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MANUFACTURING PROCESS AND MATERIAL CHARACTERIZATION OF WOVEN JUTE COMPOSITES

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ABSTRACT

Composite materials are materials that are the combinations of at least two different materials or phases. Jute is a plant that can also be utilized as a composite reinforcing phase to form a composite structure. Recently jute composites are preferred in many areas because of their low cost, low density, ease of availability and renewability. Thermoplastic and thermoset resins can be used as matrix phase to form a jute composite.
In this experimental study; epoxy and polyester resins were combined with several woven jute plies to form composite laminate. Plies were treated first with 2.4 liters of deionized water and 240 grams of sodium hydroxide for 4 hours. After this step, 6 liters of deionized water and 10 % diluted acetic acid were applied to woven ply. Before and after these chemical steps, microstructure changes were observed by the help of an optical microscope. Resin and hardener of polyester and epoxy polymers were prepared in specific ratios before applied to jute fibers. These mixtures were applied to fabric plies by hand lay – up method. After applying polymer mixtures to laminated fabrics, hot press was used for curing at 100 °C, 4 hours and 0.5 ton pressure.
Thickness of the plies was measured before and after the chemical treatment and also after the curing process and hardness of the cured laminate was also measured by using Durometer. Microstructural changes of jute fibers in terms of dimensions were analyzed to see if the chemical treatment played a role in the dimensional changes. Investigation of microstructural changes and physical properties of jute fibers before and after treatment of sodium hydroxide and acetic acid are one of the main focus of this research. It was observed that thicknesses of jute fabric plies were increased after treatment by sodium hydroxide and acetic acid treatments. According to the study, treatment of jute fabric plies with sodium hydroxide has increased their length while treatment of the same fabrics by acetic acid after the sodium hydroxide treatment decreased the same dimension. Another observation was made for the usage of polyester and epoxy for the impregnation of the jute fabrics and also the ease of use for small amounts of polymer. It was seen that more amount of polyester is needed than epoxy to wet the same amount of area and while a high speed mixer could be employed for mixing the two component epoxy, three component polyester cannot be mixed with the same device and hand stirring should be employed for this purpose which uses low mixing rate.

Keywords: Jute fiber, Natural fiber composites, Microstructure

DOKUMA JÜT KOMPOZİTLERİN ÜRETİM SÜREÇLERİ VE MALZEME KARAKTERİZASYONLARI

ÖZET

Kompozit malzemeler en az iki malzeme veya fazdan oluşan malzemelerdir. Jüt de kompozit bir malzeme oluşturmak için güçlendirme fazı olarak kullanılabilen bir bitkidir. Günümüzde jüt kompozitler düşük maliyet, düşük yoğunluk, kolay bulunabilirlik ve yenilenebilir malzeme olma özelliklerinden dolayı bir çok alanda tercih edilmektedirler. Termoset ve termoplastik reçineler jüt fiber kompozitleri oluşturmak için matris fazı olarak kullanılabilirler.
Bu deneysel çalışmada; epoksi ve polyester reçineler kompozit plaka oluşturmak için dokuma jüt katmanlarla birleştirilmişlerdir. Katmanlar ilk önce 2.4 litre damıtılmış su ve 240 gram sodyum hidroksit ile 4 saat boyunca kimyasal işleme tabi tutulmuşlardır. Bu adımdan sonra, 6 litre damıtılmış su ve % 10 oranında seyreltilmiş asetik asit dokunmuş katmanlara uygulanmıştır. Uygulanan bu kimyasal işlemlerin öncesi ve sonrasında optik mikroskop kullanılarak mikroyapı incelemesi yapılmıştır. Polyester ve epoksi polimerlerin reçine ve sertleştiricileri jüt katmanlara uygulanmadan önce belirli oranlarda karıştırılarak hazırlanmışlardır. Bu karışımlar kumaş katmanlara el yatırma yöntemiyle uygulanmışlardır. Serili kumaşlara polimer karışımları uygulandıktan sonra sıcak pres altında 100°C'de 4 saat ve 0.5 ton değerinde kuvvet uygulanmıştır.
Katmanların kalınlıkları kimyasal işlemler öncesi ve sonrasında ölçülürken katmanlı yapının sertliği de Durometre kullanılarak ölçülmüştür. Jüt fiberlerin boyutsal bazdaki mikroyapı değişimleri de incelenerek kimyasal işlemlerin boyutsal değişimleri etkileyip etkilemediği incelenmiştir. Jüt fiberlerin mikroyapısal ve fiziksel özelliklerinin sodyum hidroksit ve asetik asit işlemleri

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öncesi ve sonrasındaki değişimleri bu çalışmanın ana odaklarından biridir. Jüt kumaş katmanların kalınlıklarının sodyum hidroksit ve asetik asit işlemlerin sonucunda arttığı gözlemlenirken, uzunluklarının sodyum hidroksit işlemi sonucu arttığı, bu işlem sonrasında yapılan asetik asit işlemi sonrasında ise aynı katmanların uzunluklarının ise azaldığı görülmüştür. Bir diğer gözlem de jüt kumaşların polyester ve epoksi kullanarak doyurulması ve az miktardaki polimerin kullanım kolaylığı üzerine yapılmıştır, Aynı miktardaki alan için daha fazla polyester kullanılırken daha az miktardaki epoksi ile aynı alanın ıslatılabildiği görülmüş, iki komponentli epoksinin karıştırılması yüksek hızlı karıştırıcı ile mümkün olabilmişken üç komponentli polyester ise aynı cihaz kullanılarak karıştırılamamış ve el ile daha düşük hızlarda karıştırılarak hazırlanmıştır.

Anahtar kelimeler: Jüt fiber, Doğal fiber kompozitler, Mikroyapı

1. INTRODUCTION

Today composite materials are favored in many areas because of their lightweightness, high strength over weight ratio, corrosion resistance and many others to say. Composite materials can also be produced by using natural materials as well. These natural materials can be in the form of fiber, particle and powder and are used as reinforcing phase inside a composite material. The application areas of composite materials range from military to civil areas such as aircrafts, boat hulls, sporting goods, automobiles just to mention a few. Composite materials in which their reinforcing material is originated from natural resources are becoming more attractive because of their recyclability, availability and cost. They have some drawbacks such as high water absorption, lower durability and fire resistance that should be improved.

One of the most important advantages of using polymers as the matrix material is the ease of use, low cost and low density. In some applications, the properties of polymers are improved by using fillers, powders and fibers to fit the high strength and high modulus requirements [1].

The natural resources can be from plants, vegetables, trees and animals. Depending on the source; bast, leaf, seed, fruit, wood, stalk and grass are the main categories of natural fibers. Sisal, jute, flax, hemp and kenaf are some of the instances of the bast fibers. Mechanical properties of some natural fibers are given in Table 1 that includes jute fiber which is the main subject of the recent study.

Cellulose, hemicellulose, lignin, waxes, and several water – soluble compounds are the main components of the natural fibers [3]. Jute fiber is a natural fiber used as reinforcement for making composites because of its desirable properties which are low cost, low density, easy accessibility and renewability. In addition to that properties, jute fibers have relatively high specific strength and elastic modulus. Jute fiber is generally preferred in textile industry to produce bags, sack, mats and carpets.

Alluvial soil and mild weather with high humidity rates of up to 80 % are needed to raise jute plant. Temperature variations could be from 20 °C – 40 °C [4]. Jute fibers have relatively low density values ranging from 1.3 to 1.49 g/cm^3. Strength of jute fibers depends on the coarseness of the jute plant. Elongation at break is between 1.0 to 1.8 % for jute fibers [5].

A jute fiber fabric can be processed by using thermoset or thermoplastic polymers. Thermoplastic polymers show small or no reticulation property and they melt easily [6]. Cured thermosets will not melt easily and flow, but they once be cured then they cannot be reshaped [7]. Thermosets have several advantages over thermoplastics; they are more thermally stable and chemically resistant and have higher glass transition temperatures than thermoplastics [8]. On the other hand, thermoplastics have low processing cost. They can be remelted and reshaped. Thus, by easily reshaping, flexibility and recyclability are desirable evidence of thermoplastics. Thermoplastics also show good mechanical properties [9]. Polyethylene, polyvinyl chloride, nylon, polystyrene, polyolefin and polypropylene are types of thermoplastic polymers [10]. Some properties of epoxy, polyester and vinyl ester are given in Table 2.

Alkaline treatment removes fiber constituents including hemicellulose, lignin, pectin, fat and wax which exposes cellulose and increases surface roughness to improve interfacial bonding with the reinforcing phase [12]. The treatment of natural fibers by sodium hydroxide (NaOH) is widely being used to modify the cellulosic molecular structure [13]. It is the most commonly used chemical compound for cleaning the plant fiber surfaces [14]. NaOH changes the fine microstructure of the native cellulose 1 to cellulose 2 which are two of the four polymorphs of cellulose by alkalization process.

X.Y. Liu and G.C. Dai have studied the effects of sodium hydroxide and maleic anhydride – grafted propylene (MPP) emulsion onto jute fiber mat. Their process was based on the film stacking method. By using this process they have found that surface of jute fiber was uneven [15]. Another study was made by A.C. Karmaker and J.A. Youngquist by using jute fiber as reinforcing material and polypropylene as the matrix material. They also added MPP to investigate the mechanical behavior of the composite structure. They realized that MPP improved the performance of the composite material [16]. Al – Mobarak et al. studied jute mat reinforced polyvinylchloride (PVC) composite by treating the fibers with acetic acid solution. It has been shown that tensile strength, flexural strength, Young's modulus and tangent modulus of acetic acid treated jute composite structure were observed to be higher than those of non-treated jute composite structure. At the same time acetic acid treatment of jute mat improved the hydrophobic feature of the jute mat [17].

Table 1. Mechanical properties of some natural fibers [2]

Type of Fiber	Specific Gravity	Tensile Strength (MPa)	Elastic Modulus (GPa)	Specific Modulus (GPa/g/cm^3)
Jute	1.3	393	55	38
Sisal	1.3	510	28	22
Flax	1.5	344	27	50
Sun hemp	1.07	389	35	32
Pineapple	1.56	170	62	40

Table 2. Some properties of epoxy, polyester and vinyl ester [11]

Property	Epoxy	Polyester	Vinyl ester
Density (g/cm^3)	1.1-1.4	1.2-1.5	1.044
Modulus of elasticity (GPa)	3-6	2.0-4.5	3.2
Tensile strength (MPa)	35-100	40-90	80
Thermal expansion (ºC)	50-300	50-110	-
Water absorption (%)	0.1-0.4	0.1-0.3	0.150

The aim of this study is to see the effects of several chemicals on the physical and microstructural properties on the jute fibers. Two different thermoset polymers were also tried to assess their compatibility for hand lay – up method and hot press manufacturing process. Hand – lay up method was employed by using a paint brash for the application of epoxy and polyester polymers. Compared with some other fibers, jute fiber is a coarse fiber type and is hard to work with some polymer types. Sodium hydroxide and acetic acid have been used to investigate the improvements of the chemical treatments on the microstructure and physical properties. Microstructural changes of the fibers were analyzed by using an optical microscope and physical changes were recorded by using durometer and caliper.

2. EXPERIMENTAL STUDIES

2.1 Sample Preparation

Jute fiber fabrics used in this study have a mass of 250 gr/m^2 which are loosely woven. The fabrics were cut to 23 cm × 23 cm dimensions and a total of 5 plies were prepared. These fabrics were marked in three regions with black, red and blue colors in order to examine these three regions under the optical microscope. In Figure 1, these three regions marked with black, red and blue colors are shown, as can be seen from the figure, the regions are all marked around the fabric ply number "3".

The masses of jute fiber fabrics were measured by precise scale. The fabrics were kept in deionized water for 30 minutes to remove dirt from the surfaces. Then they dried for one day at room temperature. After the cleaning process, the fabrics were investigated by using an optical microscope to observe microstructural properties. Specific points on several fabrics that were marked with black, red and blue colors were measured to see the dimensional changes before and after the chemical treatments.

2.2 Sodium Hydroxide and Acetic Acid Treatments

In this study 10:1 ratio was used for sodium – hydroxide and deionized water solution. 240 g of NaOH was mixed with 2.16 liters of deionized water to come up to 2.4 liters solution and the fibers were waited to stand in the solution for 4 hours and then

fabrics were kept in deionized water for cleaning. After the rinsing process the fabrics dried at room temperature for 24 hours. After sodium hydroxide treatment, optical microscopy was used to observe changes in dimensions of the fibers.

Figure 1. Jute fabric ply number "3" painted with three different colors

For acid treatment, 5 liters of 10 % acetic acid (CH_3COOH) was prepared. The diluted acid solution was poured into washtubs and jute fibers were soaked inside the solution. The fabrics were treated with this acid solution for 30 minutes before being rinsed with deionized water and then waited for 2 days at room temperature. In Figure 2, jute fabric plies inside the acid solution can be seen. Thicknesses of jute plies were measured by caliper before and after the chemical treatments and recorded for comparison. The manufacturing method plays an important role in the physical properties of composite structures.

2.3 Material Characterization for Jute Fabric Plies

Microstructural characterization of jute fibers before and after NaOH treatment and after acetic acid treatment have been carried out with the help of optical microscope by measuring their dimensions to see the effects of the chemical treatments on the microstructural dimensions. The measured dimensions were recorded and given in table format. Optical microscope images were also provided.

Figure 2. Jute fabric plies inside the acetic acid solution

2.4. Manufacturing of Jute Composite Plates

2.4.1. Polyester resin preparation

Unsaturated polyester resin was used as the matrix material for the lamination process. The hand lay – up process was adopted to impregnate the resin to the fabrics. The unsaturated polyester resin's mechanical properties are given in Table 3. A mixture of 100 g of polyester with 0.2 g of cobalt octoate as accelerator and 1 g of mek – p as hardener was prepared before lay – up. After putting the polyester to a small bin, first accelerator was added and stirred with a glass stick for 3 minutes and then hardener was added inside the resin – accelerator mixture. An additional 3 minutes was needed to stir the fluids to obtain a homogenous mixture.

Table 3. Mechanical properties of the unsaturated polyester resin used in the study

Tensile strength (MPa)	Elongation at break (%)	Flexural strength (MPa)
60	1.5	110

2.4.2. Epoxy resin preparation

Jute fabric ply number 5 was cut into pieces of 10 cm x 10 cm to their final dimensions. 40 g of resin and 10 g of hardener was mixed as 4:1 ratio of resin to hardener was indicated by the manufacturer. To mix the resin – hardener mixture, instead of stirring the mixture by hand, Speedmixer™ was utilized. Epoxy resin and hardener was weighted by a precise scale and Speedmixer™ was used at 3000 rpm for 2 minutes to obtain a homogenous mixture. Figure 8 shows the resin – hardener mixture and the Speedmixer™ equipment which employs dual asymmetric centrifugal technology.

2.4.3. Hand lay – up technique

Resin material can be transferred to the woven fabrics in several ways; infusion, resin transfer molding, hand lay – up method and etc. In this study, researchers preferred hand lay – up process because of its simplicity and relatively low cost. After the transfer of resin to the woven fabrics by using hand lay – up method, hot press was employed for the curing process. Before the application of the hand lay – up technique, metal plate was cleaned with the help of acetone. Release agent was applied on both surfaces and waited for fifteen minutes.

The lamination for the jute woven fabrics using epoxy resin was carried out by using four plies. Before the first ply was put to the plate, epoxy resin mixture was applied to the surface of the plate by a brush to be sure that the first ply would stick on the surface. After the lamination of the first ply, each play was put on the top of the previous ply and epoxy resin mixture was applied. After the lamination of the last ply, the laminated plies were put to a roasting bag to prevent the leaked resin to the upper and lower tabs of the hot press. Figure 3 shows the lamination process and hot press used in the curing process of the composite plates.

After completing the lamination process, laminated plies consisting four layers of jute woven fabrics were put into the hot press machine. The force applied by the hot press was adjusted to 0.5 tons. The curing cycle of the laminated plies is shown in Figure 4. As seen from the figure, the temperature on the surfaces of the upper and bottom tabs of the hot press reached from ambient temperature to 100 °C and the laminated plies stayed at this temperature for four hours. This time is needed for the fully curing of the laminated plies to become a composite plate. After this process, the composite plate was taken from the hot press before it reaches the ambient temperature. The hardness and thickness measurements were carried out. Hardness measurements were obtained from different points of the composite plates by using a Durometer and arithmetic mean values were taken as the hardness value of each plate.

(a) (b)

Figure 3. (a) Lamination process (b) Hot press

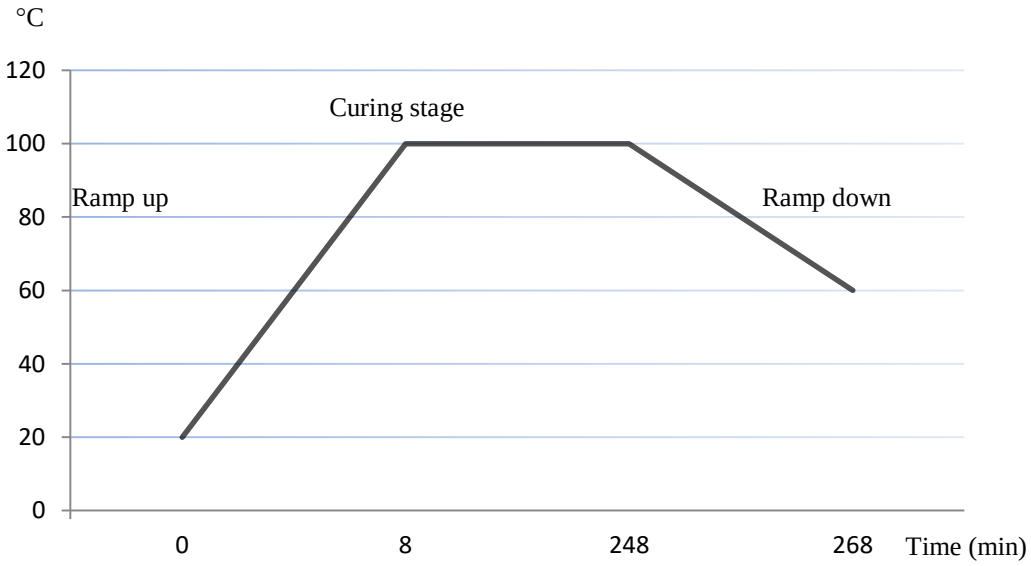


Figure 4. Curing cycle of the laminated plies

3. RESULTS AND DISCUSSION

Table 4 shows the comparison of untreated and treated jute fabrics with regard to the thickness of each ply. It was observed that thicknesses of jute fabrics were increased after treatment by NaOH and acetic acid. The thickness of most of the plies increased by more than 100 %.

The microstructural properties of the fibers have been observed by using an optical microscope and the results have been recorded. To make this happen, ply number 4, blue colored spot was chosen for inspection. Several other spots on different plies were also painted but only ply number 4, blue colored spot's dimensional changes were recorded to see the changes. In Table 5, the dimensional changes of this colored spot before NaOH treatment, after NaOH treatment and after acedic acid treatment are given. Based on the data given in Table 5, the following observations were obtained:

- The length of blue colored spot belonging to ply number 4, has seen an increase in length after NaOH treatment of about 27 %, while its length has decreased of about 15 % after acetic acid treatment. The fibers were first treated by diluted water and then by NaOH and the last treatment was made by using acetic acid.

The following figures (Figure 5, 6, 7) show ply number 4, black red and blue colored spots under optical microscope before and after chemical treatments. It was observed that chemical treatment with NaOH enlarged the fibers in longitudinal direction while acetic acid treatment dwindled the fibers. The thickness of the fibers regardless of basic or acidic chemical treatments increased.

The hardness and thickness values were recorded after the curing process. Table 6 shows the thickness of cured composite plate in which epoxy was used as the matrix material while Table 7 shows the cured composite plate in which the polyester resin was used as the matrix material. To obtain the thickness of each composite plate, five measurements from different regions of the plates were taken and arithmetic average was calculated. It can be seen from the Tables 6 and 7 that the thickness of the cured composite plate using polyester resin is much higher than the cured composite plate using epoxy resin. The average thickness of cured composite plate using polyester resin is 6.44 mm while that of cured composite plate using epoxy resin is 2.57 mm.

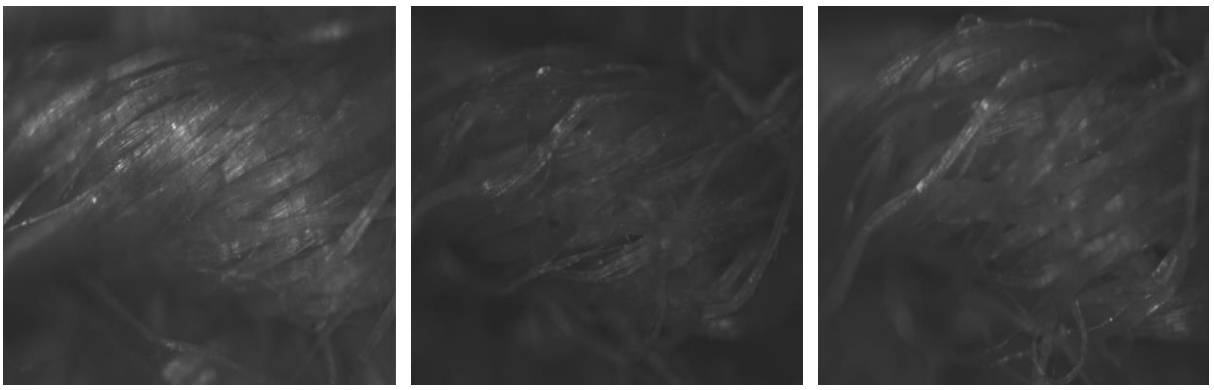
Another property measurement of the cured composite plate was the hardness. The hardness of the plates were measured by using a Durometer and Shore D scale was utilized. Table 8 shows the measured hardness values of epoxy resin used plate while Table 9 shows the measured hardness values of polyester resin used plate.

Table 4. Comparison of untreated and treated jute fabric thickness

Ply number	Thickness of untreated jute fabric (mm)	Thickness of NaOH and acetic acid treated jute fabric (mm)
1	0.60	1.4
2	0.63	1.5
3	0.67	1.4
4	0.65	1.1
5	0.62	1.4
Average	0.63	1.3

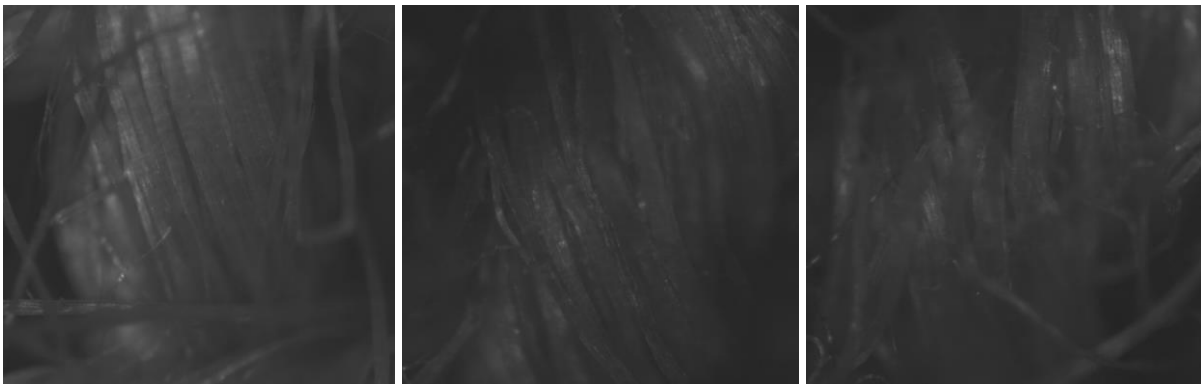
Table 5. Microstructural properties of a jute fiber before and after chemical treatments

Ply number	Color of spot	Chemical treatment	Length (µm)
4	Blue	Before NaOH	798.1
4	Blue	After NaOH	1015
4	Blue	After acetic acid	859.2



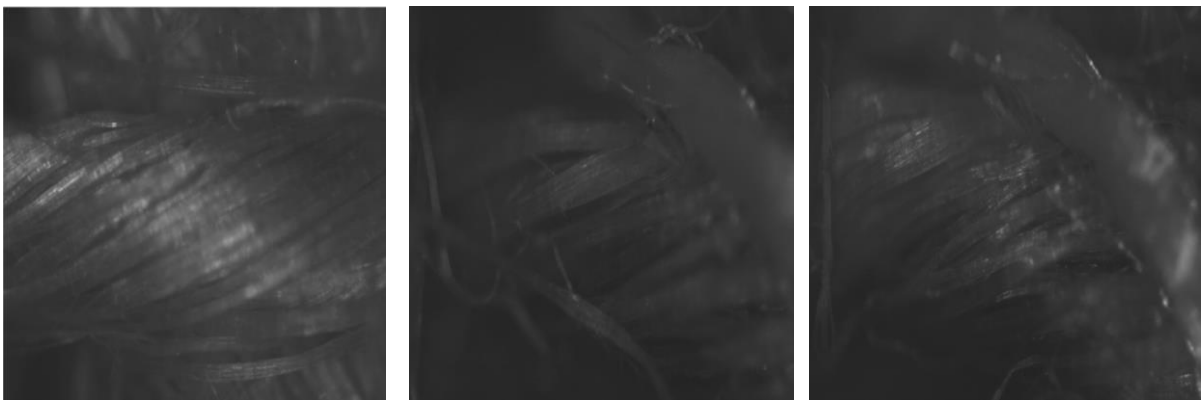
(a) (b) (c)

Figure 5. Microscopic images of ply number 4 black colored spot (a) before NaOH treatment (b) after NaOH treatment (c) after acetic acid treatment



(a) (b) (c)

Figure 6. Microscopic images of ply number 4 red colored spot (a) before NaOH treatment (b) after NaOH treatment (c) after acetic acid treatment



(a) (b) (c)

Figure 7. Microscopic images of ply number 4 blue colored spot (a) before NaOH treatment (b) after NaOH treatment (c) after acetic acid treatment

Table 6. Thickness measurements of cured composite plate using epoxy resin

Caliper measurement	Thickness of cured composite plate (mm)
1st region	2.60
2nd region	2.52
3rd region	2.51
4th region	2.58
5th region	2.68
Average	2.57

Table 7. Thickness measurements of cured composite plate using polyester resin

Caliper measurement	Thickness of cured composite plate (mm)
1st region	6.80
2nd region	6.30
3rd region	6.5
4th region	6.2
5th region	6.4
Average	6.44

Table 8. Durometer measurements of epoxy resin used plate

Durometer Measurement	Shore D Hardness
1st region	81
2nd region	75
3rd region	73
Average	76.3

Table 9. Durometer measurements of polyester resin used plate

Durometer Measurement	Shore D Hardness
1st region	75
2nd region	85
3rd region	80
4th region	82
5th region	80
Average	80.4

4. CONCLUSION

In this experimental study, a composite plate composed of jute fabric/epoxy and another composite plate composed of jute fabric/polyester were manufactured and several properties of uncured jute fiber fabrics and cured composite plates were investigated. Thickness of each untreated and chemically treated jute fabrics were recorded as well as the thickness of two cured composite laminate, microstructure of some fibers were investigated to see the effects of chemical treatment by using an optical microscope and dimensions were measured. Hardness of two cured composite laminate were also measured by using a Durometer. It was seen that thickness of jute fabrics increased considerably after chemical treatments namely NaOH and acetic acid. Although the same process was applied to both of the uncured composite laminae, the thickness of cured polyester resin laminate is bigger than that of cured epoxy resin laminate. It is considered that in order to wet the surfaces of the chemically treated fabrics by hand lay – up process, more polyester wetted the surfaces of the fabrics while less epoxy was enough to wet the surfaces of the jute fabrics. The hardness of the cured polyester resin laminate was obtained higher than that of cured epoxy resin laminate, the difference between two values are only 5 %. This difference shows a slight change between the hardness of two plates. One of the point between using two resin systems is as polyester has three components (resin, hardener, accelerator) compared to epoxy which has only two components (resin and hardener); for small amounts of required resin, it is preferable to use epoxy resin instead of polyester. It is also concluded that chemical treatment by using NaOH increases the dimensions of the jute fibers while chemical treatment by using acetic acid decreases the dimensions of the fibers.

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