Fabrication and Characterization of Bio Composite Based on Jute Fiber and Pine Rosin Modified Potato Starch

Karishma M. Sakhare[1]*, Shashikant P. Borkar[2], Ravindra D. Kale[3]

[1]*Research Scholar, Textile Manufactures Department, Veermata Jijabai Technological Institute, Mumbai, 400019, India,
[2]Professor, Textile Manufactures Department, Veermata Jijabai Technological Institute, Mumbai, 400019, India,
[3]Professor, Department of Fibres and Textile Processing Tech, Institute of Chemical Technology, Mumbai, 400019, India,

ABSTRACT:

Background- Composite materials have garnered significant attention from researchers in recent times. These materials exhibit notable variations in their physical and chemical characteristics and are comprised of two distinct components known as the matrix and the reinforcement.

Aim- The primary objective of this study was to investigate the fabrication and analysis of a bio-composite material using jute fibre and potato starch modified with pine rosin.

Methodology- The use of pine rosin modified starch as a matrix material, in conjunction with jute fibre as a reinforcing agent, has been employed in this study. Fibre characterizations were conducted to assess the mechanical and hygroscopic qualities, as well as to perform Fourier transform infrared spectroscopy (FTIR), Thermal gravimetric analysis (TGA), Differential scanning calorimetry (DSC), and Scanning electron microscopy (SEM).

Result- Following comprehensive testing, JSTP15, a composite material containing 15% pine rosin in jute starch, exhibited superior tensile strength, modulus, flexural strength, modulus, and impact strength. JSTP15 also displayed significantly reduced water absorbency, approximately 26% less than the pine rosin-free composite, JSTP0. TGA analysis indicated a slower thermal deterioration rate in JSTP15 than in JSTP0. In contrast, DSC data suggested that pine rosin's inclusion increased thermal degradation. Furthermore, chemical affinity studies were conducted with various solutions, including benzene and a 5% acetic acid solution.

Conclusion- The modification of starch resin with the optimum percentage of pine rosin can enhance the mechanical, thermal, as well as water-resistant properties of the jute fibre reinforced composites.

1. Introduction

The use of biocomposite materials has a significant potential for fostering a sustainable and secure environment via the substitution of synthetic plastics and materials with organic constituents across many domains of application. There has been a significant amount of attention given to composites derived from natural and renewable resources in recent years [1]. A material composed of a biodegradable polymer matrix that is strengthened by the inclusion of natural fibers is often referred to as a biocomposite material [2]. The use of natural fibers derived from both renewable and nonrenewable sources, such as jute, oil palm, flax, and sisal, for the production of composite materials has garnered significant interest in recent decades [3]. Jute fibres have many benefits, such as their environmentally friendly nature, moderate mechanical qualities, and cost-

effectiveness, which make them a desirable substitute for synthetic fibres across various applications [4].

The introduction of a plasticizer induces the formation of a void space between the polymer chains of starch, resulting in a decrease in the degree of crystallinity shown by the plastic material. The plasticized starch has enhanced versatility, enabling its combination with diverse polymeric substances to cater to a wide range of applications. In their study, Sun et al. (2017) used citric acid, adipic acid, borax, and boric acid as cross-linking agents in the production of cross-linked starch/PHA composites [5]. In their study, Iman et al. (2012) used glutaraldehyde as a cross-linking agent to produce composites consisting of starch and jute cloth [6]. The primary constituents of rosin are rosin acids, which possess acidic properties. Typically, rosin consists of

around 90% acidic components and 10% neutral components. The chemical reactivity of rosin is attributed to its mono-carboxylic acid functionality. The primary rosin acid found in pale, purified, and unaltered rosin is abietic acid. This acid is characterized by its empirical formula, $C_{20}H_{30}O_2$. The chemical structures of the abietic acids as presented by Maiti et al. in 1989. Abietane-structured acids exhibit distinctive features, namely the presence of conjugated double bond systems, rendering them very prone to undergoing chemical changes [7].

As stated by Mohammed et al. (2015) [8], the compound exhibits insolubility in water but demonstrates solubility in several organic solvents. The evaluation and commercialization of this product are based on its color, where the presence of lighter colors of yellow brown is indicative of superior quality. Rosin finds extensive utilization throughout several domains, including the manufacturing of adhesives, ink manufacture, and paper sizing [9]. The composite material investigated in this work was composed of jute fibre as the reinforcing agent, and potato starch that had been modified using a crosslinking agent (specifically, pine rosin) and 30% glycerol as a plasticizer, with the weight ratio of the matrix being considered. The green composite was manufactured via a compression molding apparatus. The concentration of pine rosin was modified within the range of 0 to 30% based on the weight of the matrix. It should be noted that the fibre volume fraction remained constant at 0.2 for all concentrations. This study aimed to evaluate the effects of different concentrations of pine rosin on mechanical, thermal, and hygroscopic qualities. The primary objective of this study is to investigate the

production and analysis of a biocomposite material using jute fibre and potato starch modified with pine rosin.

2. Materials and methods
2.1 Material
Potato starch powder was supplied by Loba Chemie Pvt. Ltd. in laboratory and reagent (LR) grade, while glycerol was also given. The jute fibre strands, and M/s. were obtained from the Central Research Institute for Jute & Allied Fibres in Kolkata, West Bengal, India. Seba Associates, a company based in Bhubaneswar, Odisha, offers solid crystal pine rosin.

2.2 Methods
The composite samples generated may be categorised into two distinct groups: The first kind of composites consists of jute fibre reinforced unmodified starch matrix, whereas the second type comprises jute fibre reinforced pine rosin modified starch matrix. The fibre volume percentage was maintained at a consistent value of 0.2 throughout all composite samples, since the primary focus of the composites was the modification of starch by the utilisation of pine rosin. Different quantities of pine rosin were used to modify the starch powder in the matrix samples of jute fibre reinforced pine rosin modified starch. Various weight percentages of pine rosin (5%, 10%, 15%, 20%, 25%, and 30%) were used as modifiers for starch in this study. The matrix produced for all the samples was plasticized with 30% glycerol on a weight basis. The composite material was fabricated with a hot compression moulding process. Table 1 presents the calculations and codes pertaining to all compositions.

Table1.Calculation and codes for composites								
Codes for composite samples	Total wt. of matrix (gm)	Con. of Starch on wt. Basis of matrix (%)	Con. of Pine Rosin on wt basis of matrix (%)	Con. of glycerol on wt. basis of matrix (%)	Wt. of jute fibre (Fibre volume fraction 0.2)	Wt.of potato starch (gm)	Wt. of Glycerol (gm)	Wt. of pine Rosin (gm)
JSTP0	144	70	0	30	60	100.8	43.2	0
JSTP5	144	65	5	30	60	93.6	43.2	7.2
JSTP10	144	60	10	30	60	86.4	43.2	14.4
JSTP15	144	55	15	30	60	79.2	43.2	21.6
JSTP20	144	50	20	30	60	72.0	43.2	28.8
JSTP25	144	45	25	30	60	64.8	43.2	36.0
JSTP30	144	40	30	30	60	57.6	43.2	43.2

2.3 Preparation of Jute fibre and pine rosin
The jute strands were cut to a predefined length of 15 mm using scissors. Subsequently, they were processed in the opening machine to eliminate any waste material and to facilitate the separation of the fibres. This separation was necessary in order to get a consistent mixture with the resin. Subsequently, the fibres underwent a 24-hour conditioning period at ambient

temperature. The solid and brittle pine rosin given by the firm was then transformed into a powdered form using a conventional grinder.

2.4 Preparation of the Starch paste
The starch was blended with a quantity of water in a pot that was three times more than the predicted amount based on the combined weight of the starch and fibre.

The temperature range of the mixture was maintained at 60°C to 70°C, while using a magnetic stirrer to provide continuous stirring for a duration of 20 minutes. This approach was used to prevent the development of lumps and create a uniform mixture. A 30% glycerol solution was added into the mixture based on its weight, followed by agitation for an additional duration of 10 minutes. Subsequently, the combination was placed in a water bath that was heated to a temperature of 80°C. Throughout this process, the mixture was continuously agitated until the starch underwent gelatinization and the viscosity of the mixture increased. Following the cooling of the starch paste, a precise quantity of pine rosin powder was introduced and homogeneously mixed using a glass rod.

2.5 Preparation of composite
Once the resin was created, precise measurements of chopped and opened fibres were manually incorporated into the resin. Subsequently, the resulting mixture was manually agitated to ensure a consistent dispersion of fibres inside the matrix. In order to enhance the viscosity, the mixture was allowed to stand at ambient temperature for a duration of 24 hours. Subsequently, the resin and fibre amalgamation were introduced into a mould with dimensions of 200 mm × 200 mm x 5 mm, with the objective of producing a uniform composite sheet. In order to prevent the resin/fibre combination from flowing out, the mould was placed into a compression moulding machine and subjected to a slow application of pressure. In order to facilitate the drying process of resin or fibre sheets, it is necessary to subject the mould to a temperature of 80°C for a duration of 30 minutes. The curing process was conducted at a temperature of 120°C for a duration of 3 hours, while being subjected to a pressure of 50kg/cm$^{2.}$ The procedure for the production of the biocomposite, as seen in Figure 1, is as follows.

Fig. 1 Fabrication route of biocomposite. (A) Strand of jute fibre, (B) Chopped jute fibre (15 mm), (C) Opened jute fibre, (D) Starch Powder, (E) Preparation of Resin, (F) Resin ready for use, (G) Resin/fibre mixture, (H) Pre-cured biocomposite sheet on mould and (I) Biocomposite after Curing.

2.6 Mechanical characterizations
The mechanical properties of jute-starch composites were evaluated in the absence and presence of pine rosin. The practical usefulness of materials is contingent upon their mechanical qualities. The primary objective of the mechanical characterization was to investigate the impact of varying concentrations of pine rosin in starch on the composite sample, with the aim of determining the optimal combinations. Subsequently, the most suitable combination of pine rosin and starch was chosen for a comprehensive examination. This measure was implemented in order to mitigate data burdens and prevent any misunderstandings. The tensile, flexural, and impact characteristics of a jute composite that was made were evaluated using standardized testing methods, namely ASTM D638-03, ASTM D790-07, and ASTM D256-06a. The jute composite was tested both with and without the inclusion of pine rosin.

2.7 Tensile Testing
A composite specimen with dimensions of 115mm x 19mm x 4mm was extracted for the purpose of conducting tensile testing, following the guidelines outlined in ASTM D638-03. The experiment was conducted with a crosshead speed of 2 millimetres per minute and a gauge length of 65 millimetres. A total of five specimens for each variety were assessed under controlled conditions of 23°C temperature and 55 percent relative humidity (RH), and the resulting mean results were reported. The Tensile Testing Machine was

used to ascertain the tensile properties of composite materials.

2.8 Flexural Testing

The specimens used in the flexural testing were produced following the guidelines outlined in the ASTM D790-07 standard. In accordance with the prescribed norm, the 3-point bending technique was used. In this experiment, a specimen was used, with nominal dimensions of 120mm x 12.7mm x 4mm. The span length of the specimen was set at 80mm, while the crosshead speed was maintained at a constant rate of 2mm/min. A total of five duplicate samples for each variety were subjected to conditioning and testing under controlled environmental conditions, namely at a temperature of 23°C and a relative humidity of 55%. The experiments were conducted using the same universal testing machine (UTM) that had been previously used for conducting tensile tests.

2.9 Impact testing

In this experiment conducted on the Izod Impact Tester, a striker with an energy of 2.7 joules was used. The dimensions of the specimen were measured to be 63.5mm x 12.7mm x 4mm. Following the guidelines outlined in ASTMD 256-6a, a "V" notch was created in the middle of the specimen's face. The notch had a depth of 2.54mm and an angle of 45°. The notch was cut out using a specialised notch cutter, resulting in a final measurement of 63.5mm x 4mm for the face. Five specimens of each species were conditioned and evaluated under controlled environmental conditions, namely at a temperature of 23°C and a relative humidity of 55%. The impact strength in KJ/m² was calculated by dividing the observed absorbed impact energy by the cross-sectional area of the specimen.

2.10 Water absorbency

The study investigated the impact of water absorbency on five identical samples, each representing different versions, namely JSTP0, JSTP5, JSTP10, JSTP15, JSTP20, JSTP25, and JSTP30. The water absorbency was assessed using the guidelines outlined in the ASTM D570-98 standard. The initial weight of the conditioned specimen was measured for each sample, followed by the immersion of the seven specimens in distilled water.

A sample size of five was selected for each specimen. The container housing the specimen was maintained at a temperature of 23.6°C for a duration of 24 hours subsequent to its immersion. Individual samples were collected from the water, and any surplus water was removed using a dry towel. The weights of the samples were afterwards measured again with a precision of 0.001g. The below equation was used to compute water absorption.

$$\text{Increase in weight \%} = \frac{\text{Wet weight} - \text{Conditioned weight}}{\text{Conditioned weight} \times 100}$$

2.11 Chemical resistance property

The chemical resistance qualities of distilled water, benzene, hydrochloric acid, acetic acid, sodium hydroxide, and sodium carbonate solution were evaluated using the ASTM D 543–95 test standard. In order to conduct the experiment, a selection of five specimens each of JSTP0 and JSTP15 were selected. The specimens were pre-weighed and thereafter immersed in the chemicals for a duration of 24 hours, while being maintained at ambient temperature. Following a 24-hour duration, the specimens were extracted, and surplus chemicals were eliminated by interposing these specimens between two filter sheets. The weight and thickness of each specimen were analysed to determine the percentage change, followed by the calculation of the average change.

2.12 FTIR, TGA, DSC, and SEM characterization

Two versions, namely jute-starch without pine rosin (referred to as JSTP0) and jute-starch with 15% pine rosin (referred to as JSTP15), were selected for characterisation using FTIR, TGA, DSC, and SEM techniques in order to facilitate comparison. The decision was made on the basis of the mechanical test results.

2.13 Fourier transforms infrared spectroscopy (FTIR)

The Fourier transform infrared spectrum (FTIR) was acquired using a Perkin Elmer Spectrum 100 FT-IR Spectrometer. The spectra of the samples were obtained within the wavelength range of 4000-650 cm⁻¹.

## 2.14 Thermo-gravimetric analysis (TGA)

The thermal characteristics of the samples were examined using a DTG 60H TGA apparatus (Shimadzu, Japan) in a controlled nitrogen environment. Thermogravimetric data was collected throughout a temperature range of 30°C to 600°C, with a heating rate of 10°C per minute and a nitrogen gas flow rate of 100 ml per minute. During the experiment, the specimens were placed into an aluminium container.

2.15 Differential scanning Calorimeter analysis (DSC)

The experimental technique used in this investigation was the utilisation of DSC60 to investigate the process of differential scanning calorimetry. The samples under examination were subjected to a controlled heating rate of 10°C per minute under a nitrogen environment. Additionally, the materials were contained within an aluminium pan during the duration of the experiment.

The experiment was conducted with a nitrogen flow rate of 100 ml/min and within a temperature range spanning from 30°C to 300 °C.
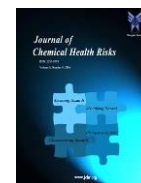
2.16 Scanning electron microscope analysis (SEM)

The surface morphology of fragmented samples was analysed using a scanning electron microscope (SEM) model Philips XL-30, manufactured in the Netherlands. Prior to capturing the micrographs, the samples underwent a process of sputter-coating with gold.

3. Results and Discussions
3.1 Mechanical Properties

For JSTP0, JSTP5, JSTP10, JSTP15, JSTP20, JSTP25, and JSTP30, mechanical properties such as tensile strength, tensile modulus, flexural strength, flexural modulus, and impact strength were investigated.

3.1.1 Tensile properties

From figure 2, it can be seen that optimum results for tensile strength and tensile modulus were obtained for JSTP15. The tensile strength of JSTP0 is 6.28 MPa, JSTP15 is 10.19 MPa, and for JSTP30 it is 5.90 MPa. Tensile strength increases up to a 15% concentration of pine rosin and then decreases (figure 2 (a)). A similar pattern was observed for tensile modulus, which is 263.81MPa for JSTP0, 492.35MPa for JSTP15, and 262.14 for JSTP30 (figure 2 (b)). Pine rosin is mostly composed of rosin acids, which include abietic acid. The structure of abietic acid contains two reactive centers; one is monocarboxylic acid and the other is a hydrophenanthrene ring. The carboxyl group of monocaboxylic acids is responsible for hydrogen bonding with the hydroxyl group of starch.
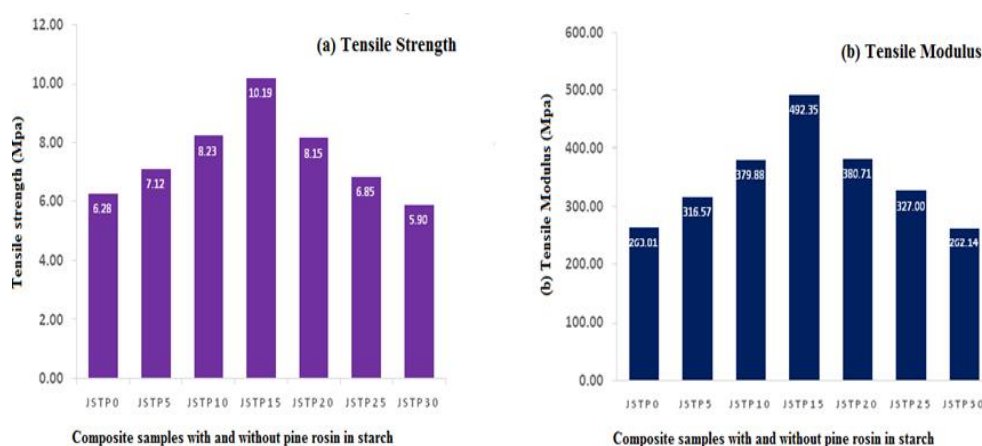

Fig. 2 (a) Tensile strength **(b)** Tensile modulus of different concentration of pine in starch composites

Improvements in tensile strength and tensile modulus may be attributable to the development of new hydrogen bonds between starch and pine rosin and the rigid structure of the rosin acid hydrophenanthrene moiety in the pine rosin modified starch jute composite. Additionally, improved tensile strength and modulus may also be due to the higher molecular weight of pine rosin. From SEM images 8 (b and d), it is determined that there are rougher surfaces on pine rosin modified starch jute composite as compared to unmodified starch jute composite. This indicates that the interfacial bond between jute fibre and pine rosin modified starch matrix has been improved, which is reflected in overall enhanced tensile properties, but these properties deteriorate after 15% of pine rosin concentrations, which may be due to decreased plasticizing effects and increased brittleness of pine rosin.

3.1.2 Flexural properties

Figure 3 shows that the flexural strength and modulus of pine rosin modified and unmodified starch jute composites follow the same trends as tensile strength and modulus. The highest values of flexural strength and modulus were obtained at 15% pine rosin concentration (JSTP15). These are 17.05 MPa and 710.55 MPa, respectively. At 30% pine rosin concentration (JSTP30), flexural strength and modulus are 12.43 MPa and 552.87MPa, respectively, while for unmodified starch jute composite (JSTP0), they are 11.53 MPa and 479.87 MPa, respectively. The improved flexural properties appeared to be due to improved adhesion of the pine rosin modified starch matrix and jute fiber. Better adhesion between fibre and matrix provides improved stress transfer between them (Mohammed et al 2008). The higher molecular weight and compact structure of pine rosin also play an important role in the improvement of flexural properties. But increasing the concentration of pine rosin after 15% affects negatively the flexural properties of the composite. The reason behind this may be the brittle behaviour of pine rosin and the reduced plasticizing effect.
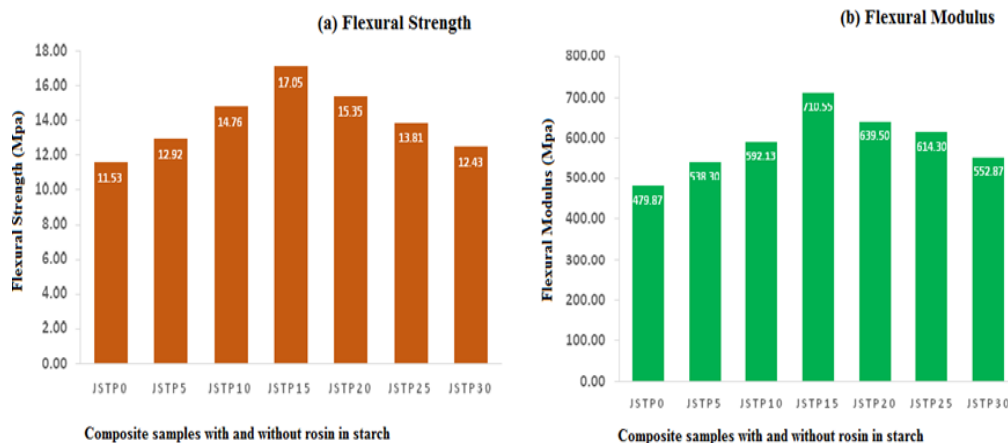
Fig. 3 (a) Flexural strength **(b)** Flexural modulus of different concentration of pine in starch composite

3.1.3 Impact properties

From impact tests, presented by figure 4, it is clear that adding pine rosin to starch for making a matrix to fabricate a jute reinforced composite has significantly improved impact strength up to a 15% concentration of pine rosin. The trend is not increasing throughout the concentration of pine rosin up to 30%; it is an increasing trend up to 15% and then decreasing thereafter. The highest value of impact strength was obtained for JSTP15, at 58.66KJ/m^2, while the lowest value was obtained for a composite having a pine rosin concentration of 30% (JSTP30), at 46.85KJ/m^2. If a comparison has been made between a composite having a 15% pine rosin concentration and a composite without pine rosin, impact strength was improved by around 11KJ/m^2 for the composite of 15% pine rosin. The appearance of increasing trends from 0 to 15% pine rosin concentration reflects that the interfacial bond between pine rosin modified starch matrix and jute fibre also improved along with the compactness of the composite, but after that, the trend ran in reverse direction, which might be due to the decreasing plasticizing effect of pine rosin and increasing brittleness of the composite. In previous research, it has been shown that impact strength increases with increasing plasticizing effect and vice versa [10].
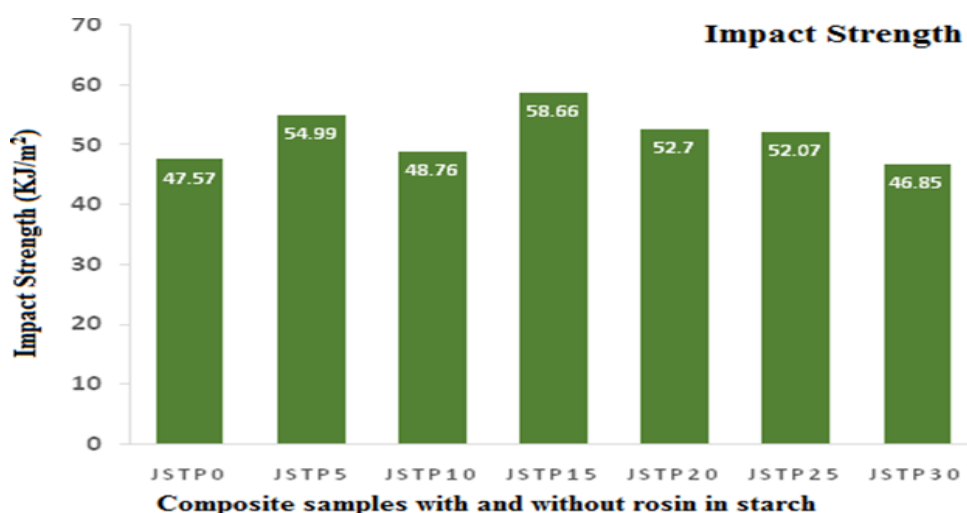

Fig. 4 Impact strength of different concentration of pine in starch composite

3.2 Water absorbency

Values from the evaluation of water absorbency tests with and without pine rosin modified starch jute composite have been listed in table 2, from which it is observed that all the samples of composite absorb water, but the addition of pine rosin affected the water absorbency. Trends suggest that initially the water absorbency of the composite reduces up to 15% pine rosin concentration (79.922% water absorbency for JSTP0 and 53.278% for JSTP15) while it increases thereafter to 30% concentration of pine rosin (93.875% for JSTP30). It happens because of an improvement in

rigidity due to the presence of a hydro phenanthrene moiety in rosin acid, which makes the latter more hydrophobic in nature [11]. The compact structure of pine rosin is another factor that plays an important role as a barrier to water. The decreasing trend of the water-resistant property of composite beyond 15% pine rosin concentration indicates that there may be an insufficient number of hydrogen bonds between the components of matrix and reinforcement.

Table 2 Water Absorbency							
Time (hrs.)	Water absorbency (%)						
	JSTP0	JSTP5	JSTP10	JSTP15	JSTP20	JSTP25	JSTP30
24	79.922	79.146	76.190	53.578	56.652	89.615	93.875

3.3 Chemical resistance property

A chemical resistance test was done for JSTP0 and JSTP15, applying five different chemical solutions such as Benzene, 5% acetic acid, 10% HCL, 10% sodium hydroxide, and 20% sodium carbonate. Percentage weight gain and change in thickness were measured for both composite samples. The results have been listed in tables 3 and 4 for percentage weight gain and change in thickness, respectively. From the results listed in table 3, it can be noticed that weight gain for samples having 15% pine rosin is significantly lower than the sample that has no pine rosin. The highest difference in weight gain was obtained for 5% acetic acid solution (110.50% weight gain for JSTP0 and 28.07% weight gain for JSTP15; that means the difference is around 82%) and the lowest difference in weight gain was obtained for benzene (91.89% weight gain for JSTP0 and 86.59% weight gain for JSTP15; that means a difference of around 5%). The lower decrement in weight gain of JSTP15 in benzene solution may have been due to an improvement in the compactness of the composite because of pine rosin. The highest decrement in the weight gain of JSTP15 in 5% acetic acid solution may have been due to the hydrophobic nature of pine rosin, which also reduces the possibility of interaction between starch and acetic acid due to the phynanthrene structure present in it. The presence of pine rosin in JSTP15 is the cause of the improvement in the compactness of the composite, which also plays an important role in restricting the entrance of acetic acid solution into the composite. The stability of JSTP15 in acetic acid solution is very satisfactory as compared to JSTP0. However, in other solutions, the chemical absorbency of JSTP15 has been reduced significantly, but still, these results are not satisfactory. The higher weight gain of JSTP0 and JSTP15 in 10% HCL solution is due to hydrolysis of starch, but the hydrolysis was restricted due to the intervention of pine rosin in JSTP15, so the weight gain of JSTP15 is comparatively lower than JSTP0.

The loss of weight of JSTP0 and JSTP15 in 10% NaOH solution is due to the high sensitivity of starch and pine rosin towards strong alkalis [9,12]. Percentage weight losses for the two samples are approximately similar. In mild alkali (20% sodium carbonate solution), weight gain is very high for both the composites. This may be due to the affinity of starch and acidic pine rosin towards sodium carbonate solution. The lower weight gain of JSTP15 as compared to JSTP0 may have been due to the improved compactness of the former.

Table 3. Effect of pine rosin on chemical resistance of composite in the form of weight gain						
	0% pine rosin			With 15% pine rosin		
Name of chemicals	0 hrs. weight (gm)	24 hrs. weight (gm)	Weight gain %	0 hrs. weight (gm)	24 hrs. weight (gm)	Weight gain %
Benzene	1.825	3.502	91.89	2.454	4.579	86.59
5% Acetic acid	1.799	3.787	110.50	2.426	3.107	28.07
10% HCL	1.876	3.825	103.89	2.542	4.341	70.77
10% Sodium Hydroxide	2.330	1.882	-19.31	2.488	1.942	-21.94
20% sodium carbonate	1.807	4.288	137.29	2.150	4.114	91.34

3.4 Thickness

Differences in change in thickness were measured as differences in change in swelling. These differences follow the same trend as the differences in weight gain obtained for JSTP0 and JSTP15. Thus, the dimensional stability of JSTP15 has been improved; the highest improvement obtained for 5% acetic acid solution, for which swellings were 2.50mm and 0.81mm for JSTP0 and JSTP15 respectively, which seems quite satisfactory. Improvement in dimensional stability towards the mentioned chemical solution occurred due to the presence of pine rosin in the sample, which created rigidity in the sample and restricted it from swelling. The thickness of samples in sodium hydroxide solution could not be measured because of their dissolution. The results have been plugged into Table 4.

Table 4 Effect of pine rosin on chemical resistance of composite in the form of swelling						
Name of chemicals	0% pine rosin			With 15% pine rosin		
	0 hrs. Thickness (mm)	24 hrs. Thickness (mm)	Swelling (mm)	0 hrs. Thickness (mm)	24 hrs. Thickness (mm)	Swelling (mm)
Benzene	6.28	7.90	1.721	6.28	8.0	1.62
5% Acetic acid	5.00	7.50	1.721	6.19	8.0	0.81
10% HCL	4.98	6.00	1.651	5.85	7.50	1.02
10% Sodium Hydroxide	6.10	Dissolved	-	6.07	Dissolve	-
20% Sodium carbonate	5.00	7.1	2.69	6.1	8.79	2.1

3.5 FTIR, TGA, DSC and SEM characterization

Based on the mechanical performance of the composites, a jute-starch composite with 15% pine rosin was chosen for analysis, with jute-starch without pine rosin serving as a comparative sample. As a result, FTIR, TGA, DSC, and SEM studies were used to describe JSTP0 and JSTP15.

3.5.1 FTIR

The FTIR spectral diagrams of JSTP0 (0% pine rosin) and JSTP15 (15% pine rosin) shown in figure 5 indicate that both composites are comprised of compounds which contain OH groups. For JSTP0, the peak of this functional group is found at around 3302 cm^{-1} in the form of OH stretching. It may have come from starch, glycerol, or jute fibre.[23][28] For JSTP15, this functional group is found at around 3351 cm^{-1} and 2924 cm^{-1} in the form of OH stretching. It may have come from starch, glycerol, jute fibre or carboxylic acid of pine rosin. The peak obtained at 2924 cm^{-1} indicates the presence of OH stretching from the carboxylic acid of pine rosin. The shifting of the OH group from 3302cm^{-1} of JSTP0 to 3351cm^{-1} of JSTP15 indicates that the interaction of both jute fibre and starch with pine rosin has been improved. Therefore, the overall strength of the composite has been found to be improved for JSTP15. It has been observed that there is the presence of an intense peak at 1694cm^{-1} in the FTIR of JSTP15, but this peak is not present in the FTIR of JSTP0. This indicates C=O stretching of carboxylic acid in pine rosin present in JSTP15 but not in JSTP0. Peaks obtained at around 989 cm^{-1} and 1019 cm^{-1} of JSTP0 and JSTP15, respectively, indicate C-O stretching, which may have come from primary and secondary alcohol of the pyranose ring of starch and jute fibre [13].
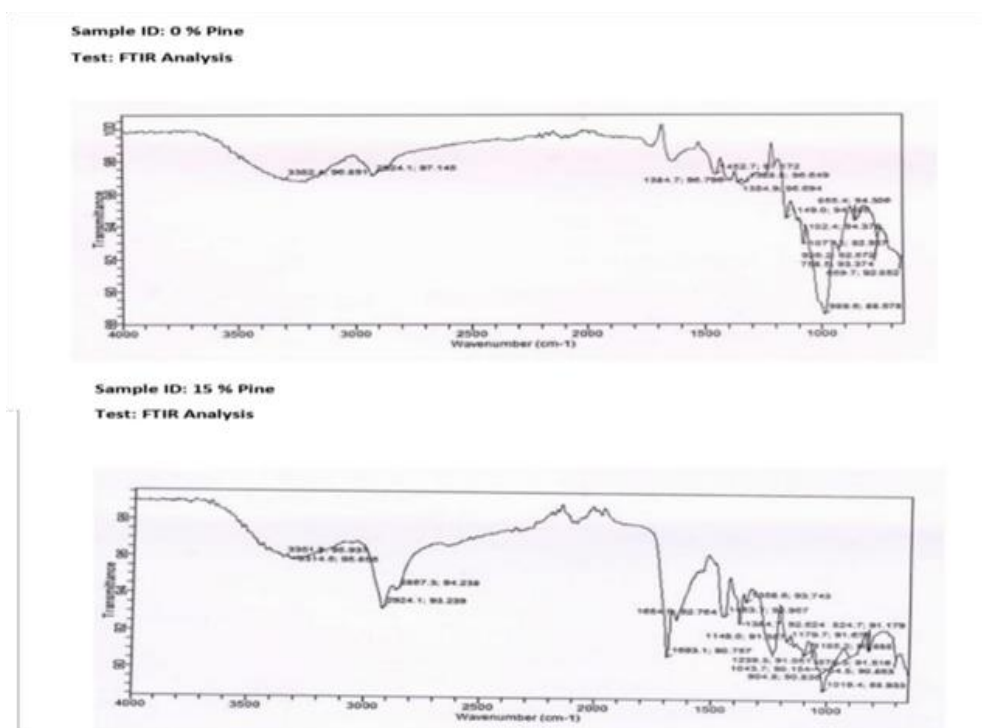
Fig.5 FTIR analysis of JSTP0 and JSTP15

3.5.2 TGA

For analyzing thermal degradation, thermo-gravimetric analysis (TGA) was performed for JSTP0 and JSTP15. Figure 6 and table 5 depict that both the composites exhibit thermal degradation in three stages between 29°C and 500°C. The first stage of JSTP0 degradation occurred between 28°C and 150°C due to evaporation of moisture and unbounded glycerol (Muhammed et al. 2015), the second stage occurred between 150°C and 270°C due to evaporation of bonded glycerol and breaking of the hydrogen bond between jute and starch, and the third stage occurred between 270°C and 500°C due to starch decomposition into gaseous compounds due to presence of carbon, hydrogen and oxygen [14] and degradation of hemicellulose of jute fibre [15].

Similarly, carbon, degradation of JSTP15 occurred between 28°C to 170°C due to evaporation of moisture and unbounded glycerol [16], second stage degradation of JSTP0 occurred between 170°C to 300°C due to evaporation of bonded glycerol [14,17] breaking of

hydrogen bond between jute and starch, and degradation of pine rosin third stage degradation occurred between 270°C to 500°C due to decomposition of starch into gaseous compounds due to presence of carbon, hydrogen and oxygen [14, 18] and degradation of abietic acid, pimaric acid, and some non-acidic materials present in pine rosin [19].

The analysis indicates that JSTP15 exhibits significantly enhanced thermal stability compared to JSTP0. JSTP0 experiences 10% weight loss at 62.39°C, while JSTP15 reaches the same point at 110°C. This difference remains consistent up to 90% weight loss. Notably, for 50% and 60% weight loss, the temperature difference between JSTP15 and JSTP0 is minimal (around 6°C), highlighting substantial thermal stability improvement in JSTP15. This enhancement likely results from increased interaction between pine rosin and starch/jute fiber, reinforced by the robust rigidity and polar hydrogenated phenanthrene ring structure found in pine rosin's rosin acid [20].

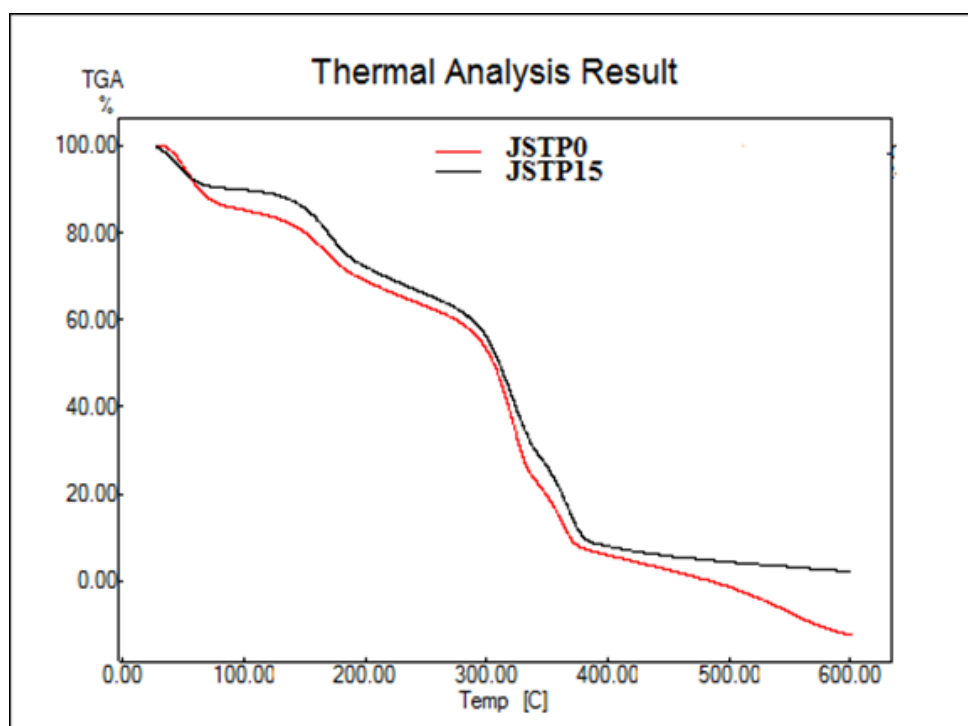| Table 5. Weight loss % against temperature									
Samples	10	20	30	40	50	60	70	80	90
	Temperature °C								
JSTP0	62.39	150.01	191.93	273.89	305.52	318.12	328.15	348.68	368.77
JSTP15	110.00	170.56	219.96	289.99	311.18	324.76	341.54	362.96	382.45

Fig.6 TGA analysis of JSTP0 and JSTP15

3.5.3 DSC

Pictorial representation of Differential Scanning Calorimetry of JSTP0 and JSTP15 has been shown in figure 7. From the diagram, it is indicated that the glass transition temperature (Tg) of JSTP15 has been shifted slightly towards the right as compared to JSTP0, which shows that the thermal stability of JSTP15 has been improved. Glass transition temperature (Tg) for JSTP0 was discovered to be 85.69°C, while glass transition temperature (Tg) for JSTP15 has been found to be 91.49°C, which can be assumed to be a significant improvement in the glass transition temperature. Improvement in glass transition temperature may have been due to high molecular weight, rigid structure of pine rosin which restricts mobility of molecular chains and, additionally, higher intensity of hydrogen bonding among pine rosin, starch and jute fibre also plays an important role in increasing the Tg of JSTP15. This result also gives justification for the results of TGA.
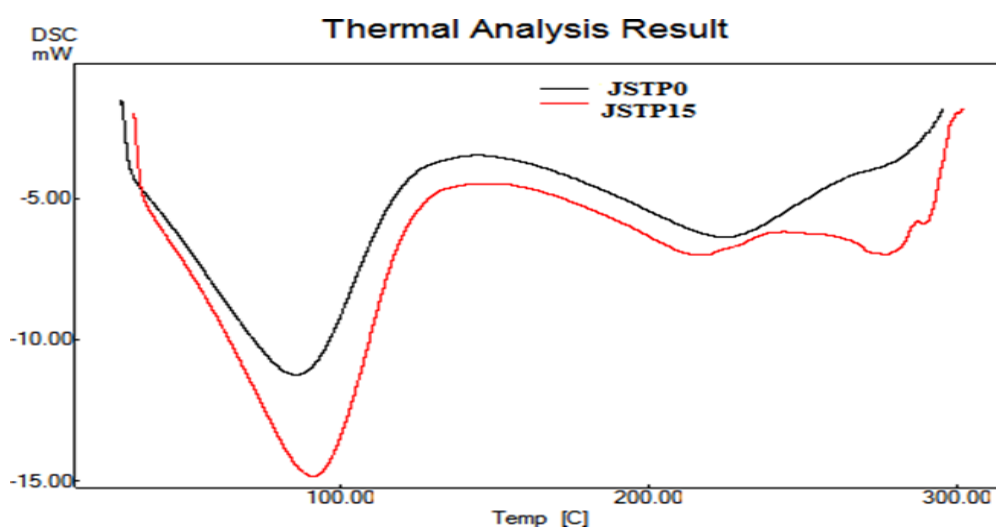

Fig.7 DSC analysis of JSTP0 and JSTP15

3.5.4   SEM

Figure 8 in the SEM analysis displays longitudinal and cross-sectional views of fractured samples: JSTP0 (Fig. 8a, 8c) and JSTP15 (Fig. 8b, 8d). A comparison reveals that the addition of pine rosin to JSTP15 leads to a rougher surface, enhancing adhesion to jute fibers, thus improving mechanical properties. The cross-sectional view further illustrates that JSTP15 (Fig. 8d) is denser, reducing free space and enhancing rigidity and compactness due to the presence of pine rosin. This enhancement positively impacts tensile, flexural, and impact properties.
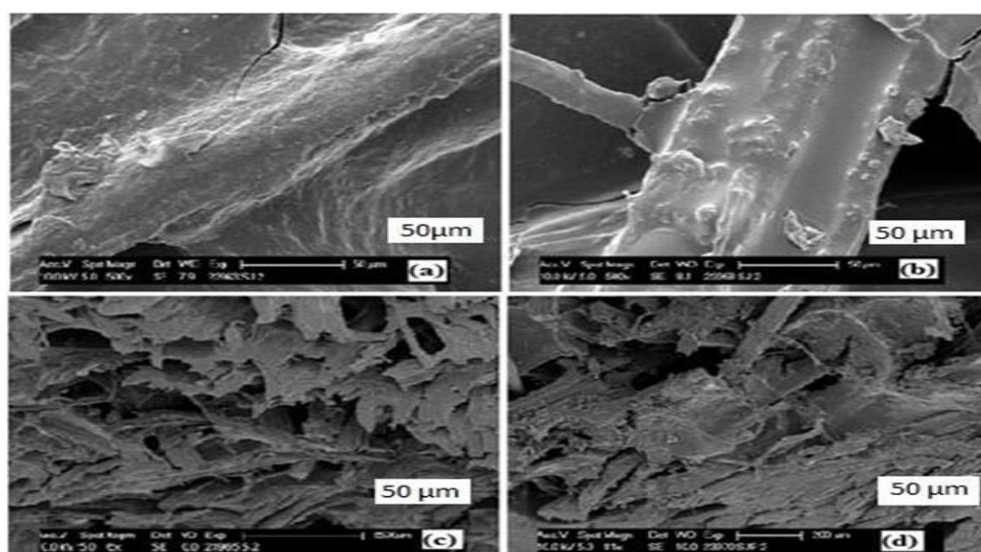

Fig.8 SEM Photographs of JSTP0 and JSTP15

4. Conclusion

This study enhances the properties of jute fiber-starch composites through the introduction of pine rosin. Notably, this modification significantly strengthens the composite material, evident in the superior tensile strength (10.19 MPa) and tensile modulus (492.35 MPa). It also results in improved flexural strength (17.05 MPa), flexural modulus (710.55 MPa), and impact strength (58.66 KJ/m2). Furthermore, the modified composite demonstrates reduced water absorbency, approximately 26% less than the unmodified composite, and enhanced thermal stability with a slower thermal degradation rate. Chemical affinity tests indicate reduced interaction with various chemical solutions, except for 10% Sodium Hydroxide. SEM imaging reveals altered surface morphology due to the presence of pine rosin. In conclusion, optimizing the percentage of pine rosin in starch resin enhances the mechanical, thermal, and water-resistant properties of jute fiber-reinforced composites, rendering them suitable for diverse applications.

Additional Information
Conflicts of interest: None

Payment/services info: All authors have declared that no financial support was received from any organization for the submitted work.

Financial relationships: All authors have declared that they have no financial relationships at present or within the previous three years with any organizations that might have an interest in the submitted work.

Other relationships: All authors have declared that there are no other relationships or activities that could appear to have influenced the submitted work.

References

1. Torres, G., Arroyo, H., Gomez, C. (2007), "Processing and mechanical properties of natural fiber reinforced thermoplastic starch bio composites", Journal of Thermoplastic Composite Materials, 20(2), 207– 223. doi:10.1177/0892705707073945

2. Vilaseca, F., Mendez, A., Pèlach, A., Llop, M., Cañigueral, N., Gironès J. Turon X., Mutjé P. (2007), "Composite materials derived from biodegradable starch polymer and jute strands", Process Biochemistry, 42(3), 329–334. doi: 10.1016/j.procbio.2006.09.004.

3. Mohammed AA, Omran AA, Hasan Z, Ilyas RA, Sapuan SM. Wheat biocomposite extraction, structure, properties, and characterization: A review. Polymers. 2021 Oct 21;13(21):3624.

4. Gupta, M., Srivastava, R., Bisaria, H. (2015), "Potential of jute fibre reinforced polymer composites: A review", International Journal of Fiber and Textile Research, 5, 30-38.

5. Sun, S., Liu, P., Ji, N., Hou, H., Dong, H. (2017) "Effects of various cross-linking agents on the physicochemical properties of starch/PHA composite films produced by extrusion blowing", Food Hydrocolloids, 1-12. doi: 10.1016/j.foodhyd.2017.11.046.

6. Iman, M., Maji, T. (2012), "Effect of crosslinker and nanoclay on starch and jute fabric based green nanocomposites", Carbohydrate Polymers, 89(1), 290–297. doi: 10.1016/j.carbpol.2012.03.012.

7. Maiti, S., Sinha Ray, S., Kundu, A. (1989), "Rosin: a renewable resource for polymers and polymer chemicals", Prog. Polym. Sci, 14(3), 297–338. doi:10.1016/0079-6700(89)90005-1.

8. Muhammed, S., Salit, S., Mohammad, J., Mohamad, I., Japar, S. (2015), "Effect of plasticizer type and concentration on tensile, thermal and barrier properties of biodegradable films based on sugar palm (arenga pinnata) starch", Polymers, 7(6), 1106–1124. doi:10.3390/polym7061106.

9. Coppen, J. (1995), "Gum naval stores: turpentine and rosin from pine resin - non-wood forest products",

10. Kormin, S., Kormin, F., Beg, M. (2019), "Effect of plasticizer on physical and mechanical properties of ldpe/sago starch blend". Journal of Physics: Conference Series, doi:10.1088/1742-6596/1150/1/012032.

11. Vevere, L., Fridrihsone, A., Kirpluks, M., Cabulis, U. (2020), "A Review of Wood Biomass-Based Fatty Acids and Rosin Acids Use in Polymeric Materials", Polymers, 12(11), doi:10.3390/polym12112706.

12. Uthumporn, U., Shariffa, N., Fazilah, A., Karim, A. (2012), "Effects of NaOH treatment of cereal starch granules on the extent of granular starch hydrolysis", Colloid Polym Sci, 290(15), 1481–1491. doi:10.1007/s00396-012-2674-2.

13. George, L. O., Radha, H. R., & Somasekariah, B. V. (2018). In vitro anti-diabetic activity and GC-MS analysis of bioactive compounds present in the methanol extract of Kalanchoe pinnata.

14. Prachayawarakorn, J., Chaiwatyothin, S., Mueangta, S., Hanchana, A. (2013), "Effect of jute and kapok fibers on properties of thermoplastic cassava starch composites", Materials & Design, 47, 309–315. doi:10.1016/j.matdes.2012.12.012.

15. Almeida Prachayawarakorn, J., Chaiwatyothin, S., Mueangta, S., Hanchana, A. (2013), "Effect of jute and kapok fibers on properties of thermoplastic

cassava starch composites", Materials & Design, 47, 309– 315. doi: 10.1016/j.matdes.2012.12.012.

16. Mohammed, L., Ansari, M., Pua, G., Jawaid, M., Islam, S. (2015), "A review on natural fiber reinforced polymer composite and its applications", International Journal of Polymer Science. doi:10.1155/2015/243947

17. Singh, Ha., Singh, J., Singh, S., Dhawan, V., Tiwari, S. (2018), "A brief review of jute fibre and its composites", Materials Today Proceedings, 5(14), 28427–28437. doi:10.1016/j.matpr.2018.10.129

18. Pineda-Gómez, P., Angel-Gil, N., Valencia-Muñoz, C., Rosales-Rivera, A., Rodríguez-García, M. (2014), "Thermal degradation of starch sources: Green banana, potato, cassava, and corn - kinetic study by non- isothermal procedures", Starch/Stärke, 66(7-8), 691–699. doi:10.1002/star.201300210.

19. Sharma, L., Singh, C. (2016), "Composite film developed from the blends of sesame protein isolate and gum rosin and their properties thereof", Polymer Composites, 39 (5), 1480–1487. Doi:10.1002/pc.24088.

20. Söderberg, A., Gref, R., Holm, S., Elmros, T., Hallmans, G. (1990), "Antibacterial activity of rosin and resin acids in vitro", Scandinavian Journal of Plastic and Reconstructive Surgery and Hand Surgery, 24(3), 199–205. doi:10.3109/02844319009041279.