IZMIR KATIP CELEBI UNIVERSITY ★ GRADUATE SCHOOL OF SCIENCE AND ENGINEERING

PRODUCTION AND CHARACTERIZATION OF ALTHEA OFFICINALIS L. (MARSHMALLOW) FIBER REINFORCED POLYESTER COMPOSITES

M. Sc. THESIS

Ahmet Çağrı KILINÇ

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Thesis Advisor: Assoc. Prof. M. Özgür SEYDİBEYOĞLU

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İZMİR KATİP ÇELEBİ ÜNİVERSİTESİ ★ FEN BİLİMLERİ ENSTİTÜSÜ

ALTHEA OFFİCİNALİS L. (HATMİ) LİFİ KATKILI POLYESTER KOMPOZİTLERİN ÜRETİMİ VE KARAKTERİZASYONU

YÜKSEK LİSANS TEZİ

Ahmet Çağrı KILINÇ

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Malzeme Bilimi ve Mühendisliği Anabilim Dalı

Tez Danışmanı: Doç. Dr. M. Özgür SEYDİBEYOĞLU

OCAK 2016

Ahmet Çağrı KILINÇ, a **M. Sc.** student of **Izmir Katip Celebi University** student ID Y120111015, successfully defended the **thesis** entitled "**Production and Characterization of Althea Officinalis L. (Marshmallow) Fiber Reinforced Polyester Composites**", which he prepared after fulfilling the requirements specified in the associated legislations, before the jury whose signatures are below.

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Date of Defense   : 25.01.2016

To my family,

ACKNOWLEDGEMENTS

25 January 2016   Ahmet Çağrı KILINÇ

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Abbreviations

FTIR	Fourier Transform Infrared Spectroscopy
TGA	Thermogravimetric Analysis
DTA	Differential Termogravimetric Analysis
SEM	Scanning Electron Microscopy
XRD	X-Ray Diffraction
UP	Unsaturated Polyester
CI	Crystallinity Index
MMC	Metal Matrix Composite
CMC	Ceramic Matrix Composite
PMC	Polymer Matrix Composite
AOL	Althea Officinalis L.

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I_{002} Maximum intensity value of crystalline phase peak
I_{am} Minimum intensity value of amorphous phase

PRODUCTION AND CHARACTERIZATION OF ALTHEA OFFICINALIS L. (MARSHMALLOW) REINFORCED POLYESTER COMPOSITES
SUMMARY

In this study Althea officinalis l. (marshmallow) fiber was used as reinforcement to produce polyester matrix composites. A. officinalis L. was obtained from Mordoğan, Izmir (Turkey). Extraction of fibers from stem was done by water and chemical (NaOH) retting. After extraction process mechanical and thermal properties of althea fibers were measured and morphological structure was examined. Crystallinity indexes (CI) of fibers were determined by Rigaku D-max-2200-PC XRD (X-ray diffractometer); surface morphologies of fibers and fractured regions of composites were determined and investigated by JEOL-JJM 6060 model SEM. Althea fiber/polyester composites were produced by conventional hand lay-up method. Althea fiber reinforced composite specimens were prepared with different weight fractions such as 5%, 10%, and 20%. After preparation process of composite materials, test specimens were produced by machining of composite plates. Mechanical; tensile and flexural properties of composites were examined.

The results showed extraction of althea officinalis fibers by alkali boiling in a solution including 5% NaOH led to removal of surface impurities and improved tensile strength and cellulose crystallinity about 37% and 74% respectively. Also FTIR analyses showed that alkali boiling caused change of chemical composition by decreasing and/or disappearing of some peaks which belong to hemicellulose and lignin. Densities of water and chemically retted fibers was determined as 1,26 g/cm^3 and 1,34 g/cm^3 respectively.

For composite materials, increasing fiber loading and fiber length resulted in increasing tensile and flexural strength. Tensile strength was almost three times higher for 20% loading of 50mm fibers length than neat polyester resin.

ALTHEA OFFICINALIS L. (HATMİ) LİFİ KATKILI POLYESTER KOMPOZİTLERİN ÜRETİMİ VE KARAKTERİZASYONU
ÖZET

Bu çalışmada Althea Officinalis L. (Hatmi) lifleri polyester kompozitler üretilmesinde takviye malzemesi olarak kullanılmıştır. Althea officinalis l. bitkisi Mordoğan, İzmir'den (Türkiye) toplanmıştır. Lif eldesi suda havuzlama ve kimyasal (NaOH) çıkarım ile yapılmıştır. Çıkarım işleminin ardından althea liflerin mekanik, ısıl ve morfolojik özellikleri incelenmiştir. Liflerin kristalinite indeksi Rigaku D-max-2200-PC XRD (X-ışını difraktometresi) kullanılarak; liflerin yüzey morfolojisi ve kompozitlerin kırılma yüzeyleri JEOL-JJM 6060 model SEM kullanılarak incelenmiştir. Althea lifi/polyester kompozitleri el yatırması yöntemiyle üretilmiştir. Althea lifi takviyeli kompozit numuneler %5, %10 ve %20 olmak üzere farklı ağırlık oranlarında üretilmiştir. Kompozit malzemelerinin üretilmesinin ardından kompozit plakaların işlenmesiyle test numuneleri üretilmiştir. Kompozitlerin mekanik; çekme ve eğilme özellikleri incelenmiştir.

Sonuçlar althea officinalis liflerinin %5 NaOH içeren çözeltide kaynatılmasının yüzey empüritelerinin giderilmesine, çekme dayanımı ve kristalinitenin sırasıyla %37 ve %74 artmasına neden olduğunu göstermiştir. Ayrıca FTIR analizleri göstermiştir ki althea liflerinin %5 NaOH içeren çözeltide kaynatılmasıyla hemiselüloz ve lignine ait bazı piklerin şiddetleri azalmış ve/veya pikler yok olmuştur. Suda havuzlanmış ve kimyasal olarak çıkarılmış liflerin yoğunlukları sırasıyla 1,2584 g/cm^3 ve 1,3378 g/cm^3 olarak bulunmuştur.

Kompozit malzemeler açısından bakıldığında ise artan lif takviye oranına ve artan lif uzunluğuna bağlı olarak çekme ve eğilme dayanımları artış göstermiştir. Çekme dayanımı, 50mm lif boyutlu %20 takviye oranında takviyesiz polyestere göre neredeyse üç kat daha yüksektir.

1. INTRODUCTION

1.1 Background

Environmental concern and depletion of petroleum resources have stimulated researchers and industries to use sustainable materials instead of conventional synthetic materials. In this context usage of new materials containing natural components like natural fibers have great importance in composite materials [1, 2]. Besides mechanical and physical properties such as good specific modulus values, low density, considerable toughness properties of natural fibers [3,4], low cost, biodegradable, recyclability, non-toxicity and easy accessibility properties are also attractive side of them [5,6]. These properties give an opportunity to use natural fiber reinforced composite products in various industries such as automotive, building, and furniture [7].

Chemical composition of a fiber is important because quantity of the components in it, effect mechanical properties of the fiber. Lignin, cellulose, and hemicellulose are main components of plant fibers and remaining components are pectin and wax substances [8]. Bismarck et al. (2005) indicated that increasing cellulose content of fiber results in increase of tensile modulus and tensile strength [9].

Various common plant fibers such as jute, flax, hemp, banana, sisal etc. have been investigated by many researchers owing to their availability and good properties. Some other studies advance the feasibility to use novel natural fibers, such as napier grass [10-12], snake grass (sansevieria ehrenbergii) [13], cissus quadrangularis [14], arundo donax [15], ferula communis (chakshir) [16], lygeum spartum l. [17] fibers as reinforcement for composite materials.

Under the light of this trend, novel natural fiber; chemically extracted althea officinalis l. fibers were used in this study for production of natural fiber reinforced composites. Till now, previous works which investigate the effect of extraction method on chemical composition, properties and structure of althea officinalis l. (AOL) fibers and composite application of AOL fibers are not present.

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1.2 Composite Materials

Composite materials are defined as "multiphase materials obtained through the artificial combination of different materials in order to attain properties that the individual components by themselves cannot attain" [18]. Many composite materials are composed of at least two phases; one is called the matrix, which is continuous and surrounds the other phase, and the other one is often called the dispersed phase [19]. Composite materials are classified according to both their matrix materials or dispersed phase. Composite materials are divided into three groups according to type of matrix material as metal matrix composites, ceramic matrix composites, and polymer matrix composites. Figure1.1 shows the classification of composite materials according to type of matrix.
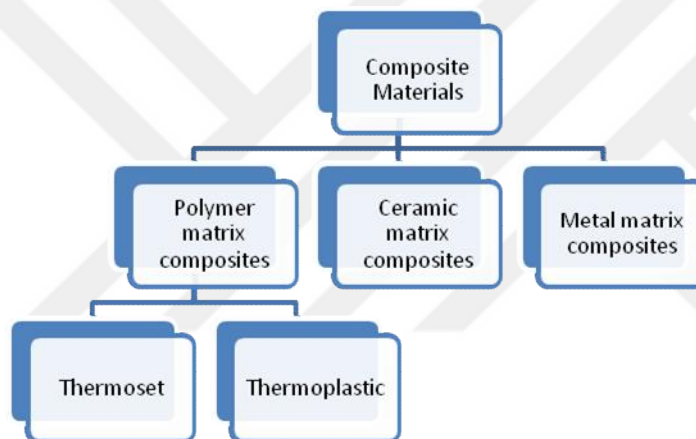
Figure 1.1: Classification of composite materials [19].

Metal matrix composites (MMCs): Metal matrix composites are defined as "combinations of two or more materials (one of which is a metal) where tailored properties are achieved by systematic combinations of different constituents" [20]. Metal matrix composites are used in production of electronic devices, some parts of automobiles and spacecrafts because of resistance to external effects and high melting temperature. But high density, high production temperature and tendency to corrosion of reinforcement-matrix interface are significant drawbacks for metal matrix composites.

Ceramic matrix composites (CMCs): Structural and functional advanced ceramics are used for ceramic matrix composites such as B_4C, Al_2O_3, SiO_2, SiC. Ceramics do not show plastic deformation, they are rigid and brittle. They have high melting temperature and low thermal and electrical conductivity. Armors composed of

ceramic sandwich (laminated) structures and various military equipments are main utilization areas of ceramic matrix composites [21].

Polymer matrix composites (PMCs): Polymer composites have numerous significant properties such as corrosion resistance, easy processing, long service life, light weight, high load carrying capability per unit mass. In addition to these polymeric composites are more suitable to interact with fluids than both ceramic and metal matrix composites. Therefore polymeric composites are preferred for automotive industry, production of machine parts and materials which are in contact with fluids [22]. Polymer matrix composites are divided into two subgroups; thermoset and thermoplastic matrix composites. The most important commercial thermosets are unsaturated polyester, epoxy and phenolic resins. According to the properties of dispersed phase, composites classified by three main divisions: Particle-reinforced, fiber-reinforced and structural composites [23]. Figure1.2 shows classification of composite materials according to properties of dispersed phase.
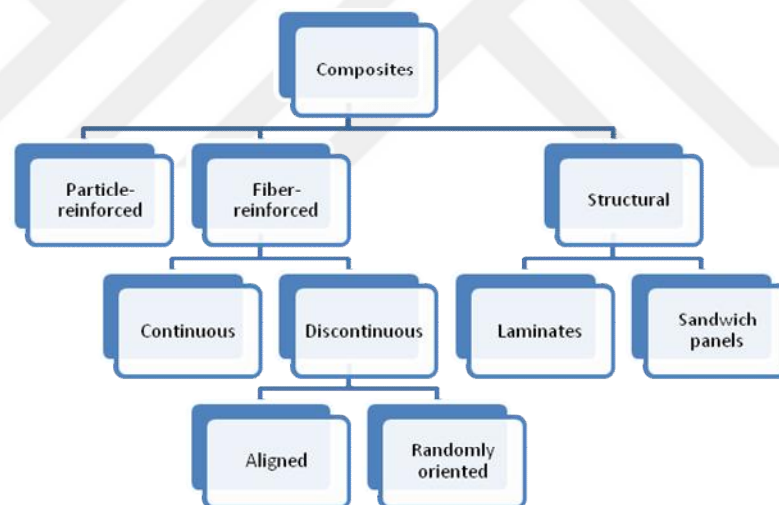
Figure 1.2: Classification of composite materials according to properties of dispersed phase [23].

1.3 Natural Fiber Reinforced Composites

Natural fiber reinforced composites are composite materials formed by a matrix and a reinforcement of natural fibers such as wood fibers (hardwood and softwood) or non-wood fibers. Natural fibers can be defined as substances produced by animals, minerals or plants.

Discovery of natural fiber reinforced composites goes back to 3000 years ago. Egyptions used straw reinforced clay/mud composites for construction of their huts.

In 1958, a car named as Trabant made in Germany is another example of natural fiber composites. Roof, doors and bonnet of Trabant composed of cotton fiber/phenolic resin composite [24]. Today many of automotive manufacturers use natural fibers composites in their products such as door panels, seat backs, sides and back door panels [25]. Examples of natural fiber reinforced composites are shown in figure 1.3.

Figure 1.3: Examples of natural reinforced composites [25].

1.4 Natural Fibers

Natural fibers refer to any hair like raw material which can be obtained from plants (vegetable), minerals or animals. Some examples of natural fibers are shown in figure 1.4.

Figure 1.4: Examples of natural fibers a) plant fiber, b) basalt fiber, c) silk fiber, d) wool fiber [24].

Natural fibers are divided into three main groups according to their origins, whether they are derived from animals, plants, or minerals [9]. Figure 1.5 shows classification of natural fibers depending on their origins.

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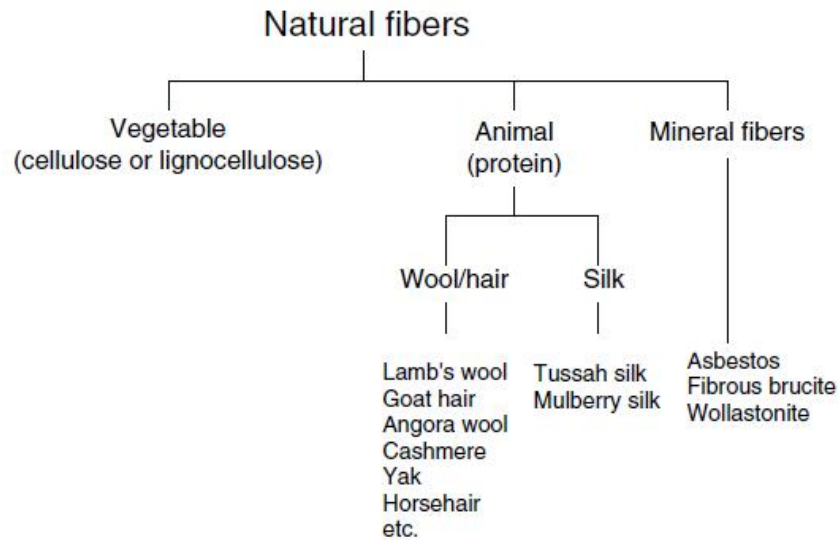
Figure 1.5: Classification of natural fibers [9].

1.4.1 Plant fibers

Plant fibers are extracted fibers from different plants such as leaf, seed, root, stem or wood. Flax, jute, ramie, kenaf, pineapple leaf, sisal are most common plants for extraction of fibers [26]. Classification of plant fibers are shown in figure 1.6.
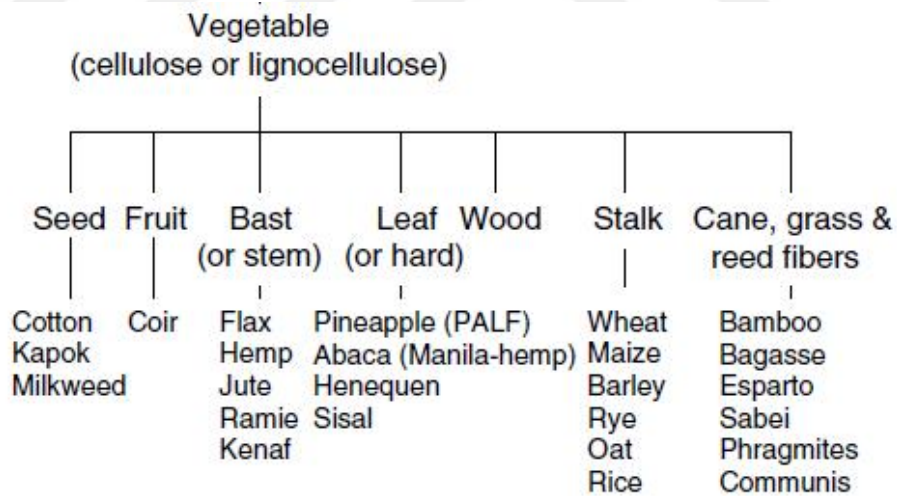
Figure 1.6: Classification of plant fibers [9].

Plant fibers have complex structure and chemical composition. Misra et al. [9] indicated that "plant fibers can be defined as composite material designed by nature". For plant fibers, amorphous hemicellulose or lignin is matrix and crystalline cellulose microfibrils are reinforcement phase which are embedded in matrix". With the exception of cotton, the components of natural fibers are cellulose, hemicellulose, lignin, pectin, waxes and water soluble substances, with cellulose, hemicellulose and

lignin as the basic components with regard to the physical properties of the fibers"
[27]. Figure 1.7 shows plant structure.
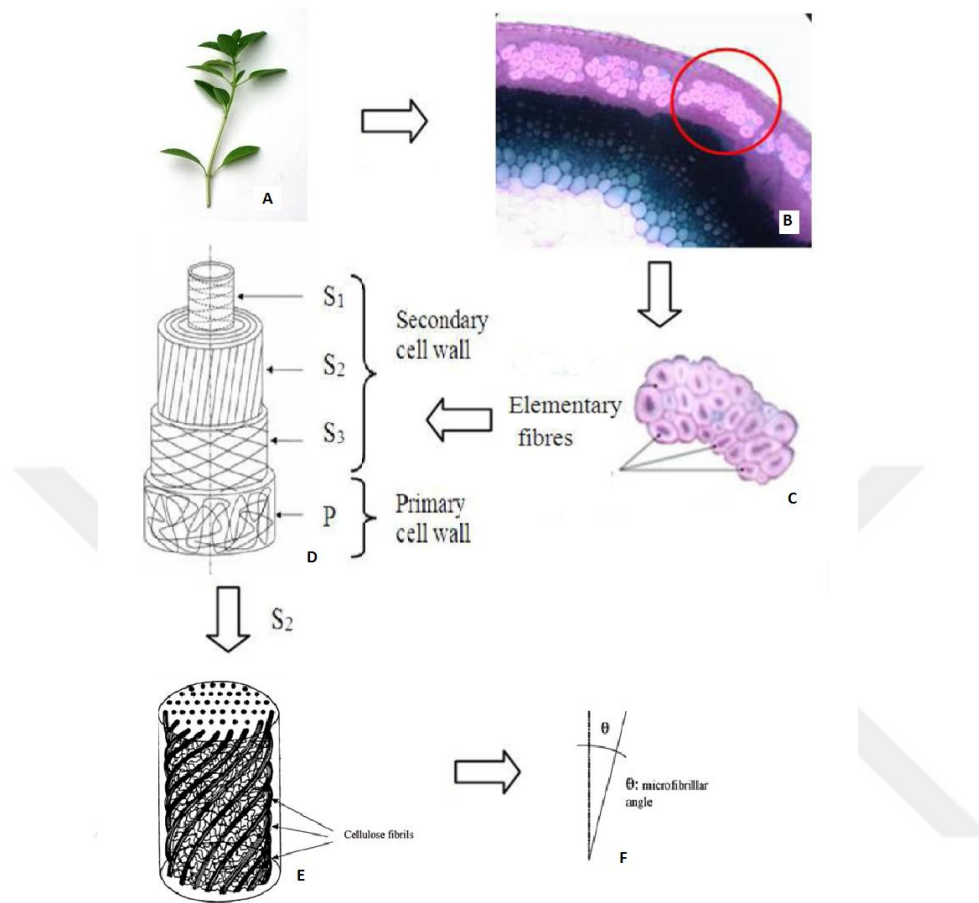
Figure 1.7: Fiber structure; a) plant, b) cross section of plant stem, c) fiber bundle, d)
elementary fiber structure, e) microfibrillar arrangement in S_2 layer, f)
microfibrillar angle [28, 29].

Fiber bundles are composed of several elementary fibers, linked to get herby pectin
and lignin. Elementary fibers have a multi-wall structure. The outer wall represents
"the primary wall". The primary wall forms small proportion of whole fiber. It
contains hemicellulose, low crystalline cellulose, pectin and small amount of wax.
"The secondary wall" is divided into three parts and forms most of the fiber. It can be
thought as natural composite composed of parallelly oriented cellulose microfibrils
embedded in an amorphous matrix composed of lignin, pectin and hemicellulose.
Each three layers have distinct structures; chemical composition, microfibrillar
arrangement and wall thickness. S2 is the thickest part. S2 forms about 80% of whole
secondary wall. Therefore, properties of fiber are mainly depends on structure of S2
layer [29]. Cellulose is main constituent of plant fibers. Cellulose I is crystal
structure of natural cellulose and it is resistant to strong alkali although acids

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hydrolyze cellulose to water soluble sugars. Hemicellulose has shorter and branched chains and thus this hemicellulose has amorphous structure. Alkalis can solve hemicellulose and acids can easily hydrolyze it. Rigidity of plant originates from lignin. "Lignin is three-dimensional copolymer of aliphatic and aromatic constituents with very high molecular weight." [9].

1.5 Fiber Extraction

Fiber production starts with growing of plant. Once a plant that reaches a desired maturity is then harvested and the next step is retting. Obtaining technical fiber bundles includes extraction and separation of fibers from woody core of plant and this process is called retting [9]. It can be said that there are three main retting methods for fiber extraction; biological, mechanical and chemical retting [30]. Figure 1.8 shows the retting methods.
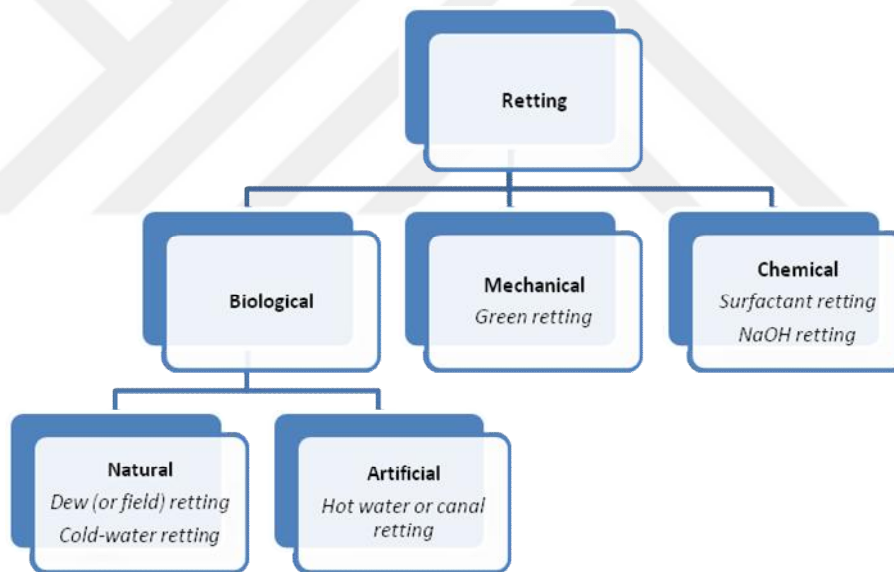
Figure 1.8: Retting methods [9, 30].

1.5.1 Biological retting

Biological retting includes degradation of non-fibrous matter such as pectin and hemicellulose which act as glue between fibers without damaging cellulose part. [30]. There are two traditional biological retting methods; dew retting and water retting [31].

Dew retting contains placing of harvested plants in fields and then decomposition of cementitious substances occurs by filamentous fungi present both on plants and in

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soil. Humidity and temperature differences during the day speed up this process. [9, 31].

For water retting, stems are soaked in tanks filled with water. In this process, plant absorbs water. Water is suitable medium for growing of micro-organisms which are present in plants as well as in water. Micro-organisms grow by consuming pectin, hemicellulose, free sugars, proteins etc. of plant and this allows easy separation of fiber from the woody core of plant [9, 30, 31].

1.5.2 Mechanical retting

Mechanical retting corresponds to separating of fibers by mechanical means. Not having woody part makes leaves suitable for mechanical retting. For hand decortication that is historical way of mechanical decortication, leaf is first pounded and then pulp is scraped away with a knife. With a development of mechanical decorticators, this time consuming process is no more used. In the mechanical decoration process, rotating wheel crushes and beats leaves with blunt knives, so that only fibers remain [30]. Mechanical decorticaton process was shown in figure 1.9.
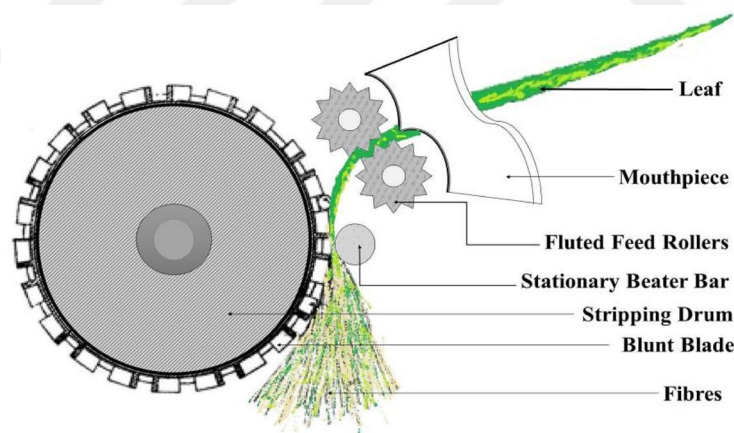
Figure 1.9: Cross sectional view of stripper/decorticator [30].

1.5.3 Chemical retting

Chemical retting refers to extraction of fibers from stem by using chemical solutions such as sodium carbonate, potassium or sodium hydroxide, chlorinated lime and sulfuric acid solutions in heated tanks. Depending on the process conditions, chemical treatment reduces the retting time to a few minutes or up to 48 hours.

Using of alkali in chemical retting results in increased surface area by dissolving of hemicellulose and lignin component, increased cellulose crystallinity by repackaging

of cellulose microfibrils during process and cleaner fiber surface by removal of pectin and wax substances [9, 32, 33].

1.6 Althea Officinalis l. (marshmallow) Plant

Althea officinalis l. is annual plant which is known as "marshmallow" and widely abundant in Turkey. The plant naturally grows widely on the banks of rivers and field borders and length of plant can reach up to 2 meters. Althea officinalis l. plant belongs to malvaceae family and in Turkey there are four species of althea occur; Althea hirsuta l., Althea officinalis l., Althea americana ten. and Althea cannabina t.

The name "althea" is derived from Greek word "altho" which means to cure. Owing to the high mucilage content, the plants were used commonly for soothing chapped skins, minor wounds, sore throats, external and internal flammations.

In Turkey, althea officinalis l. is used for many purposes as a medicine by rural folk. The infusion prepared by the flowers and leaves of althea officinalis is used in ailments of the lungs and as a demulcent. The root decoction is used as diuretic; counters excess stomach acid; oral, esophageal and peptic ulcerations and gastritis. It is also applied externally to bruises, sprains, skin inflammations and splinters as an emollient, as a protective and wound healing agent [34].

1.7 Literature Survey

This section includes summary of papers about novel plant fibers which are potential reinforcement for composite materials.

Fiore et al. (2011) extracted fibers from artichoke by water retting method. The plant stems were kept in water for 25 days to achieve fibers. Real density of fiber was determined 1.580 g/cm^3 and the apparent density was determined as 1.210 g/cm^3. Decomposition temperature was determined by DT/TGA.

As it was indicated fiber has multi-stage decomposition profile. Decomposition of the fiber starts at 230 ^{o}C and gives first peak at 295 ^{o}C (decomposition of hemicellulose and pectin) and the second peak occurs at 352 ^{o}C (decomposition of cellulose). Tensile test results showed fiber exhibits brittle behavior [35].

De Rosa et al. (2010) characterized okra fibers and in 2011 they studied effects of chemical treatments on properties of the fiber. According to first study okra fibers were extracted by water retting for 15-20 days. Chemical composition analysis

showed that fiber contains 4% pectin, 5-10% lignin, 15-20% hemicelluloce, 60-70% cellulose. Also fiber starts to decompose at 220 $^{\circ}$C because of hemicellulose and gives peak at 360 $^{\circ}$C and tensile test showed that the fiber has brittle structure. Acetylation caused diminish of tensile strength and modulus, changes for physical structure and some compositional changes [36, 37].

Chirayil et al. (2014) extracted nanofibrils from isora plant. They first extracted fiber bundles from stem by biological retting. Extracted fiber bundles ware alkali treated and bleached to achieve delignified isora and then acid treated to obtain cellulose nanofibrils. It was indicated in study that alkali treatment caused dramatic decrease in lignin and hemicellulose content [38].

Seki et al. (2013) characterized ferula fibers. Ferula fibers were extracted by using 20% aqueous solution of acetone. Crystallinity index was determined as 48% and chemical composition was 1.4% lignin, 8.5% hemicellulose and 53% cellulose. Tensile strength was determines as 475 MPa for the fiber [16].

Almeida et al. (2006) extracted piassava fibers by mechanical retting and results of tensile test showed fiber has about 160 MPa tensile strength. DT/TGA showed decomposition occurs at 350 $^{\circ}$C [39].

Saravanakumar et al. (2013) investigated properties of prosopis juliflora fibers. Fibers were extracted by water retting. Fibers have about 560 MPa tensile strength and are composed of 61% cellulose, 17% lignin and 16% hemicellulose. Decomposition occurs at 330 $^{\circ}$C for prosopis juliflora fibers [40].

Indran et al. (2015) extracted fibers from cissus quadrangularis stem by water retting for 14 days and characterized fibers. Chemical composition of the fibers was determined as about 11% lignin, 8% hemicellulose and 83% cellulose. Crystallinity index, tensile strength and decomposition temperature were 47%, 342 $^{\circ}$C and 2300-4157 MPa in increasing gauge lengths for cissus fibers respectively [41]. Haameem et al. (2016) determined effect of alkali treatment on fiber properties of napier grass. Fibers were extracted by water retting and fibers were reacted with NaOH solutions (5% to 20%). Single fiber tensile test result of study revealed that tensile strength increase with increasing concentration to a specific point. This point (concentration) was %5 for napier grass. After this concentration mechanical properties decreased [11]. Amroune et al. (2015) extracted fibers from palm fruit branches. They

investigated effect of alkali treatment on properties of fiber. Fibers were soaked solutions having different NaOH concentration for various times from 12 hours to 96 hours. Results showed that chemical treatments increased tensile strength and modulus (up to 178% for the stress and 167% tensile modulus compared to untreated fibers) [42].

Fiore et al. (2014) extracted fiber from arundo donax l. plant by hand decortication. Density of arundo fiber was determines as 1.168 g/cm^3 and cellulose content was 43%, hemicellulose and lignin contents were 20% and 17% respectively. Tensile strength of fiber was determined as about 320 MPa by single fiber tensile test [15].

2. MATERIALS AND METHODS

2.1 Fiber Extraction

AOL plants were harvested after flowering in Mordoğan (İzmir-Turkey). After harvesting, stems of the fresh plants were separated from branches and foliages. Separated stems were cut into parts about 100 mm and washed with distilled water to remove any dirt and/or dust. Then cleaned parts of stems divided into half and some of them were used for water retting and rest of them used for chemical retting. For water retting, stems were soaked in a can including water and closed to allow microbial degradation till easy separation of fibers from stem. For chemical retting, cleaned stems were submerged in NaOH solution including 5% NaOH and boiled for 2 hours. Boiling time was determined by trial and error approach to easy separation of fibers from the stem. Extracted fibers were then thoroughly washed five times with warm water to remove any alkali boiling waste and prevent further retting. Figure 2.1 shows the chemical extraction process. Finally fibers were dried in an oven at 60°C for a night (12 hours) to remove moisture.

Figure 2.1: Fiber extraction process a) fresh plant, b) cleaned part of stem, c) NaOH boiling, d) warm water washing, e) chemically extracted fibers.

2.2 Characterization of Althea Fibers

Water and chemically retted Althea fibers were deeply characterized to examine effects of retting method on fibers chemical and mechanical properties. In accordance with this purpose; FTIR, XRD, density measurement, TG/DTA, SEM observation, and single fiber tensile tests and analysis were done.

2.2.1 Plant anatomy

In the preparation of the samples, firstly the stems were cut into 0.5 mm of thicknesses. Before embedding to paraffin, samples were formalin fixated and waited in ethyl alcohol and xylene respectively. After embedding to paraffin, samples were cut to 5 μm with rotary microtome. After that cut samples were dried and waited in xylene, ethyl alcohol, and distilled water respectively. The samples were then hematoxylin-eosin stained and finally the samples were closed with cover glass by using balsam.

2.2.2 FTIR analysis of fibers

Perkin Elmer Spectrum spectrophotometer was used to determine compositional change of water and chemically retted althea officinalis l. fibers with a scan rate of 16 scans per minute at a resolution of 2 cm^{-1} in the wave number region 650- 4000 cm^{-1}. In this work, chopped Althea fibers were ground into powder prior to analyze. Spectral outputs were recorded in transmittance mode as a function of wave number. FTIR device was shown in Figure 2.2

Figure 2.2: Fourier transform infrared spectroscope.

2.2.3 XRD analysis of fibers

Rigaku D-max-2200-PC XRD (X-ray diffractometer) was used for X-ray diffraction analysis. Determination of crystallinity of water and chemically retted althea officinalis fibers were done by XRD. Samples were scanned in(2θ) range of 3-70° with scan speed of 4°/min, at 40 kV and 36 mA. Segal empirical method was used for calculation of crystallinity indexes [43]:

$$CI\% = \frac{(I_{002} - I_{am})}{I_{002}} \times 100 \quad \text{equation 1}$$

I_{002} and I_{am} correspond to maximum intensity value (in arbitrary units) of crystalline phase peak and minimum intensity value (in arbitrary units) of amorphous phase. Figure 2.3 shows x-ray diffractometer.

Figure 2.3: X-ray diffractometer.

2.2.4 Determination of the densities of fibers

Micromeritics accupyc II 1340 model helium gas pycnometer was used for density measurement of water and chemically retted fibers. Helium pycnometer was shown in figure 2.4.
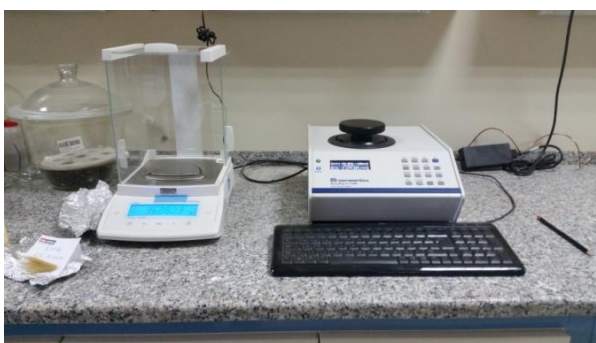
Figure 2.4: Helium pycnometer.

2.2.6 TG/DTA analysis of fibers

Decomposition temperatures of water and chemically retted fibers were determined by thermogravimetric analysis. Thermogravimetric analysis (TGA) was carried out with Perkin Elmer STA 8000 TG/DTA by heating from room temperature by 10^oC/min from room temperature to 700^oC under N_2 atmosphere. TGA device was shown in figure 2.5

Figure 2.5: TGA device.

2.2.7 Surface morphology of fibers

The surface morphologies of the water and chemically retted Althea fibers were examined by JEOL-JJM 6060 model scanning electron microscope (SEM). SEM images were obtained with an accelerating voltage of 5 kV and magnification of 50x to 2500x. Fibers surfaces were coated with Au-Pd by sputter coating prior to scanning. Figure 2.6 shows sputter coating device and scanning electron microscope.

Figure 2.6: a) Sputter coating device and b) Scanning electron microscope.

2.2.8 Single fiber mechanical properties

The tensile properties of single Althea fibers were determined by placing of fibers onto cupboards from end tabs and applying of polyester adhesive and mounting prepared single fiber system onto Shimadzu AGS-X machine with a 5 kN load cell and a crosshead speed of 1 mm/min.

Determination of fibers diameters was done via optical microscope investigation. Average of three different sections was calculated as diameter value. Figure 2.7 shows longitudinal image of fibers.
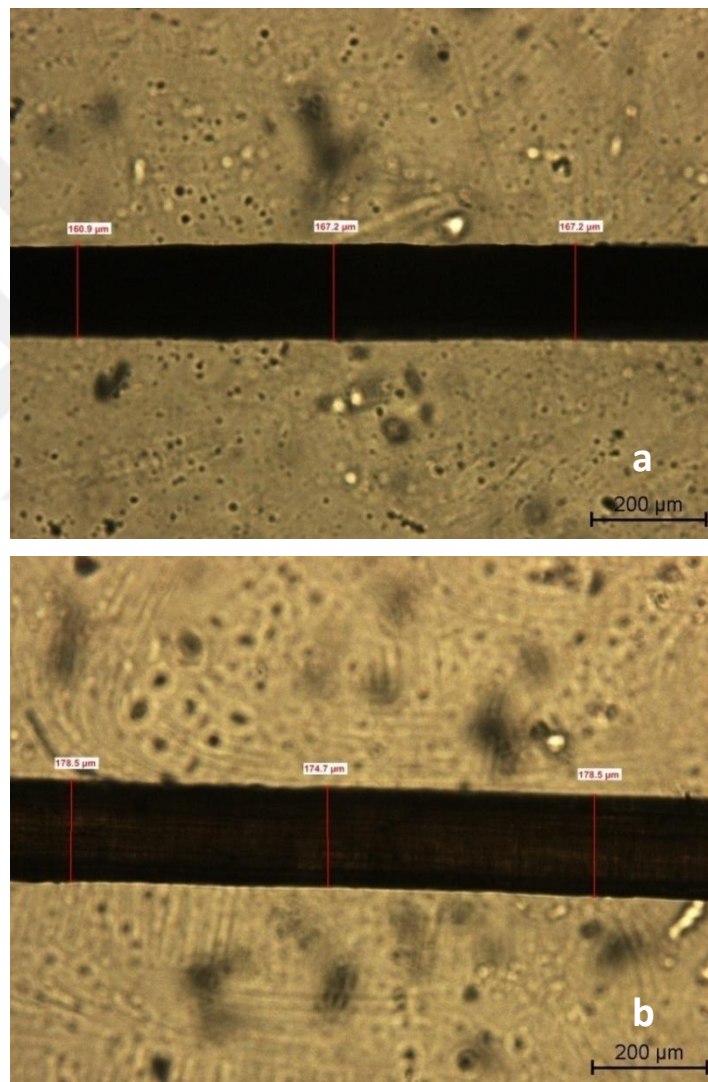
Figure 2.7: Longitudinal image of a) water retted, b) chemically retted fibers.

Althea fibers were mounted on cardboards from end tabs with adhesive polyester. The gauge length of each Althea fiber was specified as 50 mm. Twenty samples were tested and the average tensile strength, tensile modulus, and elongation at break were determined. Figure 2.8 shows single fiber mounted on tensile testing machine.

Figure 2.8: a) Mounted single fiber b) Tensile testing machine.

2.3 Composite Production

2.3.1 Polymer matrix curing

Unsaturated polyester resin was used as matrix material for this study and supplied by DYO Paints Manufacturing and Trading Company. Catalyst (Cobalt Naphthalene) and accelerator (Methyl Ethyl Ketone Peroxide) were used to cure unsaturated polyester resin.

2.3.2 Composite preparation

Dried fibers were cut to lengths of 10mm, 30mm, and 50mm to prepare the composites. The conventional hand lay-up technique was used to prepare the composite specimen with various weight fractions as 5%, 10%, and 20%.Composites are coded depending on weight fractions and represented in table 2.1.

Table 2.1 Codes of composites.

Fiber length (mm)	Weight fraction (%wt.)	Code
	5	UP1-5
10	10	UP1-10
	20	UP1-20
	5	UP3-5
30	10	UP3-10
	20	UP3-20
	5	UP5-5
50	10	UP5-10
	20	UP5-20

One percent of catalyst and one percent of accelerator was used to cure the polyester resin. Aluminum mould was prepared to product the composite specimens. Firstly surface of the mould was coated by releasing agent for easy removal of the specimens after the curing process. Mould surfaces were dried for 10 min, then fibers were spread over the mould and unsaturated polyester resin was poured into mould cavity. During curing, compressive pressure was applied on the closed mould at atmospheric temperature. Finally the Althea fiber reinforced polyester composite plates were obtained having dimensions of 19mm x 15mm x 4mm. After solidification, composite plates were post-cured for one hour in oven at 60°C.

2.4 Characterization of Composites

2.4.1 Tensile test

Tensile tests were performed according to ASTM D638 by Shimadzu AG-IS Universal Test Machine with the capacity of 250 kN. The cross head speed was set up to 5mm/min. and elongation was measured by non-contact video extensometer. Composite plates were machined to dog bone shaped tensile test specimens conforming type-1; dimensions and gage length of specimens were 160 mm x 13 mm x 4 mm and 50 mm respectively. Figure 2.9 shows tensile testing machine.

Figure 2.9: Tensile testing machine.

2.4.2 Flexural test

ASTM D790 standard was used for determination of flexural properties of composites by using Shimadzu AG-IS Universal Test Machine with the capacity of 50 kN and cross head speed was set up to 2 mm/min. Dimensions of specimens were 120mm x 13mm x 4mm. Figure 2.10 shows three point bending device.

Figure 2.10: Three point bending machine.

2.4.3 SEM analyses of composites

The fracture surface of 5, 10, and 20% reinforced fiber/polyester composites were examined by JEOL-JJM 6060 model SEM to evaluate microstructure of composite and interface between fibers and the matrix. SEM images were obtained with an accelerating voltage of 5 kV and various magnifications. Fractured surfaces were coated with Au-Pd by sputter coating prior to scanning.

3. RESULTS AND DISCUSSION

3.1 Fiber Characterization

3.1.1 Plant anatomy

In Althea officinalis's stem section (fig. 3.1), there are 9 distinct regions. First layer, cortex, consists four parts, hypodermis (fig 3.1.1-2), collenchima (fig 3.1 1-3) and epidermis (fig 3.1 1-4) with a thickness of 10-12 cells. The outer region, epidermis (fig 3.1 1-1), is formed of an individual layer of rectangular closely-packed cells. It has cells with outer walls which is cuticle. The second layer is parenchyma (fig 3.1 1-5). This layer has cells rather bigger than other cells. It surrounds sclerenchyma (fig 3.1 1-6) and phloem (fig 3.1 1-7). Sclarenchyma is the main support cells that give the mechanical rigidity to body and protects phloem from any damage (99). Phloem is the living tissue that carries organic nutrients to all parts of the plants. Third layer is xylem (fig 3.1 1-8). It is a thick layer in the plant because it transports water. The inner part of the plant has a pith (or medulla), which composed of spongy parenchyma cells that store and transport the nutrients (fig 3.1 1-9) [44].
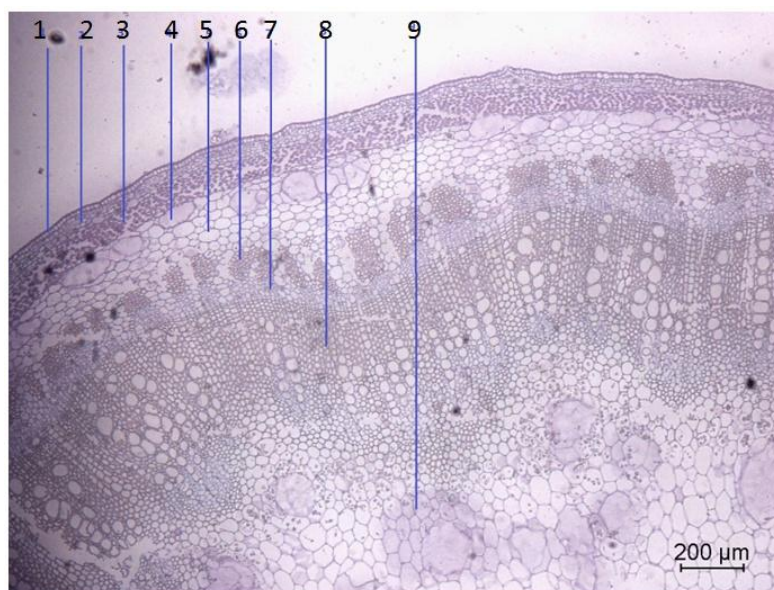

Figure 3.1: Optical Microscope image of Althaea officinalis stem cross section, 1)epidermis, 2)hypodermis, 3)collenchyma, 4)epidermis, 5)parenchyma, 6)sclerenchyma, 7)phloem, 8)xylem, and 9)pith [34].

3.1.2 FTIR analysis of fibers

Effect of alkali boiling on the compositional changes of fibers was investigated by FTIR. FTIR analysis of water and chemically retted fibers are shown in Figure 3.2. And also table 3.1 shows detailed explanation of peaks.

Table 3.1: FTIR spectral data

Wave Number (cm^{-1})	Vibration	Functional group or component	References
890	β-glucosidic linkages between sugar units	Hemicelluloses and celluloses	[45,46]
1030	C-O stretching	Hydoxyl and ether groups in cellulose	[47]
1106 1165	C-O-C stretching	Pyranose ring in polysaccharides	[48,49]
1241	C-O stretching of acetyl groups	Lignin	[50]
1320 1373	CH$_2$ wagging and CH$_3$ bending	Lignin	[52] [54]
1425 1506 1597	Benzene ring stretching and CH$_2$ deformation	Lignin	[48,49,51,53] [48,51] [37]
1636	H-O-H bending	Water content of fiber	[55]
1737	C=O stretching	Hemicellulose or carboxylic acid in lignin	[15]
2850 2920	C-H stretching	CH and CH$_2$ groups in cellulose and hemicellulose	[15, 16]
3300	O-H stretching	Lignin and cellulose	[49]

Changes in the Althea fibers as a function of alkali treatment were followed by FTIR and are shown in Figure 3.2.Spectral data examination showed that some changes did occur. Vibration peak at 2920 cm^{-1}which belongs to C-H stretching vibration of hemicellulose and cellulose, reduced after alkali boiling indicating that part of the hemicellulose was removed. Because of hemicellulose was removed from chemically retted fibers, vibration peak at 1737 cm^{-1}, assigned to a C=O stretching vibration of carboxylic acid or ester in lignin, disappeared. In the same way vibration peak at 1425 cm^{-1} and 1241 cm^{-1} which belong to benzene ring stretching vibration of lignin and C-O stretching vibration of the acetyl group in lignin were reduced respectively. These results all indicate that alkali boiling leads to the partial removal of lignin and hemicellulose [56].
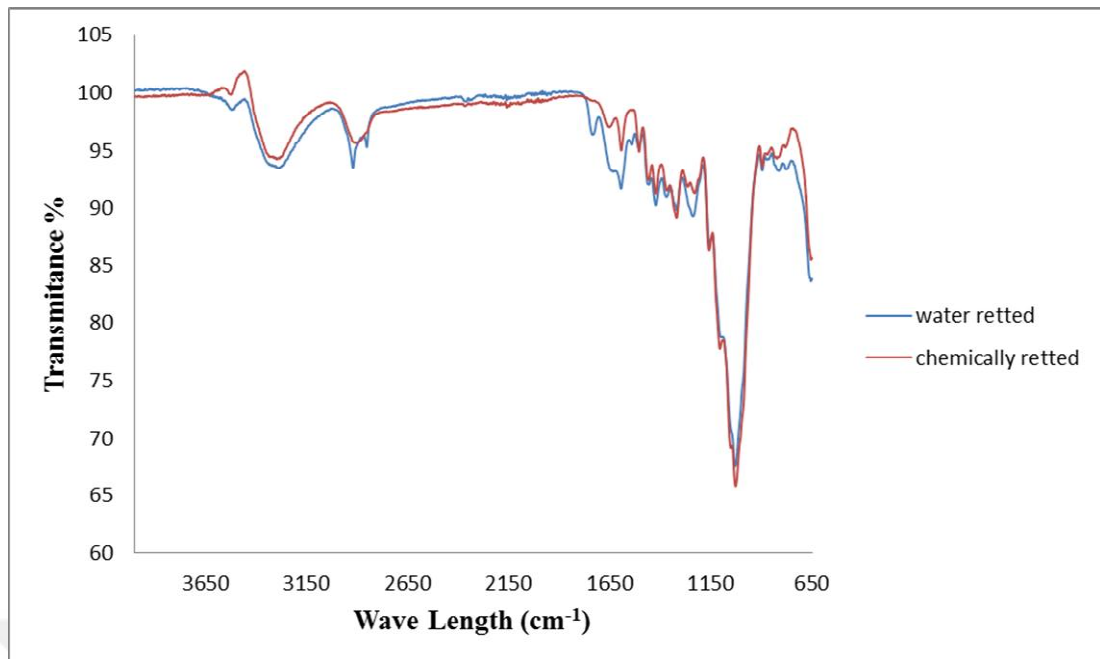
Figure 3.2: FTIR spectrum of fibers.

3.1.3 XRD analyses of fibers

Figure 3.3 shows XRD patterns of water and chemically retted Althea fibers. Peaks located at 22.16 and 22.14 2θ angle are belong to (002) lattice reflection peak of cellulose I. Minimum intensity values located between 18° and 19° belong to amorphous phase [57].

According to equation 1 (Segal empirical method), crystallinity indexes of water and chemically retted Althea fibers were determined as 65% and 74% respectively. This improvement can be explained by NaOH treatment.

As a result of chemical retting with alkali treatment, some reactions take place between fiber and NaOH. Hydrogen bonded cellulose structure disrupts due to alkali treatment and Na^+ ions penetrate into cellulose planes and increase distance between planes and water molecules fill these spaces. As a result fiber swells. Structure of fiber-OH is converted fiber-O-Na. Finally rinsing of fiber by water causes removing of linked Na^+ ions and celluloses are packaged to a new crystalline structure. This new closed packaged form have higher crystallinity [32, 47, 58- 62].
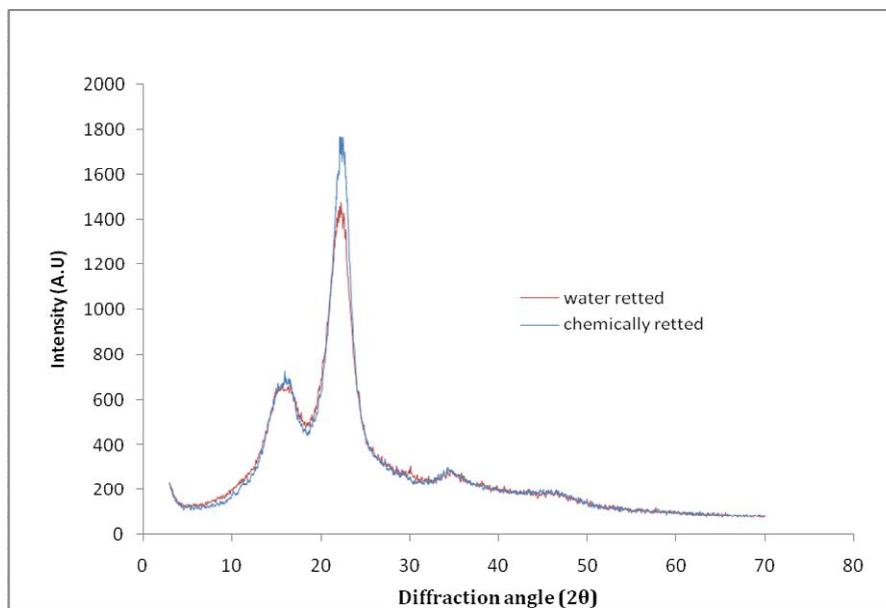
Figure 3.3: XRD patterns of water and chemically retted Althea fibers

Basu et al. reported similar results. They investigated effect of various retting method on the properties of coconut fiber. They reported crystallinity indexes of raw and chemically retted coconut fibers as 37% and 54% respectively. They attributed these results to removal of non-crystalline constituents such as hemicellulose, lignin and pectin [71].

3.1.5 Density determination of fibers

Densities of water and chemically retted fibers was determined as 1,2584 g/cm^3 and 1,3378 g/cm^3 respectively. This difference probably derived from alkali treatment. Removal of amorphous phases such as hemicellulose and wax and increasing cellulose crystallinity may cause density improvement of fibers [63]. Mean densities of different plant fibers are shown in table 3.2.

Table 3.2: Densities of different plant fibers [61].

Fiber	Density (g/cm^3)
Cotton	1.5-1.6
Jute	1.3-1.46
Flax	1.4-1.5
Hemp	1.48
Ramie	1.5
Sisal	1.33-1.5
Coir	1.2

3.1.6 TG/DTA analysis of fibers

Figure 3.4 and 3.5 show DT/TGA results of water and chemically retted fibers. Chemical retting caused shifting of decomposition temperature to higher values. It can be seen that decomposition temperature of chemically retted fiber is 339 $^{\circ}$C while water retted fiber decomposes at 334 $^{\circ}$C. Also water retted fiber has multi-stage decomposition due to amorphous contents. This decomposition behavior is derived from chemical retting. Removal of pectin, wax, hemicellulose and lignin contents results in higher decomposition temperature and also these constituents decompose at lower temperatures compared to cellulose [56].
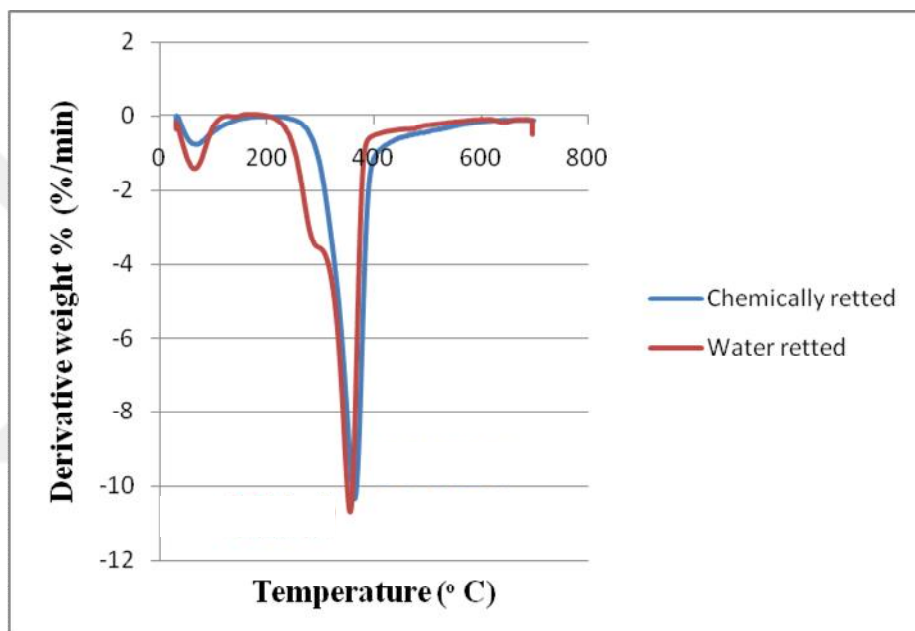

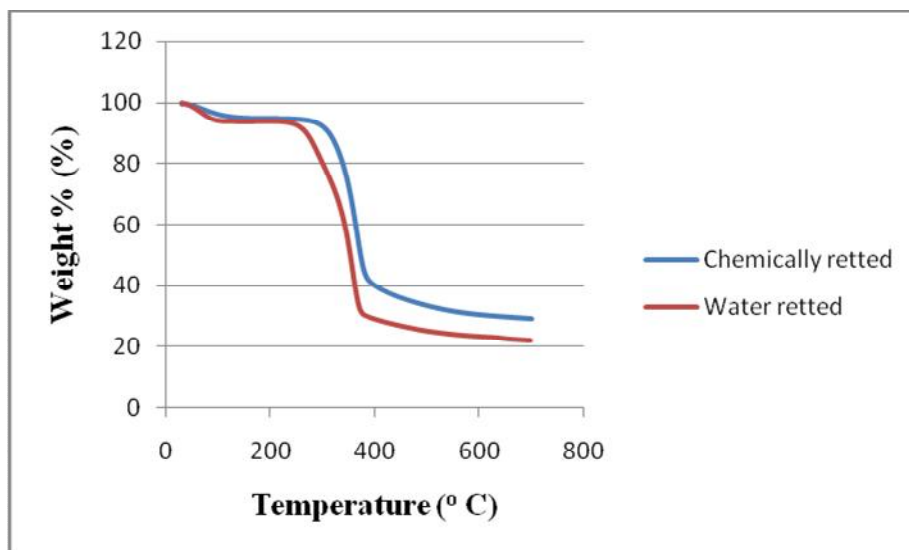**Figure 3.4:** DT curves of water and chemically retted althea fibers.

Figure 3.5: TGA curves of water and chemically retted althea fiber.

3.1.7 Surface morphology of fibers

Figure 3.6 a, b, c show water retted while Figure 3.7 a, b and c show chemically retted Althea fibers surface morphologies at different magnifications from 250x to 2500x. It was important to study the surface morphology of the fibers to observe the changes that occurred on the surface of the althea officinalis fibers because of the alkali boiling process. As it is seen from figure that surface of water retted fiber contains impurities probably lignocelluloses covered with waxy substances and hemicellulose. On the other hand chemically retted fiber surface is cleaner and rougher than water retted fiber because alkali boiling causes removal of hemicellulose, lignin and wax contents of fibers because the surface impurities on the fibers are soluble in the alkali solution and this explains the cleaner surface. Fibrillation is obvious but no deterioration or severe fibrillations of fibers are observed [12, 64].
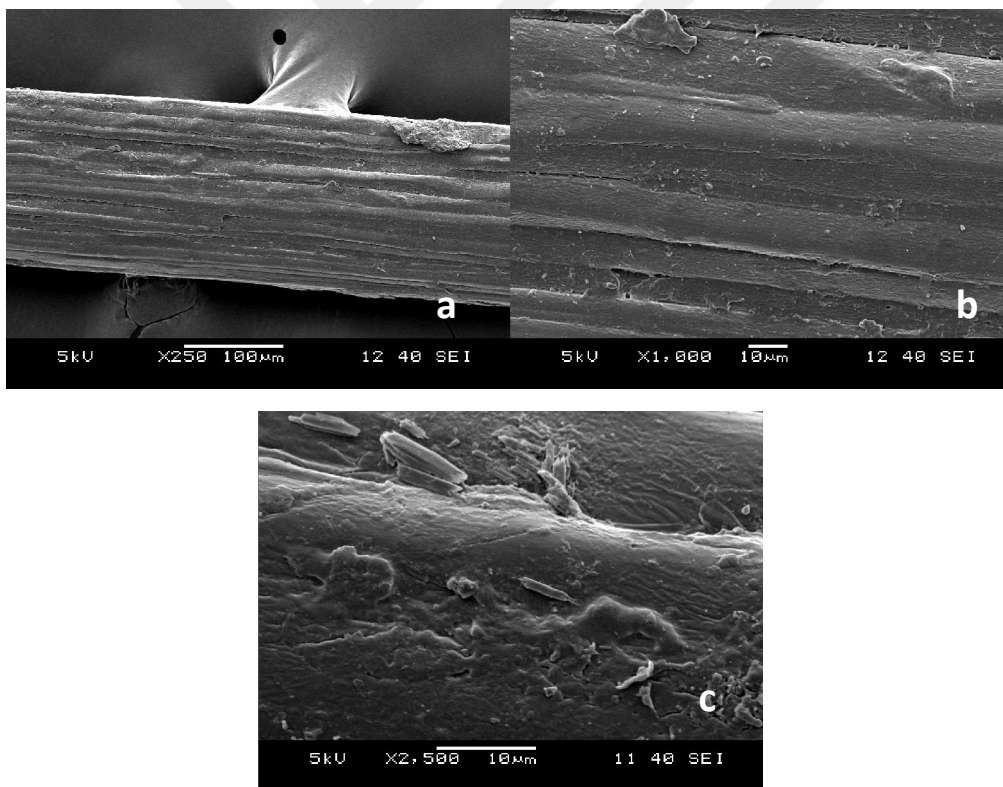
Figure 3.6: a, b, c show surface morphology water retted fibers.
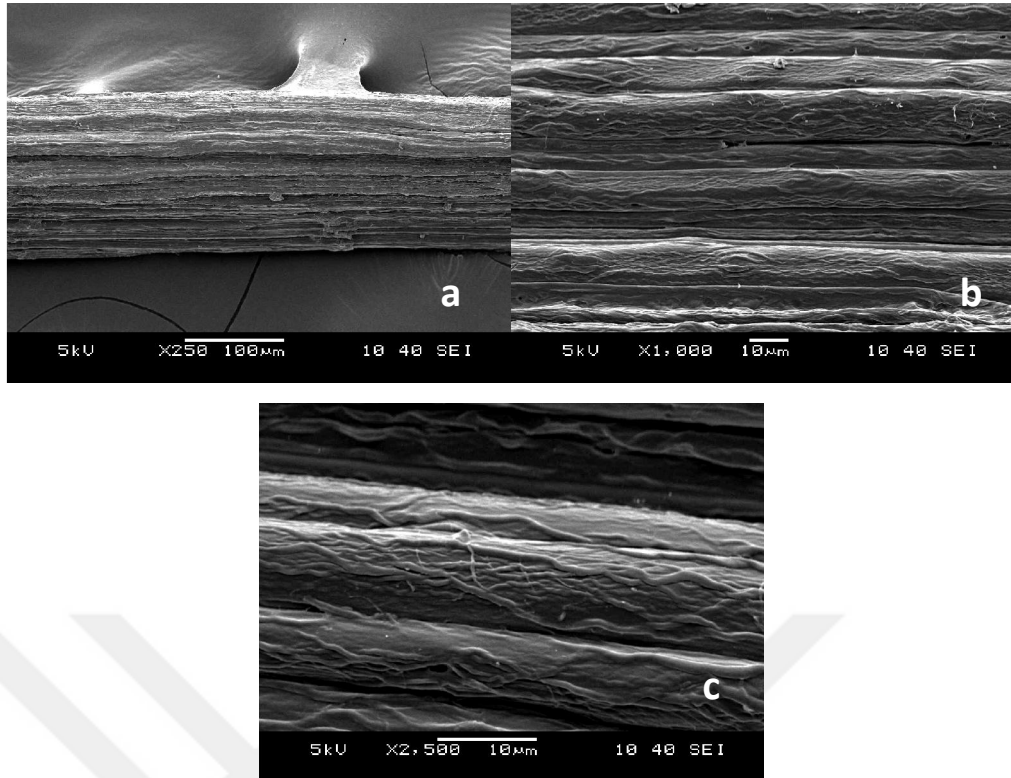
Figure 3.7: a, b, c show surface morphology chemically retted fibers.

3.1.8 Single fiber tensile testing

Stress-strain plots of water and chemically retted fibers are shown in figure 3.8. Both water and chemically retted fibers exhibit brittle behavior with a linear profile. It is obvious that chemical extraction improved tensile strength of Althea fibers. While water retted fibers have average 185 MPa, chemically retted fibers have average 275 MPa ultimate tensile strength. Extraction of Althea fibers by boiling in alkali solution including 5% NaOH for two hours resulted in about 48% improvement of tensile strength and also decreasing in elongation at break and increasing in tensile modulus by 29% and 53% respectively.

According to single fiber tensile test results it can be concluded that chemical extraction made Althea fibers more brittle. Figure 3.9, 3.10 and 3.11 show bar chart of tensile strength, elongation at break and tensile modulus of water and chemically retted fibers respectively.
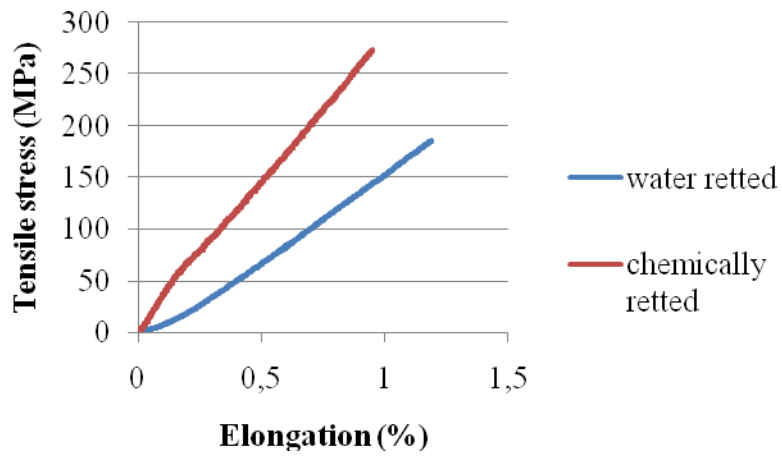
Figure 3.8: Stress-strain plots of water and chemically retted fibers.
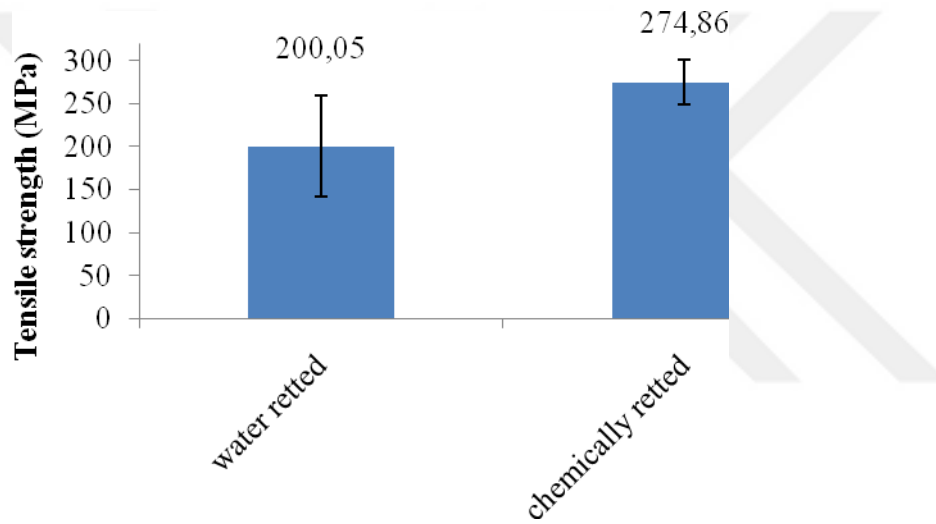
Figure 3.9: Tensile strength bar chart of water and chemically retted fibers.
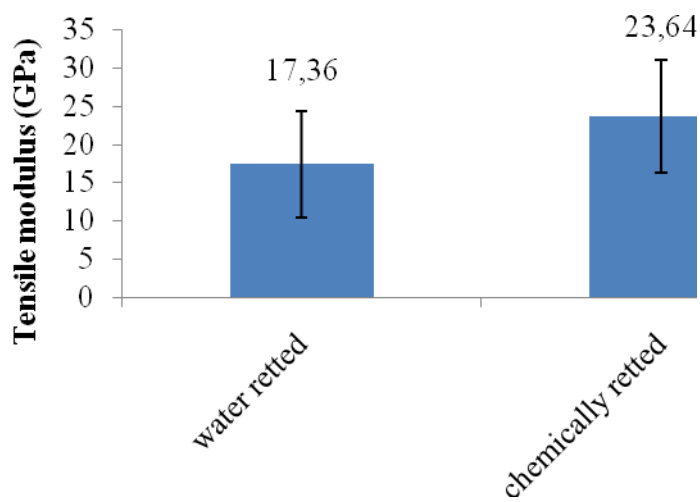
Figure 3.10: Tensile modulus bar chart of water and chemically retted fibers.
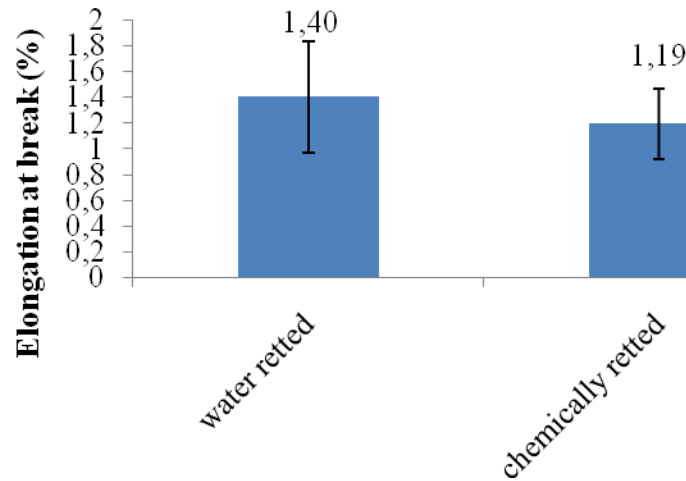
Figure 3.11: Elongation at break bar chart of water and chemically retted fibers.

As it is mentioned in section 3.1.3; as a result of chemical retting with alkali treatment and packaging of celluloses to a new crystalline structure results in higher crystallinity of cellulose and tensile strength of fiber increases [32, 47, 58- 62]. NaOH treatment not only causes increasing in cellulose crystallinity but also changing in spiral angle of cellulose micro fibrils. Cellulose micro fibrils are arranged in an order as seen in figure 3.12. Spiral angle of cellulose micro fibrils decreases by NaOH treatment and elastic modulus of fibers increase with decreasing spiral angle [65- 69]**.** Table 3.3 shows mechanical properties of various plant fibers.
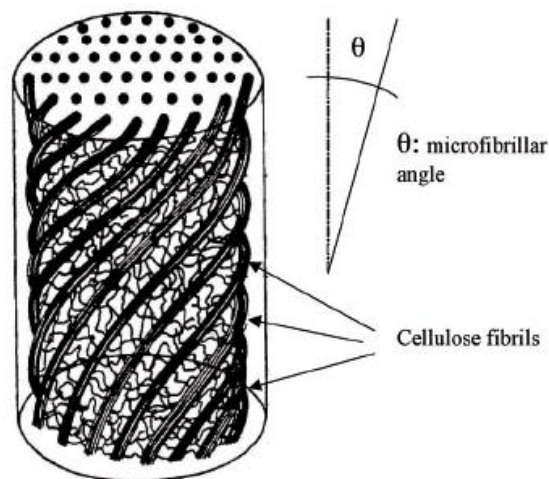
Figure 3.12: Micro fibril orientation and spiral angle [65].

Table 3.3: Mechanical properties of various plant fibers [61].

Fiber	Elongation (%)	Tensile strength (MPa)	Tensile modulus (GPa)
Cotton	3.0-10.0	287-597	5.5-12.6
Jute	1.5-1.8	393-800	10-30
Flax	1.2-3.2	345-1500	2706-80
Hemp	1.6	550-900	70
Ramie	2.0-3.0	220-938	44-128
Sisal	2.0-14	400-700	9.0-38.0
Coir	15.0-30.0	175-220	4.0-6.0

3.2 Composite Characterization

3.2.1 Mechanical characterization

The tensile properties of althea fiber reinforced polyester composites were investigated for the various fiber weight fractions. Tensile strength and modulus values with standard deviations are indicated in table 3.4. The maximum tensile strength was observed with 20% fiber loading and 50mm fiber length. Tensile modulus values of specimens were calculated by considering the slope of the elastic region of the stress–strain curve. The tensile strength increased from 21.66 MPa for UP to 82.68 MPa for UP5-20. Figure 3.13shows bar chart of tensile strengths, 3.14 shows bar chart of flexural strength of composites against the fiber weight fractions for all fibers which have 10 mm, 30 mm and 50 mm fiber length. It can be seen from figure 3.13, tensile strength increases with increasing fiber weight fraction and length. Fiber-matrix interface determines the composite strength.

Table 3.4: Tensile properties of composites.

Specimen	Tensile strength (MPa)	Tensile modulus (GPa)
UP	21.66±8.14	3.31±0.49
UP1-5	25.39±7.69	4.73±1.22
UP1-10	31.86±6.79	5.21±0.51
UP1-20	39.66±4.79	6.70±2.07
UP3-5	27.70±5.78	2.59±0.39
UP3-10	39.79±5.42	6.99±2.27
UP3-20	63.38±5.76	7.54±2.78
UP5-5	31.12±7.48	4.43±0.43
UP5-10	42.16±5.47	6.17±1.50
UP5-20	82.68±16.31	7.77±1.88
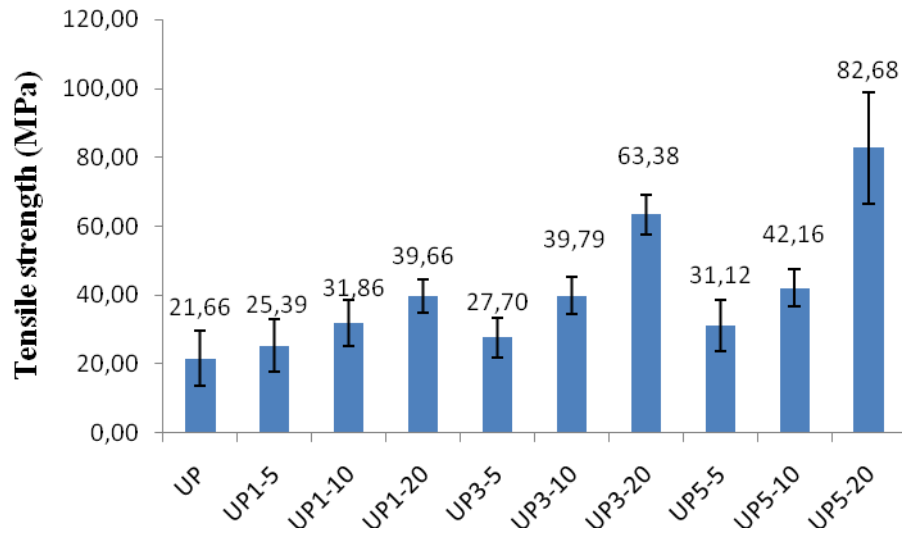
Figure 3.13: Tensile strengths of composites versus various fiber length and fiber loading.

A summary of the flexural properties of the composites with fiber weight fractions and fiber lengths are presented in Figure 3.14. Generally flexural strength increases with increasing fiber length and loading but somehow UP-3-20 has the highest flexural strength with a high standard deviation.
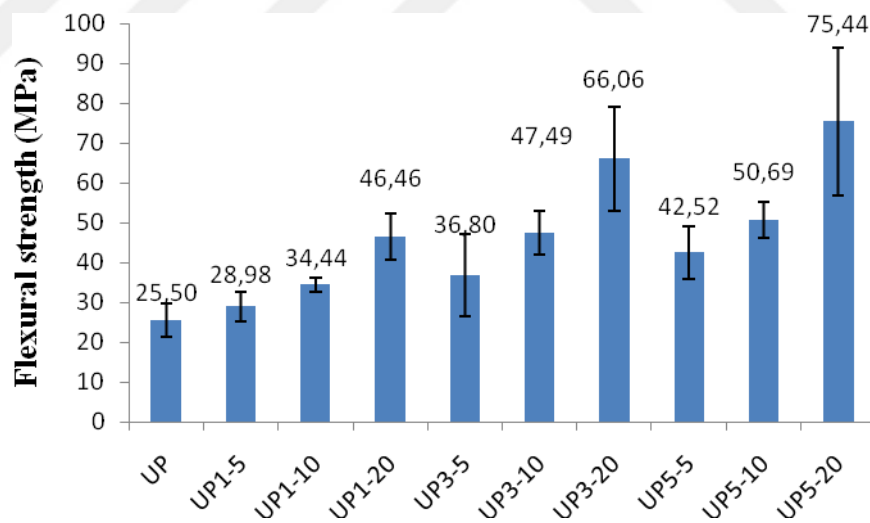
Figure 3.14: Flexural strengths of composites versus various fiber length and fiber loading.

3.2.2 SEM analysis of composites

Failure mechanisms of althea fiber reinforced composites were investigated by SEM analysis. SEM images of fractured samples are shown in figure 3.15 to figure 3.23. It can be seen that althea fibers are not homogeneously distributed in unsaturated polyester matrix especially for low loadings. This manner is probably derived from

production method. Generally existence of long fibers on the fractured surface refers to fiber pull-out. Fiber pull out is derived from weak interfacial bonding between fiber and matrix. In case of strong interfacial bonding, fibers are broken without pull out on the other hand fiber-matrix interface cannot carry load efficiently because of weak adhesion and results in fiber debonding [13, 70]. Although there are fiber pull-outs, as it can be seen in figures, there are also good bonded fibers that fractured near the crack. This situation supports the tensile test results.

Figure 3.15: Tensile failure surface of UP1-5.

Figure 3.16: Tensile failure surface of UP1-10.

Figure 3.17: Tensile failure surface of UP1-20.

Figure 3.18: Tensile failure surface of UP3-5.

Figure 3.19: Tensile failure surface of UP3-10.
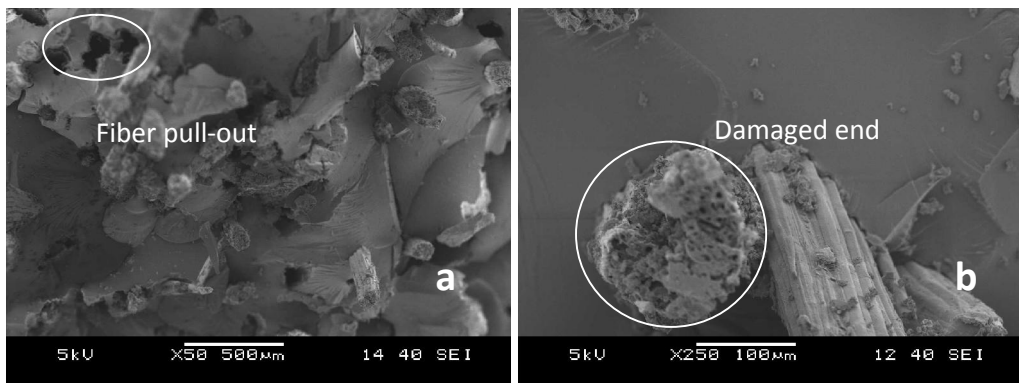
Figure 3.20: Tensile failure surface of UP3-20.
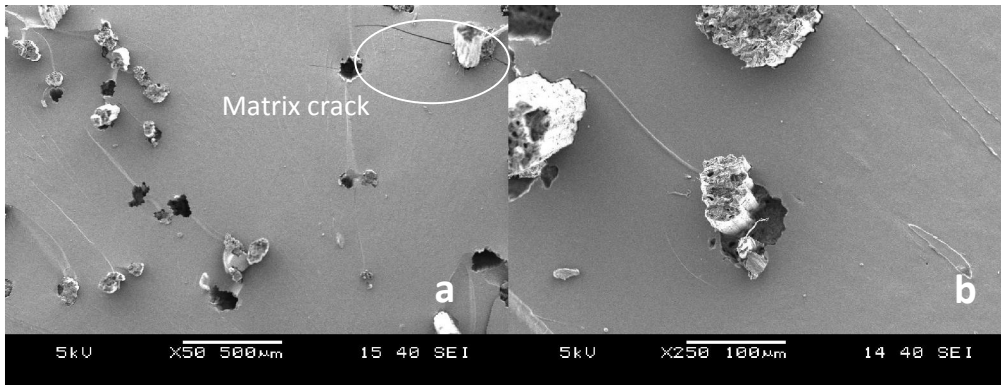
Figure 3.21: Tensile failure surface of UP5-5.
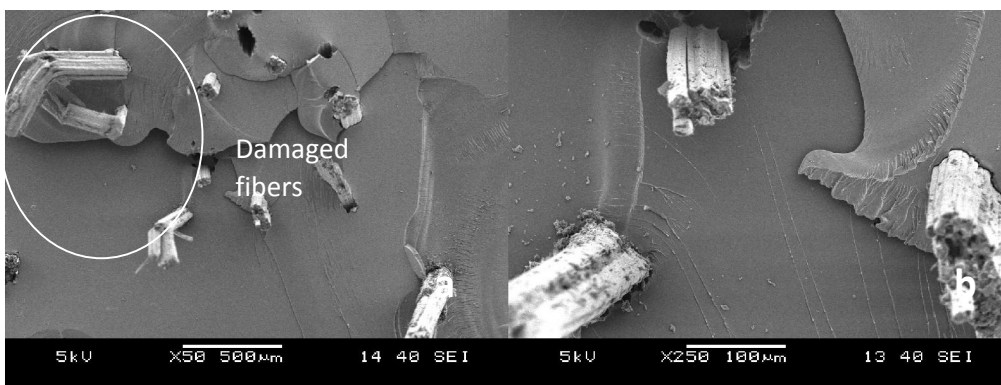
Figure 3.22: Tensile failure surface of UP5-10.
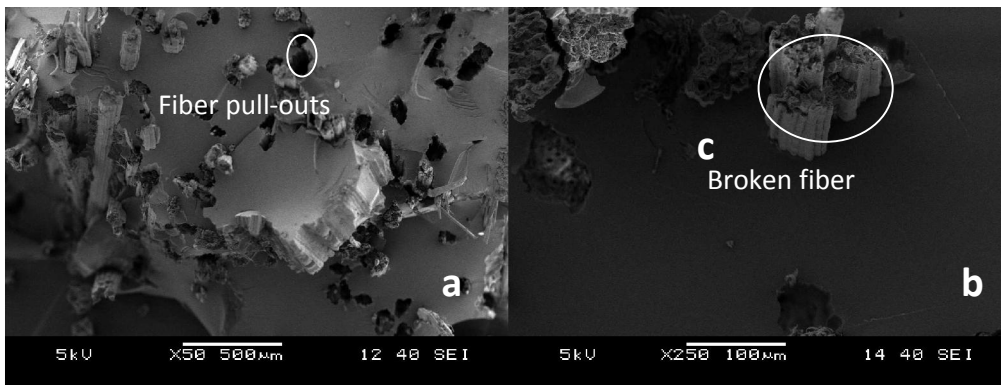
Figure 3.23: Tensile failure surface of UP5-20.
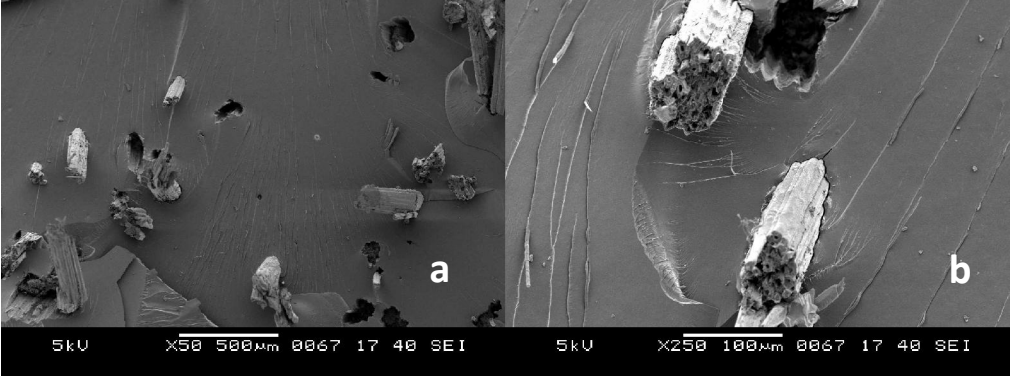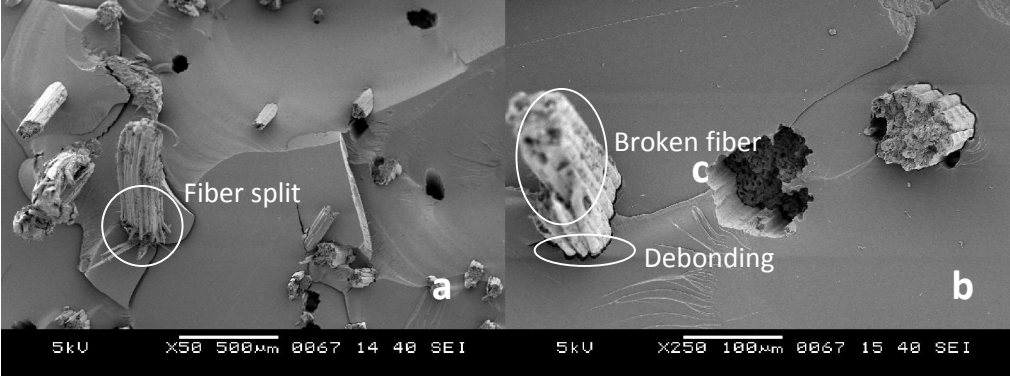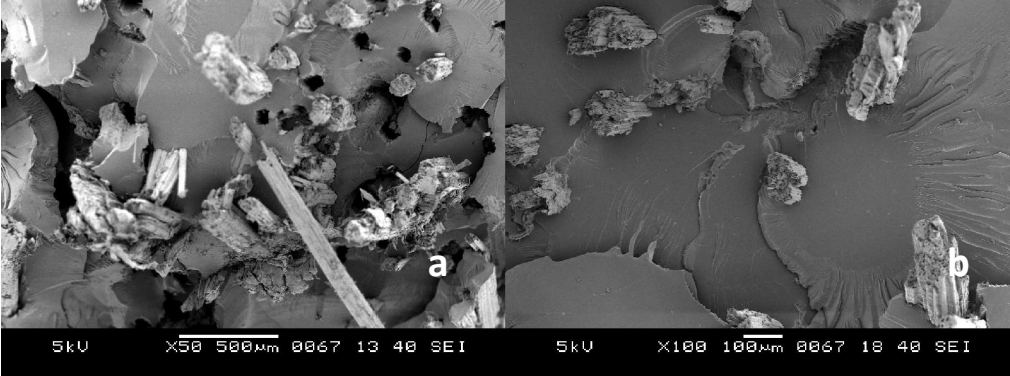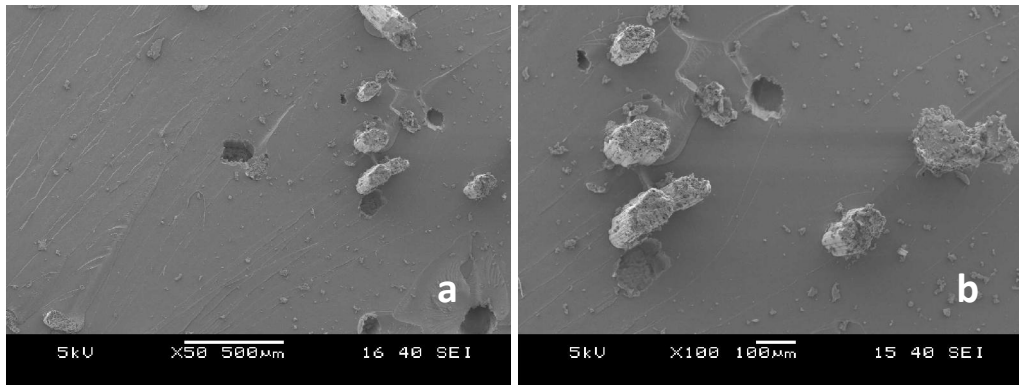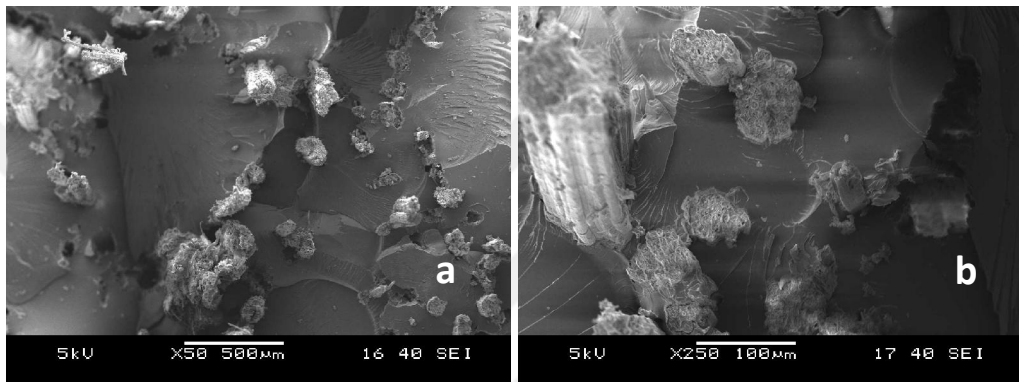
4. CONCLUSION

In this study Althea fibers were extracted by both chemical and water retting methods. Effect of retting method on the chemical and mechanical properties was investigated by using FTIR, XRD, SEM, TGA, and tensile tests.

FTIR result showed that alkali boiling caused compositional changes because some peaks belonged to hemicellulose and lignin were reduced or disappeared. XRD result showed that alkali boiling improved crystallinity index. Crystallinity index of althea fibers increased 13%. Tensile test showed that alkali boiling made fibers more brittle by increasing tensile modulus and strength. Alkali boiling caused removal of surface impurities. Removal of hemicellulose and lignin substances altered decomposition temperature of althea fibers and less hemicellulose and lignin content increased this temperature.

Althea fiber reinforced unsaturated polyester composites were investigated by tensile and flexural tests. It was concluded that tensile and flexural strengths were increased with increasing fiber loadings and lengths. SEM images of fractured samples revealed that adhesion between althea fibers and matrix can be considered as good. Although broken fibers were exist which is evidence of stress transfer from matrix to fiber, there were some fiber pull outs. In table 3.5 mechanical properties of various natural fiber reinforced polyester composites are given.

Table 3.5: Mechanical properties of various natural fiber reinforced polyester composites [61].

Fiber	Volume fraction (%)	Tensile Strength (MPa)	Tensile Modulus (GPa)	Flexural Strength (MPa)
Vakka	37%	66	1.79	93.8
Jowar	40%	124	2.75	134
Sisal	37%	50	1.60	98.1
Banana	37%	60.9	1.08	91.4
Bamboo	37%	121.5	2.23	127.1

AOL fiber reinforced polyester composites have tensile strength of between 25-82 MPa and flexural modulus of between 28-75 MPa. These results support that usage of AOL fibers as reinforcement for polyester composites is suitable. Also some properties such as being annual plant, availability and having strong fibers make AOL suitable plant for fiber extraction.

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