 10 to 50 $^\circ$  with a scan speed of 0.02 $^\circ$ /s. The crystalline thickness ( $\tau$ ) was obtained using Scherrer's equation:

$$\tau = \frac{K\lambda}{\beta \cos\theta}$$

Where K is Scherrer's constant generally 0.89,  $\lambda$  is wavelength for X-ray radiation (1.5406  $\text{\AA}$  for Copper),  $\beta$  being full width at half maximum of diffraction peak in radian and  $\theta$  is Bragg angle.

## CHAPTER 3. Results and Discussion

### 3.1. Fourier Infrared Spectroscopy Analysis

FTIR spectra for untreated, Heat treated, Heat+Alkali treated and Heat+Alkali+Silane treated Jute fibers are shown in Figure 3.1.1. In the region  $3600\text{-}3095\text{ cm}^{-1}$ , a strong absorbance band can be easily observed corresponding to the O-H group, hydroxyl of

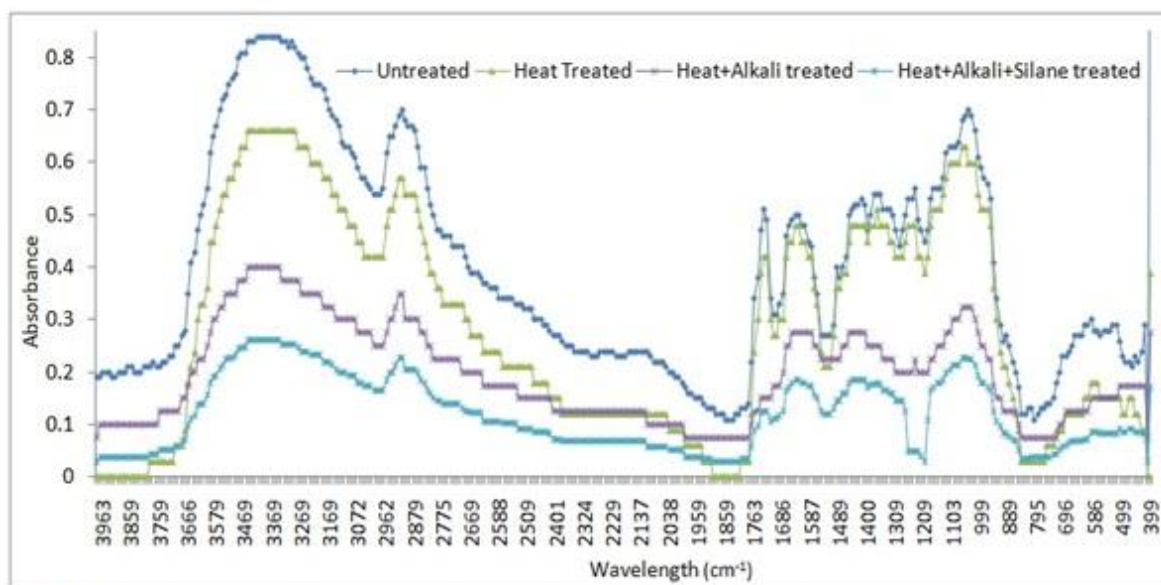


Fig.3.1.1. FTIR spectra for untreated and different treated Jute Fiber

cellulose and hemicellulose components [18]. Not much difference is visible between the spectra of the untreated and the heat treated jute fiber, this implies almost no changes have occurred after heat treatment, however, the band near  $3,410\text{ cm}^{-1}$  visibly declines after alkali treated owing to removal of hemicellulose. The absorbance band at  $1,735\text{ cm}^{-1}$  corresponding to the C=O (carbonyl) functional group of hemicelluloses and pectin is observed for the untreated and heat treated fibers, though it's not seen after alkali treatment. The disappearance of the peak can be attributed to the fact that the alkali treatment appreciably removes the hemicelluloses content, pectin and wax. The band at  $1,350\text{ cm}^{-1}$ , which corresponds to the -CH- of cellulose, and hemicelluloses, was observed for untreated fiber, but almost vanished after alkali treatment. The elimination of the peak again confirms that the alkali treatment partially eradicates hemicellulose components. The change in the peak near  $1,230\text{ cm}^{-1}$  is attributed to the C-O stretching acetyl group of

lignin, indicating partial removal of lignin content on alkali treatment. The band at  $1,130\text{cm}^{-1}$  corresponding to the C-O-C stretching of polysaccharide components in cellulose implies appreciable reduction. Degradation of cellulose components is responsible for this observation. The clearly visible trough at  $1200\text{cm}^{-1}$  in the spectra for fiber after silane treatment corresponds to Si-O-C. Band at  $700\text{cm}^{-1}$  and a shoulder at  $738\text{cm}^{-1}$  in the same spectra are associated with Si-O-C bonds.

Scanning electron micrographs (SEM) of untreated jute and after continual treatments i.e. heat, alkali and silane are shown in Figure 3.1.2. From the images, we can certainly come to the conclusion that the surface morphology has changed on different treatments.

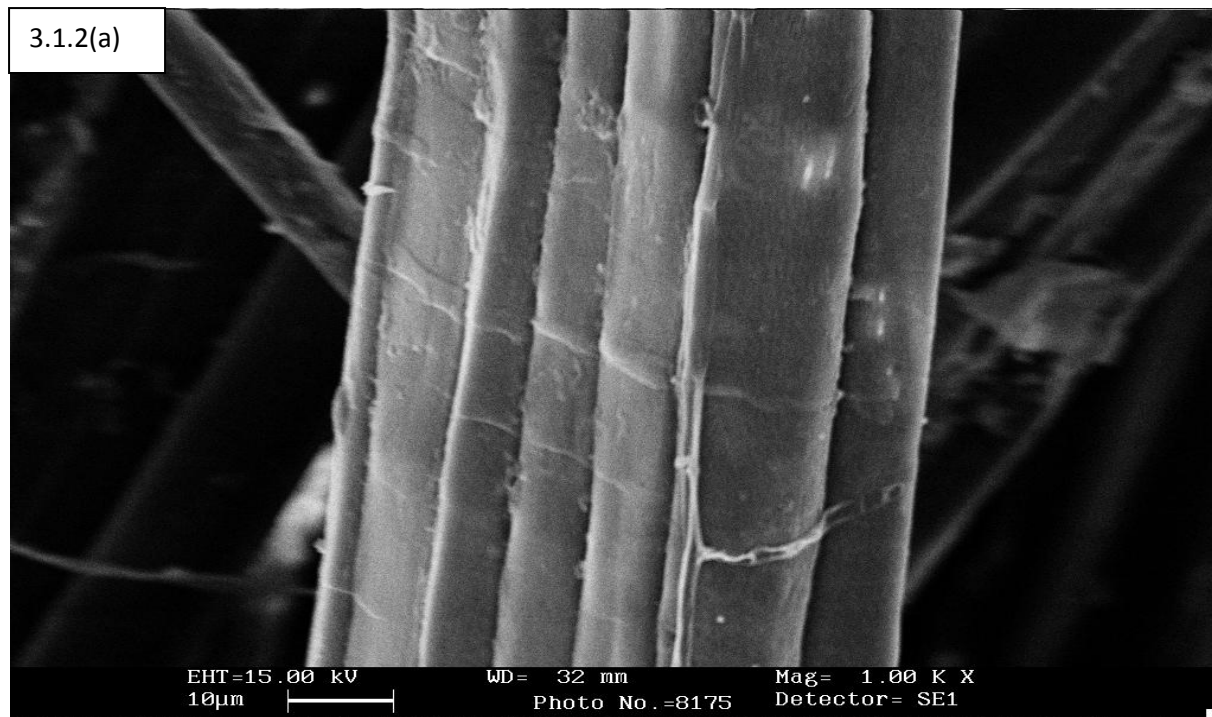


Fig.3.1.2. (a) SEM micrographs Untreated Jute fiber

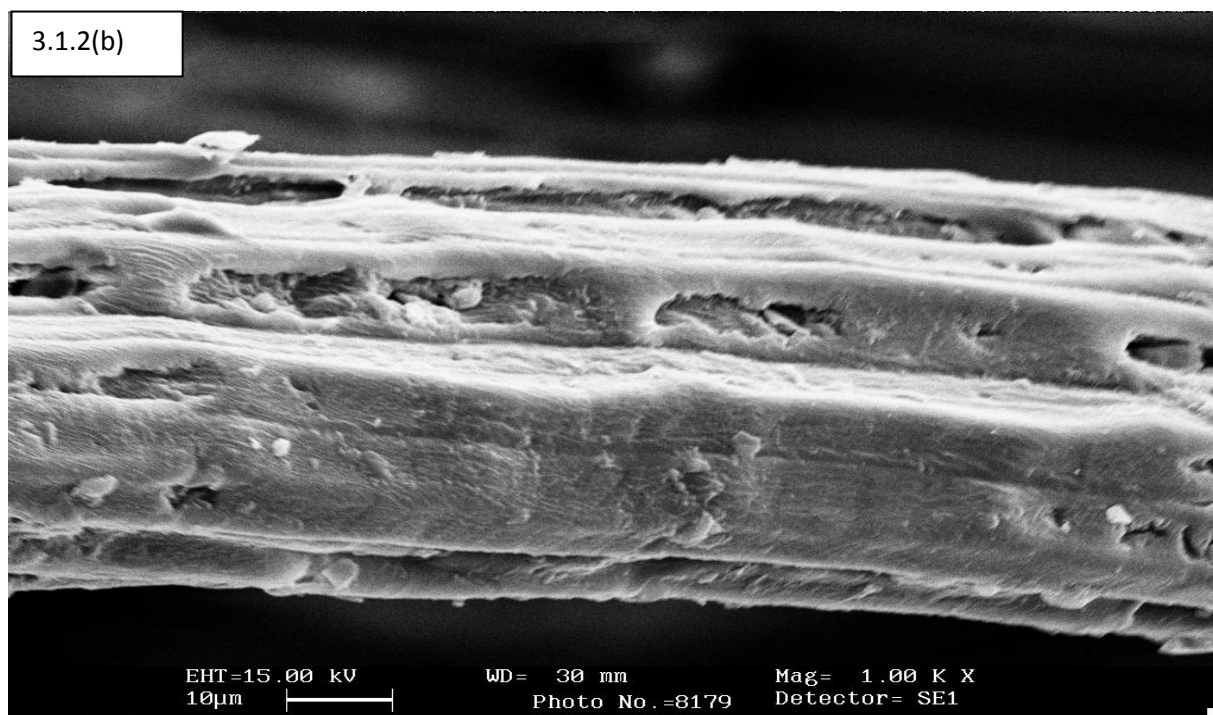


Fig.3.1.2. (a) SEM micrographs Heat treated Jute fiber

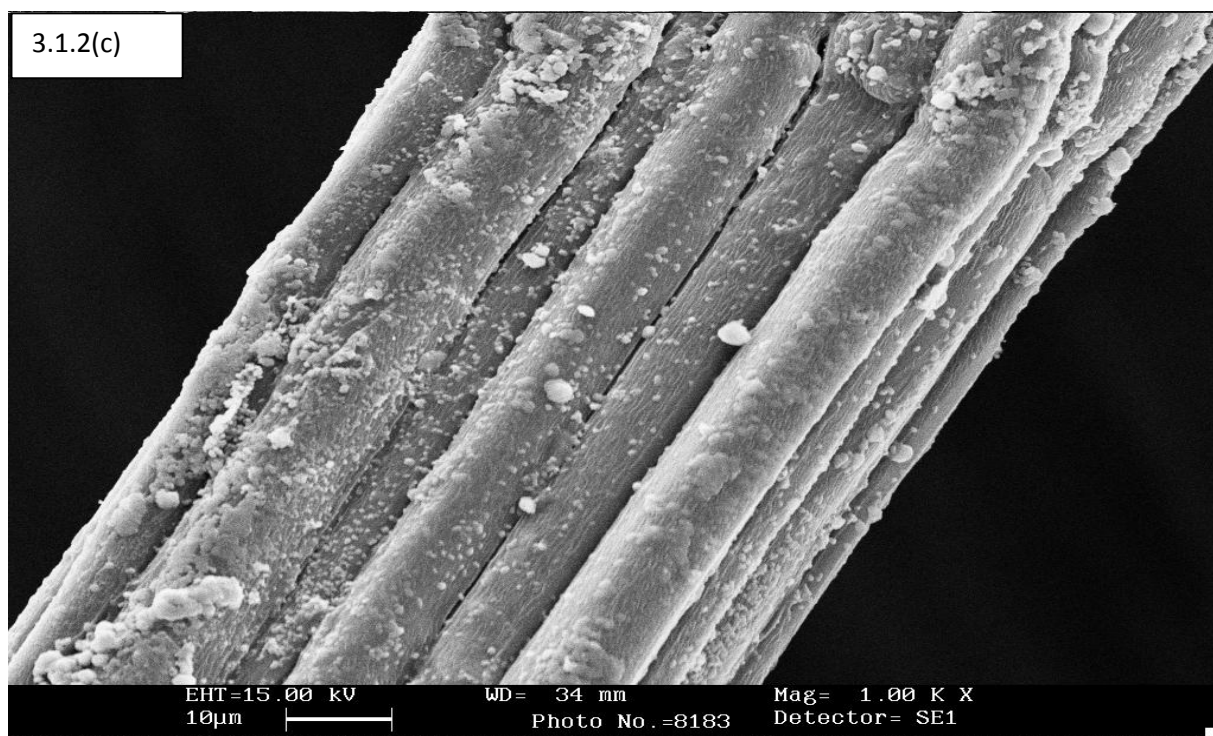


Fig.3.1.2. (a) SEM micrographs Heat+Alkali treated Jute fiber

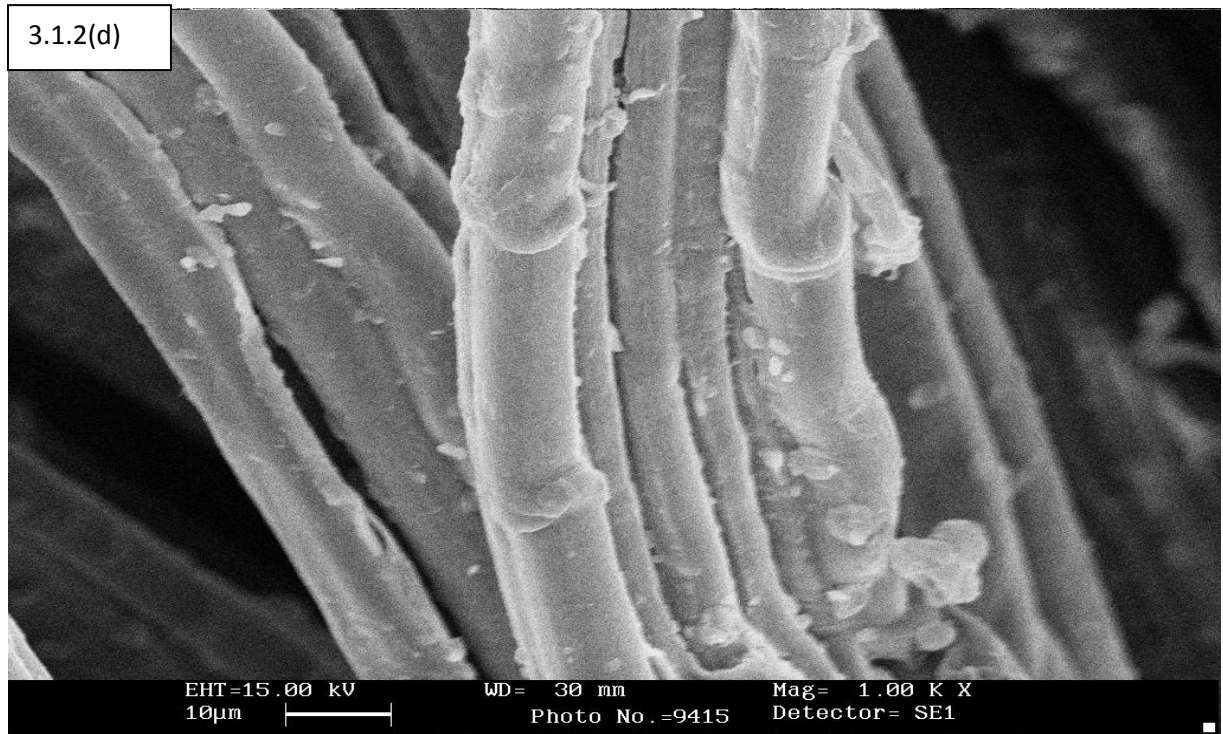


Fig.3.1.2. (a) SEM micrographs Heat+Alkali+Silane treated Jute fiber

The micrograph 3.1.2 (a) has an unwrinkled surface due to the presence of fatty acids, and wax. After Heat treatment, ruptures on fiber's surface are visible in the SEM micrograph 3.1.2(b). On NaOH treatment and also in case of silane treatment, the unruptured and very clearly visible layer on untreated fiber's surface gets washed out and exposes fibrils making surface quite rough which will help in better interlocking yielding better strength. The pits visible in micrographs 3.1.2 (c) and (d) are formed because of the partial removal of fatty, waxy, hemicellulose, and lignin.

### 3.2. Mechanical Properties

Figure 3.2.1-3.2.9 show tensile strength, Young's modulus and elongation at break for composites made using 1, 2, and 3 jute mats without treatment and after various treatments.

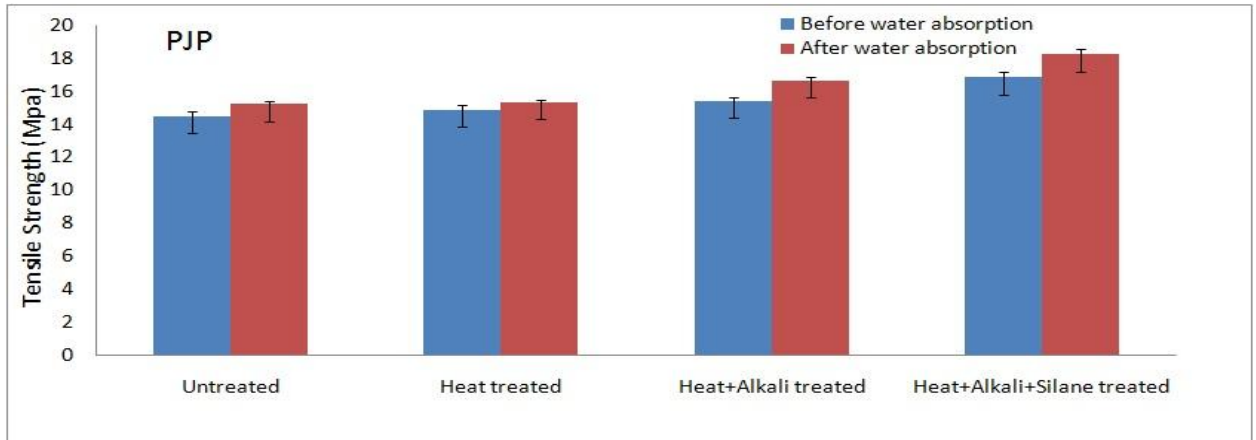


Fig. 3.2.1. Tensile Strength for composites prepared using one layer of Jute mat

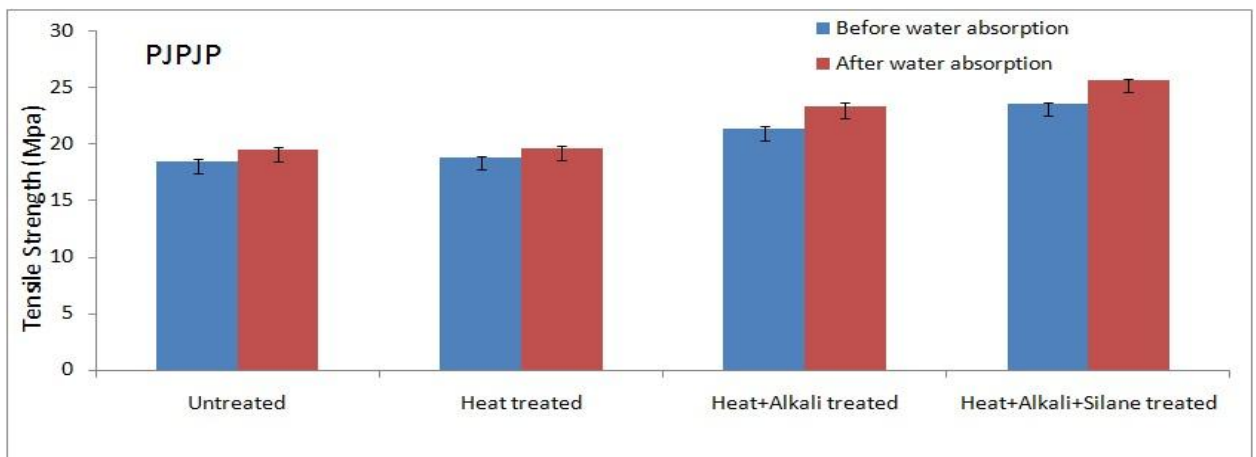


Fig. 3.2.2. Tensile Strength for composites prepared using two layers of Jute mat

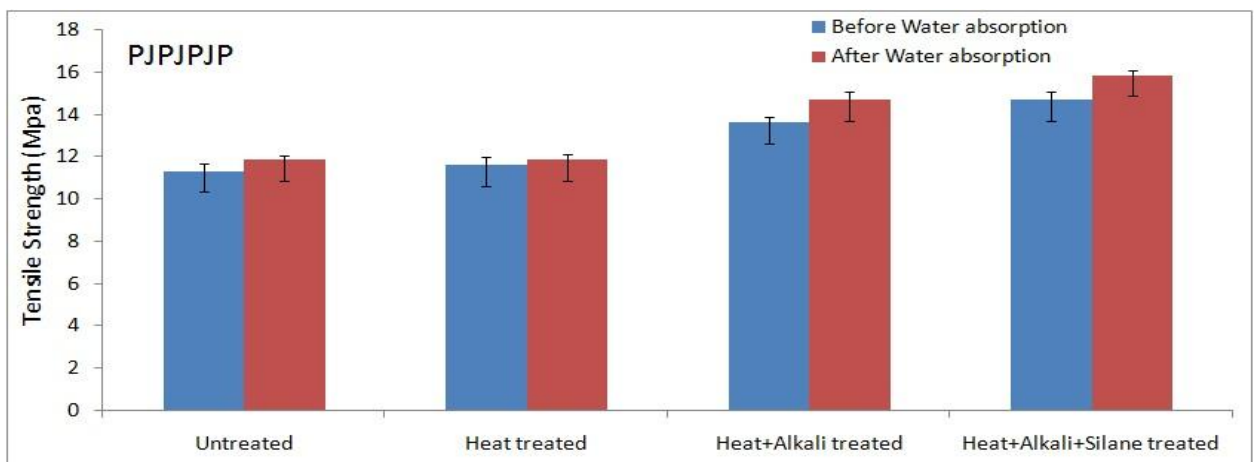


Fig. 3.2.3. Tensile Strength for composites prepared using three layers of Jute mat



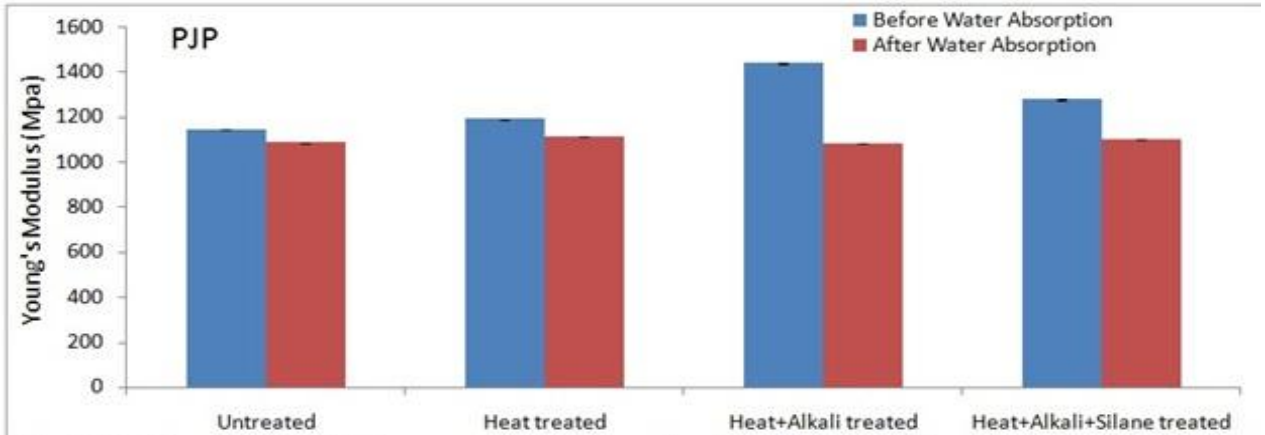


Fig. 3.2.4. Young's Modulus for composites prepared using one layer of Jute mat

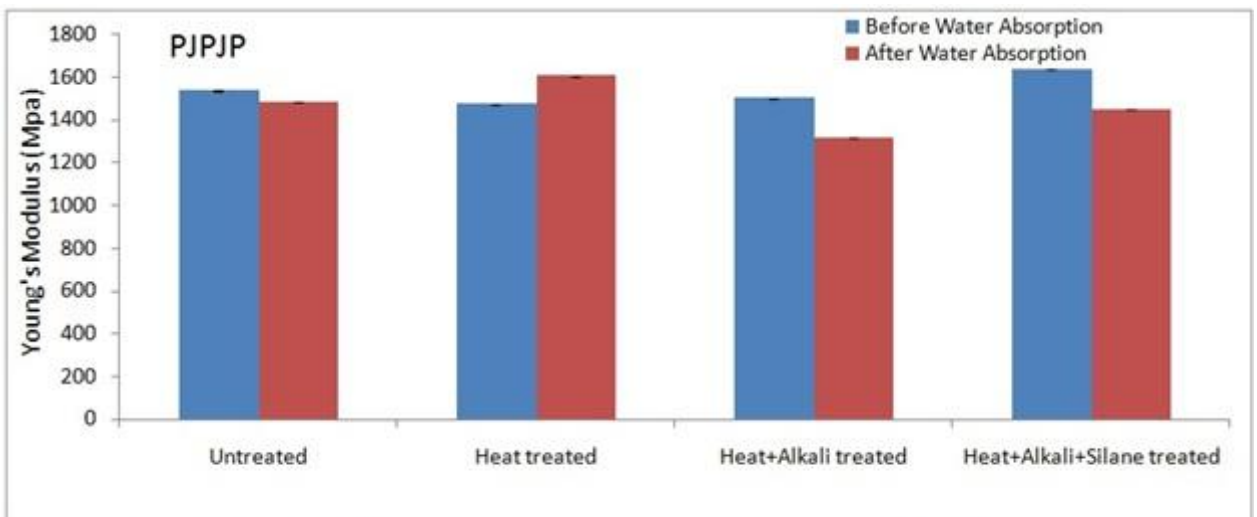


Fig. 3.2.5. Young's Modulus for composites prepared using two layers of Jute mat

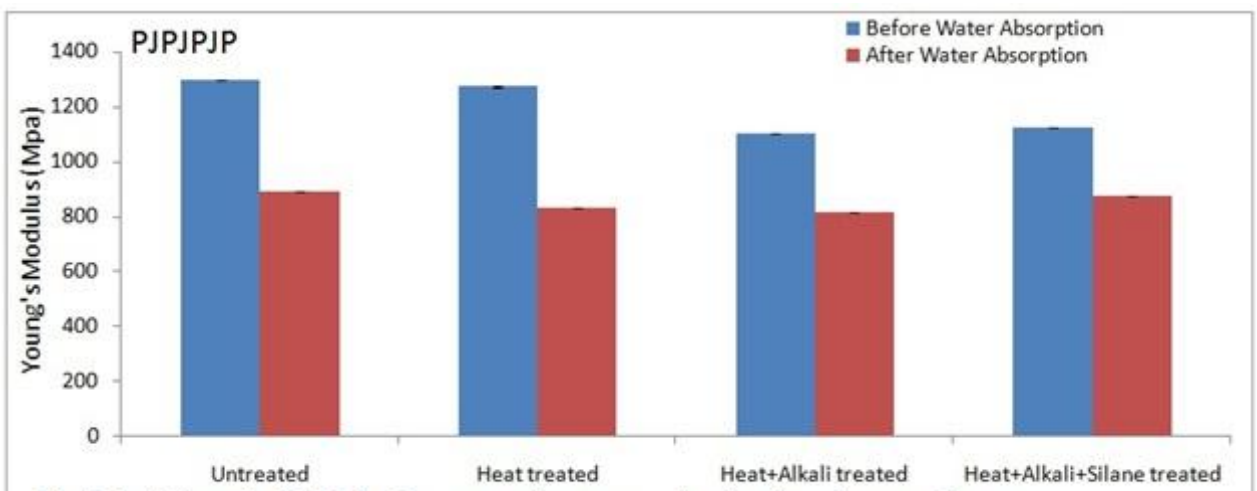


Fig. 3.2.6. Young's Modulus for composites prepared using three layers of Jute mat



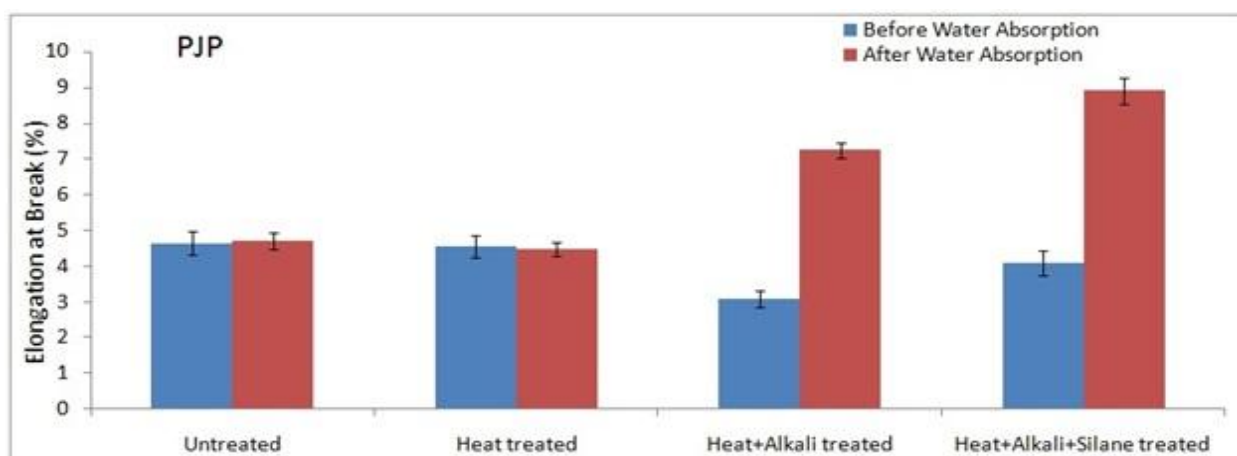


Fig. 3.2.7. Elongation at Break for composites prepared using one layer of Jute mat

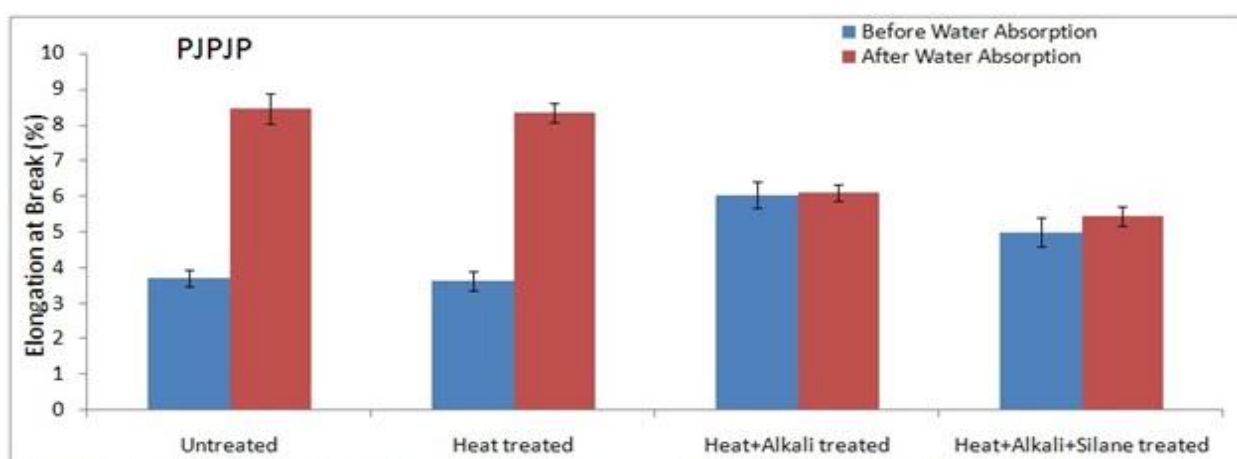


Fig. 3.2.8. Elongation at Break for composites prepared using two layers of Jute mat

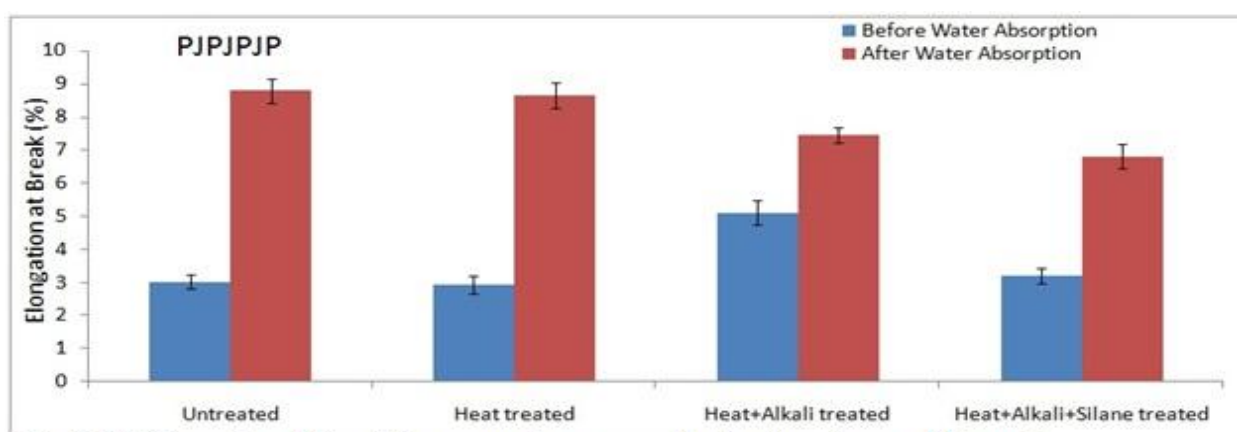


Fig. 3.2.9. Elongation at Break for composites prepared using three layers of Jute mat

From the results, it is observed that the composite containing two layers of jute mats with maximum tensile strength and Young's modulus. However, percent elongation at break decreases with the increase in jute mat layers. This is because addition of jute fiber mat into the polymer matrix is leading to reduction in the matrix mobility. It is clear that tensile strength increases after continual treatments, maximum after silane treatment. On the other hand, trend is not clear for Young's modulus and elongation at break. Young's Modulus shows very little change for after heat treatment for all cases. With single jute mat, it increases considerably after alkali treatment but decreases after silane treatment. With two jute mats, it does not change much even after alkali treatment, however, its value falls appreciably after silane treatment. With three jute mats, it decreases after alkali treatment but remains almost unchanged after silane treatment. Elongation at break, is almost unchanged after heat treatment but after alkali treatment, it decreases with single jute mat and increases for composite made using two and three jute mats. After silane treatment, its value shows considerable fall with two and three jute mats, however, with single jute mat, it increases appreciably.

Since treatments help remove impurities from the fiber surface and cause changes in the arrangement of units in the cellulose macromolecule. As a result, a rough surface with enhanced aspect ratio provide better fiber–matrix bonding leading to enhanced mechanical properties [19].

After water absorption, the tensile strength increases in value. It can be attributed to the swelling of the fibers after water absorption. Owing to swelling, the gaps between the fibre and the matrix which may have appeared during manufacturing process due to a poor impregnation, get filled resulting in an improvement the tensile strength [20]. Young's Modulus has decreased in all cases after water absorption. Opposite trend was observed in the case of elongation at break where it increased after absorbing water especially, with three jute mats where water absorption has been maximum.

Similar reasoning can be attributed to the flexural properties also. Figures 3.2.10 to 3.2.15 show flexural strength and flexural modulus corresponding to composites prepared using one, two and three jute mats without treatment and after various treatments.

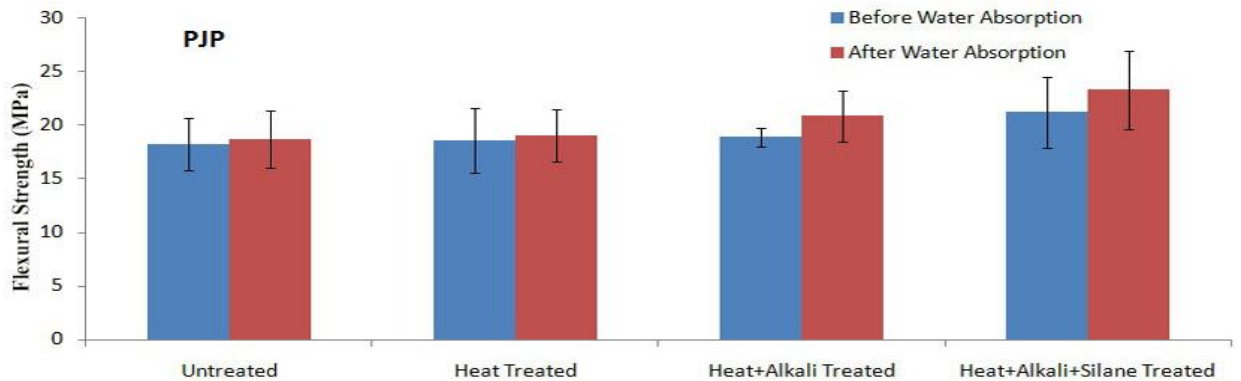


Fig. 3.2.10. Flexural Strength for different composites prepared using one layer of Jute mat

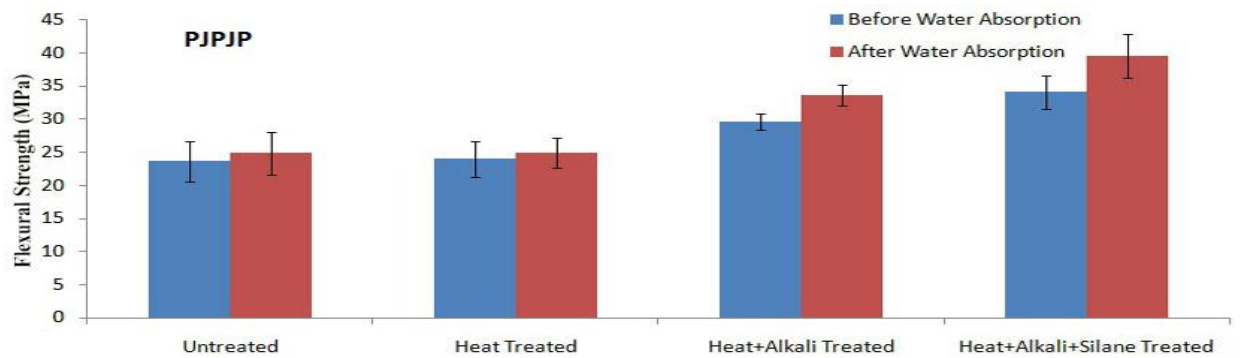


Fig. 3.2.11. Flexural Strength for different composites prepared using two layers of Jute mats

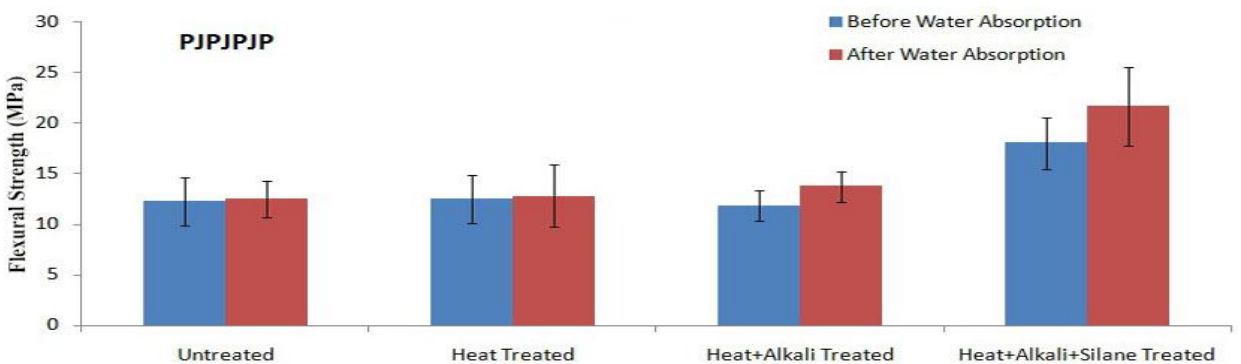


Fig 3.2.12. Flexural Strength for different composites prepared using three layers of Jute mat

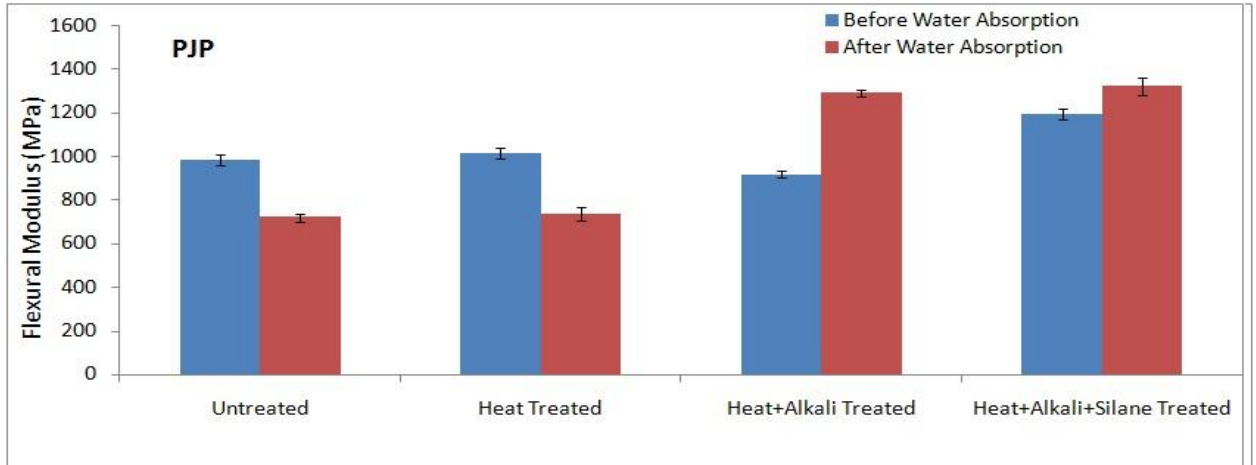


Fig. 3.2.13. Flexural Modulus for different composites prepared using one layer of Jute mat

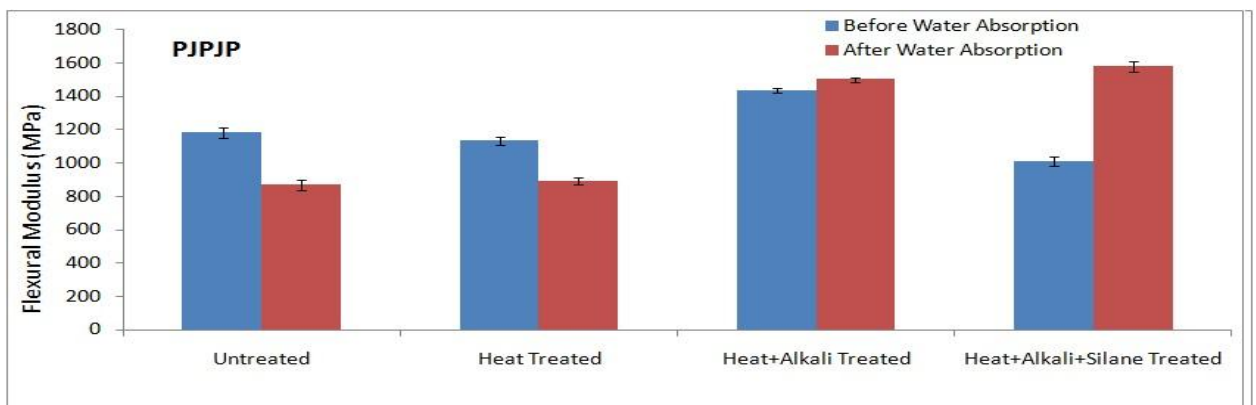


Fig. 3.2.14. Flexural Modulus for different composites prepared using two layers of Jute mat

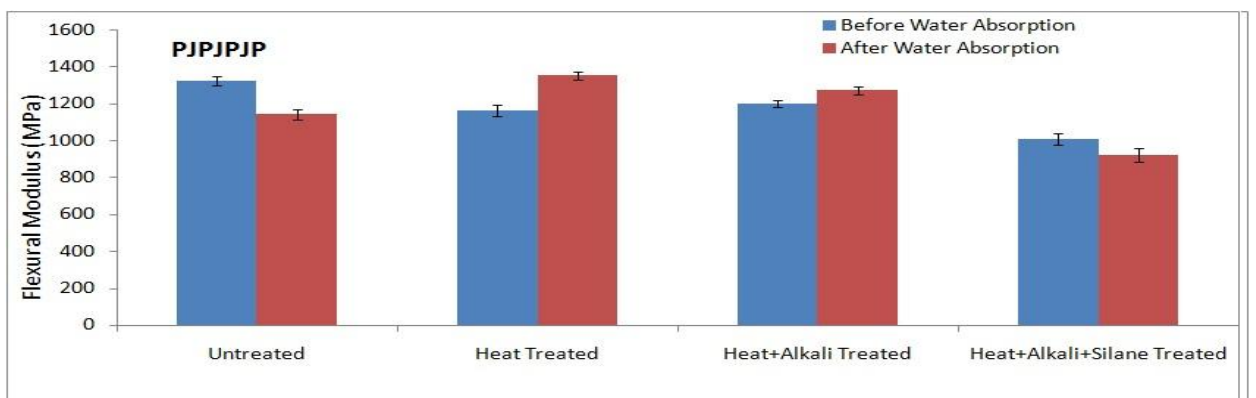


Fig. 3.2.15. Flexural Modulus for different composites prepared using three layers of Jute mat

Flexural strength shows a similar trend as exhibited by tensile strength. The value of Flexural strength ascends after continual treatments, maximum with two jute mats after silane treatment. The flexural modulus doesn't show much change after heat treatment, however after alkali treatment in case of single Jute mat, it decreases but after silane treatment, it increases. Vice Versa is the case with two jute mats after alkali and silane treatment. With three jute mats, flexural modulus remains unchanged after alkali treatment but decreased in value on silane treatment. After water absorption, the flexural strength also improved. Thus, the swelling fibre factor has played the role in the enhancement of properties in this case too.

Impact strength computed using the Izod test is shown in figure 3.2.16-3.2.18

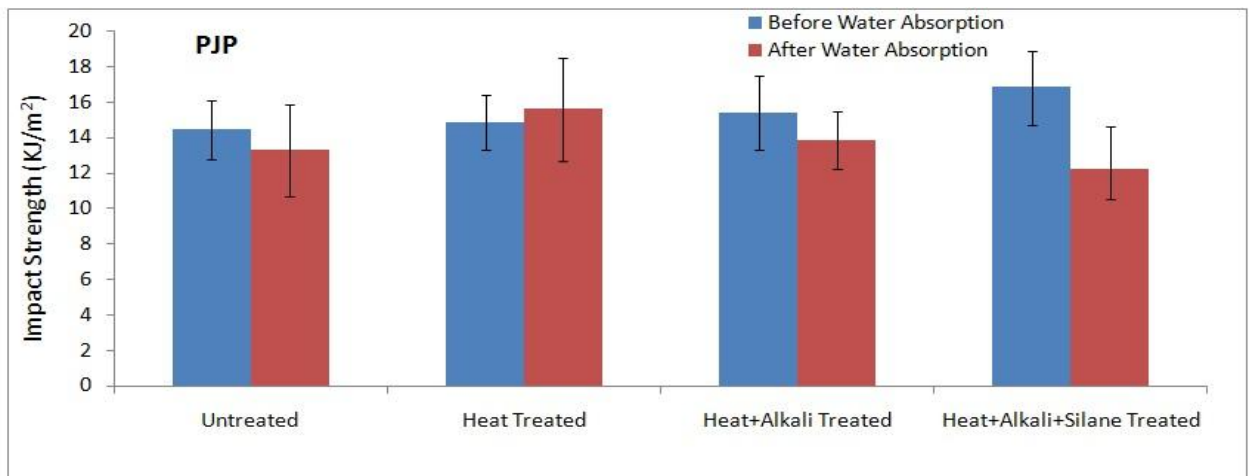


Fig. 3.2.16. Impact Strength for different composites prepared using one layer of Jute mat

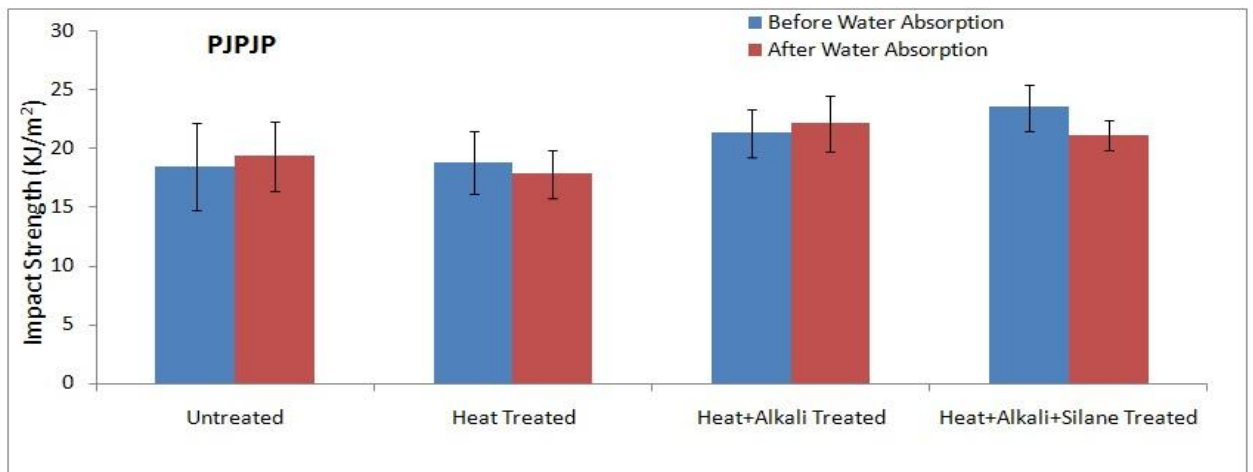


Fig. 3.2.17. Impact Strength for different composites prepared using two layers of Jute mat

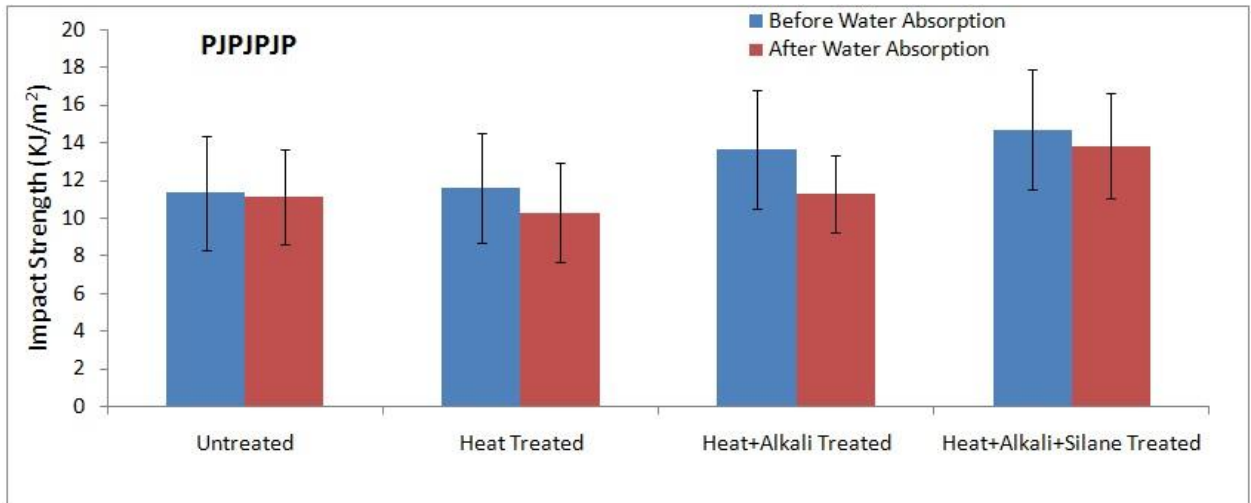


Fig. 3.2.18. Impact Strength for different composites prepared using three layers of Jute mat

Impact strength showed similar trends as those of tensile and flexural strength, however, it differs after water absorption. In all cases but for one with two layers of untreated jute mat, it decreased after water absorption. From results of tensile and flexural strength, it can be concluded with certainty that swelling of fibers leads to better interlocking. So, in case of impact strength, following argument may be given to explain the results; swelling is causing some cracks in the composite which tend to facilitate the breaking during impact tests, thus, impact strength decreased after water absorption.

For better understanding the mechanical behavior, stretched out surface of composites after carrying out the tensile test was also examined for composite prepared using single untreated jute mat and two jute mats after treatments till silane (Figure 3.2.19 (a), (b), (c) and (d)). SEM images concur the above results; as in the case of untreated single Jute mat fiber composite, fibers are observed to be bonded loosely with the HDPE matrix. Also, relatively more pullout of fibers have been witnessed, implying poor adhesion between the fiber and the matrix than the silane treated two jute mats composite which has considerably fewer gaps between the fiber and the HDPE matrix. Treatment has also made the fiber surface more hydrophobic.

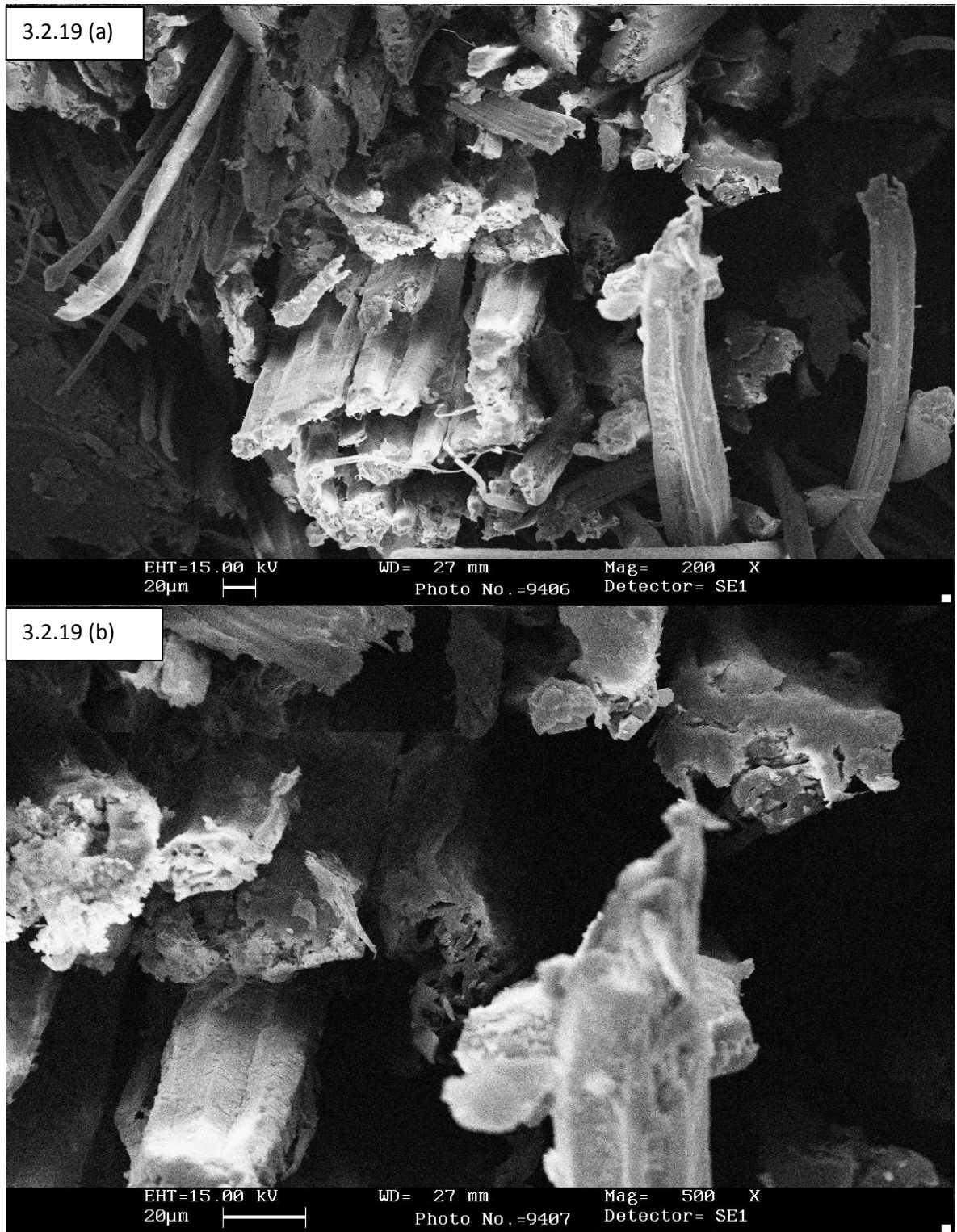


Fig. 3.2.19. SEM Micrographs for fractures surface of composite prepared using untreated single Jute mat composite



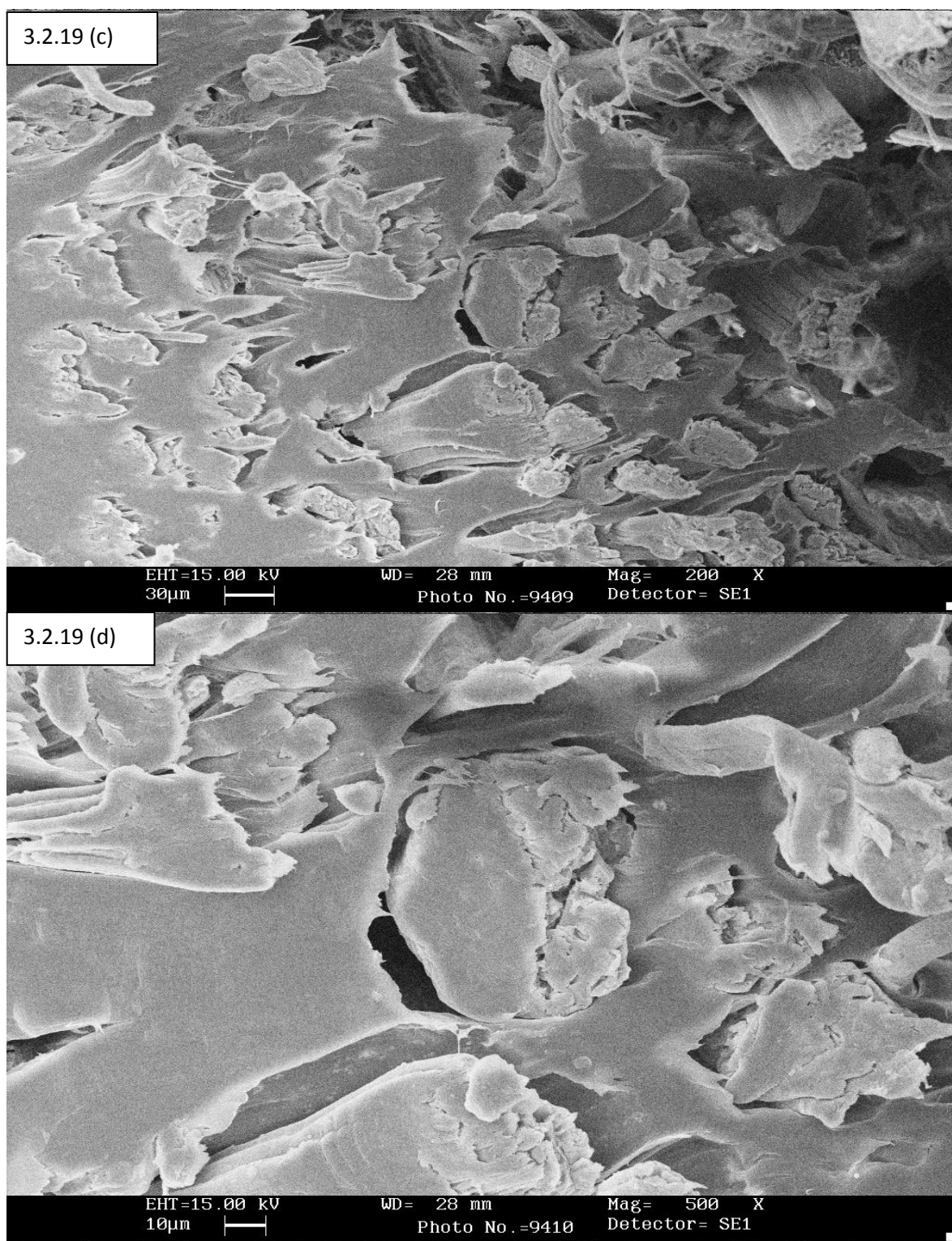


Fig. 3.2.19. (c) and (d) SEM Micrographs for fractures surface of composite prepared using two Heat+Alkali+Silane treated Jute mat composite



### 3.3. Water Absorption

The water absorption behavior for untreated, Heat treated, Heat+Alkali and Heat+Alkali+Silane treated composites was studied and is as shown in figure 3.3.1-3.3.3.

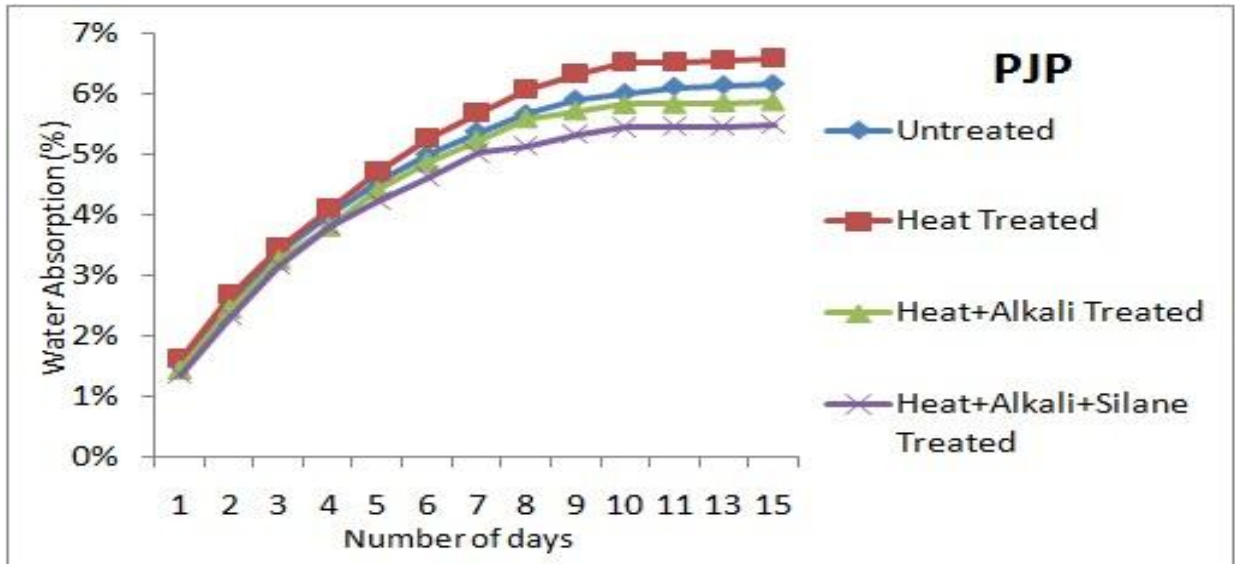


Fig.3.3.1. Water absorption for composite prepared using single jute mat without and after various treatments

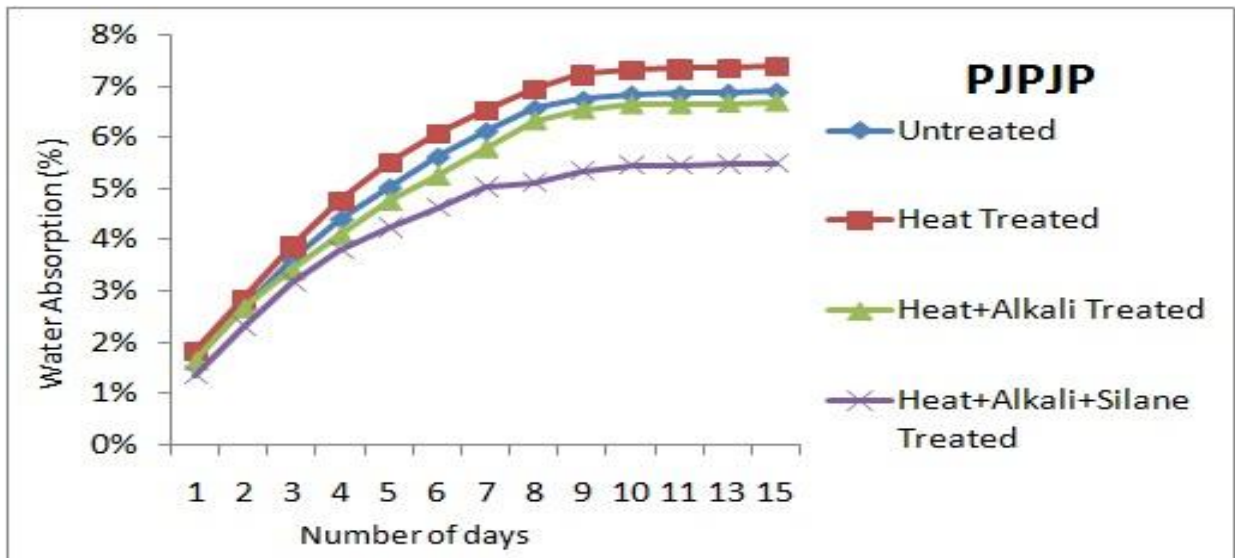


Fig.3.3.2. Water absorption for composite prepared using two jute mats without and after various treatments

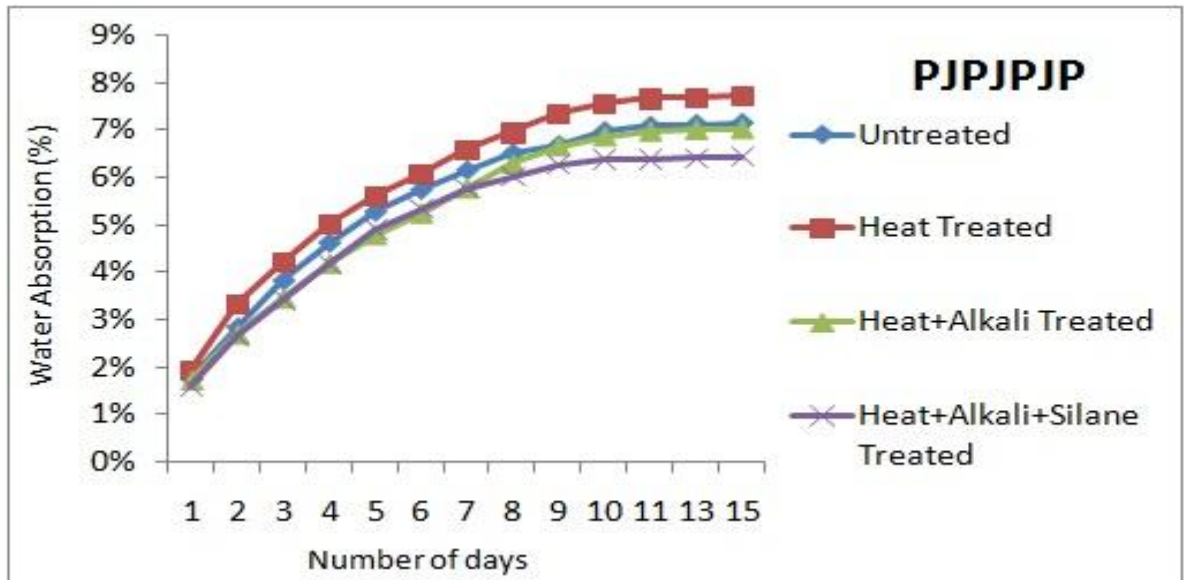


Fig.3.3.3. Water absorption for composite prepared using three jute mats without and after various treatments

It is clear that water absorption is increasing with fiber content i.e. it's minimum for single jute mat composites and maximum for three jute mat composites. It increases at a considerable rate for first 7-8 days and then appreciable fall is observed in the rate of absorption which tends to zero after 5-6 days. Also, it can be inferred that water absorption increases on heat treatment but after alkali and also after subsequent silane treatment, it decreases considerably especially after silane treatment which is in accordance with the literature that reports alkali and silane treatment as effective ways to reduce water absorption in composites. The reduction of water absorption can be attributed to the removal of hydrophilic content and also to the elimination of voids because of better bonding between reinforcement and matrix. The best results i.e. least water absorption as clear from figures 3.3.1-3.3.3, were obtained with continual treatment till that with silane.

### 3.4. X-ray Diffraction

The crystallite size was calculated for the composites with one, two and three jute mats for fiber after various treatments using the Scherrer's equation and is given in Table 3.4.1. The highest value was observed for the composite made using two jute mats after silane treatment.

Table 3.4.1 Crystallite size for Composites

<b>Treatment</b>	<b>2<math>\theta</math> at Higher Peak</b>	<b>Crystallite size (nm)</b>	<b>Composite</b>
Untreated	21.63	22.55	<b>PJP</b>
Heat	21.53	22.74	
Heat+Alkali	21.64	22.98	
Heat+Alkali+Silane	21.70	23.65	
Untreated	21.82	24.41	<b>PJPJP</b>
Heat	20.85	24.57	
Heat+Alkali	21.66	25.75	
Heat+Alkali+Silane	20.88	26.73	
Untreated	21.93	21.11	<b>PJPJPJP</b>
Heat	21.61	21.23	
Heat+Alkali	21.68	22.18	
Heat+Alkali+Silane	21.70	22.67	

It can be inferred that crystallite thickness increased on continual treatments. This implies the removal of amorphous content, whose presence leads to poor strength. In its absence, we can expect the mechanical properties to improve on treatments.

### 3.5. Thermogravimetric Analysis

Thermal Gravimetric Analysis (TGA), Derivative Thermogravimetric Analysis (DTG) and Differential Thermal Analysis (DTA) studies were carried out for Untreated, Heat treated, Heat+Alkali treated and Heat+Alkali+Silane treated Jute fiber. Figure 3.5.1 and 3.5.2 show the TGA and DTG curves for the fibers.

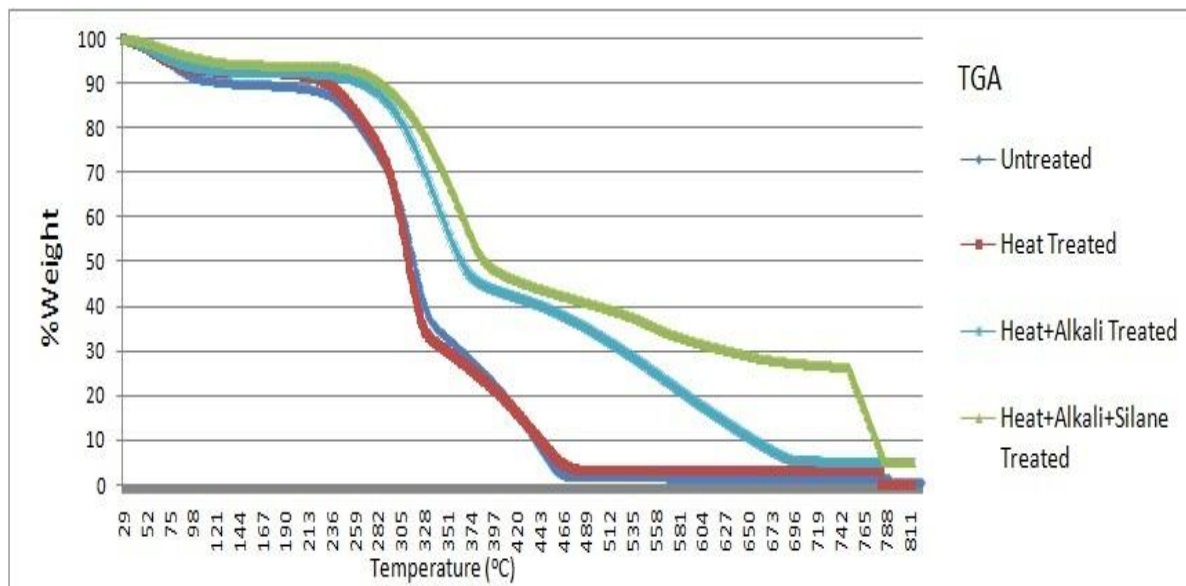


Fig. 3.5.1. TGA cuves for Jute fiber without and after various treatments

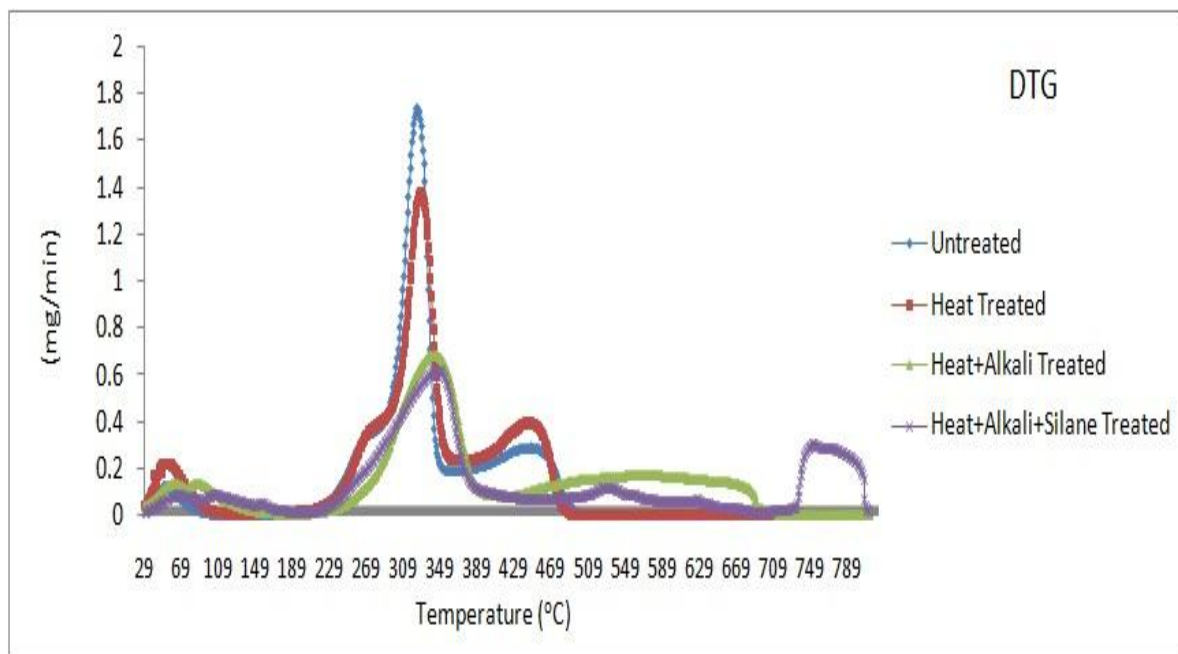


Fig. 3.5.2. DTG cuves for Jute fiber without and after various treatments

With untreated fibers, a weight loss of 8.75% was observed before temperature reached 100°C, owing to loss of moisture. Thermal decomposition took place in two stages. Half of the weight was lost by the end of stage one (200–350°C) because of the depolymerization and dehydration. This was immediately followed by the stage 2 (350–480°C) decomposition. This led to a considerable decomposition for untreated and heat treated fiber. The stage 2 decomposition could be attributed to the cellulose decomposition and combustion of char [21].

It's clear from the Table 3.5.1 that initial decomposition temperature and decomposition temperature corresponding to 30% weight loss are quite similar in case of untreated and heat treated Jute fiber.

Table 3.5.1. Thermogravimetric Analysis of Jute fiber without and after different treatments

Sr. No	Treatment	IDT* (°C)	DT** (°C) 30% wt loss	Final Residue (%)	Exothermic peak at temperature (°C)
1	Untreated	190	292	1.075	321(56μV), 448(154μV)
2	Heat	194	290	2.8	315(56μV), 456(115μV)
3	Heat+Alkali	232	327	4.9	347 (18μV), 562( 96μV)
4	Heat+Alkali+Silane	228	349	4.7	359 (12μV), 525( 74μV)

IDT: Initial Decomposition Temperature

DT: Decomposition Temperature

However, appreciable rise in decomposition temperatures is observed after Alkali and Silane treatment. So, it can be said that the thermal stability of Jute fiber increases on alkali and silane treatment which will reflect in the composites prepared using these fibers. The amount of residual char left increased appreciably from 1.075 to 4.9% on alkali treatment fiber and 4.7% after of silane treatment. Similar results were obtained by Saha et al.

[22]. They gave the argument that alkali treatment leads to reduction in hemicelluloses, leading to lignin–cellulose complex, thus giving much more stability as compared to the untreated. DTA evaluation supports the TGA studies. DTA of untreated fiber show exothermic peaks at 321°C (56  $\mu$ V) and 448°C (154  $\mu$ V), and uninterrupted exothermic combustion of the untreated sample took place at the furnace temperature with atmospheric oxygen, which implies the total disintegration of C — C and C — O bonds.

### 3.6. CONCLUSIONS

Composites prepared using two jute mats after silane treatment were found to give best results. Furthermore, the effect of heat, alkali, and silane treatment on the fiber was investigated. Results show that treatments have a favorable impact on mechanical properties of composite. After water absorption, tensile and flexural strength were found to be enhanced but impact strength decreased after absorbing water. As per thermogravimetric analysis, it was observed that thermal stability of composite increases on treatment. Treatment with alkali partially removes hemicelluloses and lignin from the fiber surface decreasing hydrophobic nature in fiber surface making it easier to bond with HDPE matrix. Silane treatment improves the interaction between fiber and the matrix resulting in better interlocking. The partial elimination of lignin, hemicelluloses, and other components like fats and wax was also confirmed by SEM micrographs and FTIR spectra. Treatment of Jute fiber improved the mechanical properties as confirmed by SEM micrographs of fractured surfaces of composite after tensile tests and enhanced the water resistance appreciably exhibiting minimum water intake.

## Recommendations

- A study can be done of thermal decomposition kinetics to calculate activation energies for decomposition of different composites to get a better estimate of thermal stability.
- Composites can be prepared using a different fiber or polymer matrix. Comparison of mechanical, thermal and water absorption behavior can be done for better assessment of properties.



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