

The Mechanical Properties of Kenaf/Polyester Composite Modified with Sodium Hydroxide and Made by Vacuum Infusion Process

Alireza Setoodeh, Mohammad Sadegh Soltani and Navid Sokhandani
Department of Mechanical and Aerospace Engineering,
Shiraz University of Technology, Shiraz, Iran

Abstract: The use of natural fibers is expanding as an alternative to synthetic fibers in many different industries. The use of natural fibers which once were a dream for environmentalists, now is a main option in producing composites. Natural fibers have advantages such as low density, low cost and compatibility with the environment. Natural fibers are hydrophilic and polar in nature. On the other hand, thermoplastic and thermoset resins are non-polar and hydrophobic. Polar fiber incompatibility with non-polar matrix leads to weak link of the fibers with matrix. One of the most important issues of concern to researchers in the field of bio-composites is the surface modification of natural fibers with chemical agents. One method of making polymer matrix composite which has attracted the attention of industrialists and researchers in the past two decades is making composite by using vacuum infusion method. Vacuum infusion process is a closed-mold method that reduces the toxic and volatile substances. It has advantage such as high quality, low cost, repeatability and manufacturing large parts compared to the hand layup process. In this study, fibers surface modified for 1 h in a solution of sodium hydroxide at a concentration of 5, 10 and 15%. Then, the tensile strength and energy absorbing of kenaf/polyester composite made by vacuum infusion process were studied. Tensile test results showed that the tensile strength of kenaf/polyester composite modified with 10% sodium hydroxide is 207/95 MPa which has an increase of 31.7% compared to composite without surface modification. The results of Thermal Gravimetric Analysis (TGA) and Fourier Transform spectroscopy (FR-IR) of modified fibers compared with those of without modification. To determine the effect of sodium hydroxide on the kenaf fiber we used Scanning Electron Microscope (SEM). To determine the fracture mechanism of kenaf/polyester composite we also used scanning electron microscope.

Key words: Kenaf fibers, natural composite, vacuum infusion, mechanical properties, fourier transform spectroscopy, density

INTRODUCTION

Today, the demand for high performance materials is achievable goal for the engineering industry. There is a need for the advanced materials that have several properties such as mechanical, chemical and electrical properties at the same time. Since, metals and other materials do not have all the above characteristics we should be searching for a way to combine the properties. Composite materials have the potential to combine the properties and to create a material with a higher efficiency than the components. Composite materials have high strength, low weight and high abrasion resistance. The combination of these properties can be found in few substances.

The main constituents of fiber composites are fiber and resin. Fibers form significant volumes of the

composite. Their type, quantity and arrangement are very important and affect tensile, compressive and flexural strength. The mechanical properties of a composite depends on factors such as fiber, the matrix and adhesion between the fibers and matrix. Due to hydroxide groups on the surface of the fibers, the adhesion of natural fibers to the matrix is poor. Many researchers have tried to modify natural fiber surface with chemical agents. In many of these studies, the main goal of researchers is to reduce the polarity of natural fibers. Reduced polarity increases fiber adhesion to matrix which leads to improved mechanical properties.

Edeerozey *et al.* (2007) studied tensile properties of fibers through surface modification of kenaf using sodium hydroxide with different concentrations. In this study, kenaf fibers were put for 3 h in a solution of sodium hydroxide at a concentration of 3, 6 and 9 for surface

modification. They showed that by increasing the concentration of sodium hydroxide from 3-6%, the amount of force required to rupture it in tensile test will be greater compared to the case of not using sodium hydroxide. The reason for increased tensile strength, according to electron microscope pictures is the reduction of impurities of fibers by sodium hydroxide. They also showed that increasing sodium hydroxide concentration from 6-9% causes serious damage to the cellulose polymer network which reduces the tensile strength of the fibers. Thiruchitrambalam and Shanmugam (2012) studied the mechanical properties of composites made by palm tree fiber and polyester resin. In this study, three types of surface modification: sodium chloride, benzoyl chloride and potassium permanganate were implemented on the palm tree fibers. They showed that the highest flexural strength is in the surface modification with potassium permanganate. In another study by Sreekala *et al.* (1997) studied the morphology and properties of natural fibers. In this study, they used sodium hydroxide, acetic acid and silane as modifier of fibers. Results of thermo-gravity analysis showed that reducing the hydroxide groups by using chemical agents, thermal resistance of fibers increases compared to the case without using chemical agents.

Fiore *et al.* (2016) used sodium bicarbonate to modify the surface of natural fiber. In this study, the fiber was put in 10% sodium bicarbonate solution for 24, 120 and 240 h. The results of tensile strength of fibers showed that putting the fibers in sodium bicarbonate solution for 120 h has doubled the tensile strength compared to the case without using sodium bicarbonate.

Mohammad *et al.* (2016) studied the tensile properties, morphology and physical structure of two natural fibers: kenaf and pineapple. In this study, they used two chemicals of sodium hydroxide and silane as surface modifiers. The results of scanning electron microscope showed that the use of the chemical agents reduces the impurities in the fiber.

There are several studies on the properties and behavior of natural fiber composites. Studying their performance, researchers want to improve their mechanical properties based on changes in construction methods and type of surface modification of natural fibers with chemical agents. Rajkumar *et al.* (2015) studied the factors affecting adhesion of natural fibers with polyester matrix. They also demonstrated that the use of chemical agents like sodium hydroxide or aminopropyltriethoxysilane increases the thermal resistance of the fibers against decomposition. Eslami-Frasani (2015) studied the effects of sodium hydroxide on tensile, flexural and Charpy mechanical properties of palm tree fibers. Saha *et al.* (2016) studied the polymer structure of natural fibers. The overall

objective of this study was to investigate the effects of various chemical agents on the reduction of natural fiber polarity. They also studied the effect of various chemical agents on the physical properties of natural fibers. Manalo *et al.* (2015) examined the tensile and flexural properties of natural fiber composite with polyester matrix. In the study, they surface modified the fiber with sodium hydroxide. The main objective of this study was to obtain the final stress and strain with temperature change. They also studied the compressive properties of the composite. The results show that alkaline surface modification increases the compressive strength twofold compared to the case without alkaline surface modification.

Ho *et al.* (2012) in a study cited the factors affecting the manufacture of composites with natural fibers. They also studied the mechanical and thermal properties of natural fibers. Holbery and Houston (2006) examined the use of natural fibers in the automotive industry. Vaisanen *et al.* (2016) in a study reviewed the mechanical and thermal properties of natural fibers in thermoset and thermoplastic resins. Taib *et al.* (2016) in a study showed that surface modification of kenaf fiber with sodium hydroxide changes the morphology and chemical structure of the surface. Their detection method was the use of X-ray photoelectron spectroscopy and atomic force microscopy. Krishna and Kanny (2016) examined kenaf fiber surface modification by using amino acid. They also studied the mechanical properties of kenaf/epoxy composite and showed that the use of surface modification with amino acids can improve thermal stability of kenaf fiber. Ticoalu *et al.* (2010) studied the mechanical properties of various types of natural fibers. They also examined the mechanical properties of composites reinforced with natural fibers made by various methods. One of the most important points of this study was the investigation of the potential and industrial applications of composites reinforced with natural fibers.

One method of making polymer matrix composite which has attracted the attention of industrialists and researchers in the past two decades is making composite by using vacuum infusion method. Vacuum infusion process in a closed-mold method that reduces the toxic and volatile substances. It has advantages such as high quality, low cost, repeatability and manufacturing large parts compared to the hand layup process. This method is widely used in the manufacture of large composite parts such as wind turbines, pressure vessels, ferry panel, parts of a bridge and (Atafar *et al.*, 2013). Kedari *et al.* (2011) designed an experiment to review the cavity in the fiber and fiber volume fraction. In the experiment, they used polyester resin and fiberglass to test seven different modes through the vacuum infusion method. They showed that by increasing output pressure, the amount of

cavity is reduced in the manufacture of composite with vacuum infusion method. Johnson and Pitchumani (2008) in order to control the flow of resin in the process of resin infusion to the mold by heating the mold reduced the viscosity and velocity of the resin. They reduced the dry points in the composite by this method.

The analysis of the processes carried out in vacuum infusion process is very complex and difficult because thermal, chemical and mechanical properties equations must be investigated at the same time. So, the simulation of the resin cure process in mold and prediction of the properties of the final product in order to produce high quality components is very significant. Symington *et al.* in an study considered the problems and benefits of this method. They also investigated the strength of materials made in this way. Atas *et al.* (2011) compared the impulse response of composites made in two ways: hand layup method and vacuum infusion method. They showed that due to the high volume percentage of fiber, composite made by vacuum infusion is of greater strength and shows more impact resistance. In an applied research (Dweib *et al.*, 2004), using vacuum infusion maderooof sandwich panels by natural cotton fiber, paper pulp and chicken feathers. They investigated the flexural properties of each of the fibers used in sandwich panel. Kuentzer *et al.* (2007) investigated the effects of infusing additional resin and resin flow resistance at the outlet on the cavities created in the manufacture of composite using vacuum infusion method. They showed that if the resin in output is not affected by external force, the created cavities will be distributed non-uniformly in the composite. Francucci *et al.* (2012) measured capillary pressure while infusing resin to fiber in the composite of jute/vinyl ester which was made by vacuum infusion method. They analyzed the effect of capillary force on the permeability of jute fiber. They found that the capillary force in natural fiber of jute is greater than synthetic fibers.

In this study, surface modification with different concentrations of sodium hydroxide made on kenaf fibers. Then the effect of sodium hydroxide on kenaf fibers was investigated by two tests IR and TGA. Finally, by making polyester kenaf composite using vacuum infusion method, we compared the mechanical properties of the resulting composites.

MATERIALS AND METHODS

Construction method: For kenaf fiber surface modification, four layers of the fiber are woven around a steel frame. Woven fibers were put into a solution of sodium hydroxide at a concentration of 5, 10 and 15%



Fig. 1: Four layers of kenaf fibers woven around a steel frame



Fig. 2: The matrix in the vacuum infusion method

for 1 h. For example, to create a sodium hydroxide solution with a concentration of 5% we need to dissolve 250 g of sodium hydroxide in 5 L of water. After 1 h, fibers are removed from sodium hydroxide solution and washed several times with water. After ensuring the withdrawal of excess sodium hydroxide in the final stage, fiber is washed with distilled water and it is put in an oven at 40°C for 24 h. Figure 1 shows the kenaf fibers woven around a steel frame (Fig. 1).

During the chemical process, sodium hydroxide is separated to its ions (Na^+ and OH^-). Then, hydrogen is separated from hydroxyl agent of the fiber and sodium ion relink with the oxygen of hydroxyl agent (Li *et al.*, 2007).

After weaving four layers of kenaf fiber, zone of the vacuum infusion process is covered with layer of separator material and then the fibers are put in the zone. Both ends of the fibers are glued to fix them. Due to absorb additional resins and improve the quality of composite surface, Dacron is put on the fibers. Then, a net layer is used for appropriate distribution in faster movement of the resin. The initial layout of the vacuum infusion process can be observed in Fig. 2. After that polyester

resins composed with 1% of the hardener material and then bubbles of mixing the resin and hardener is destroyed by putting in degassing chamber. The resulting resins with suitable vacuum is infused in prepared matrix. Parts are not cut from the surface for 3 days and after removing parts from the matrix, Dacron fabric and distribution layer are separated from the parts to post-cure the parts. In order to cure, fragments are put at 80°C for 3 h (Fig. 2).

For tensile test, parts are prepared according to the standard of ASTM D 3039 and for tensile test of raw resins the standard of ASTM D 638 is used. Three pieces of each sample was tested and mean value was calculated and reported as the final outcome. ISO 178 standard is used to three-point bending test. According to ISO 179-1, Charpy test was done on composites reinforced with kenaf.

RESULTS AND DISCUSSION

Due to the extent of content and easier comparison, abbreviations are used. These abbreviations are listed in Table 1.

Fourier Transform spectroscopy (FR-IR): Fourier Transform spectroscopy (FR-IR) is an appropriate method for analyzing chemical changes on the chemical structure of kenaf fiber. Fourier Transform spectroscopy (FR-IR) analysis for fibers modified with sodium hydroxide (KN) and fiber without surface modification (K) are shown. The test was conducted in the laboratory of the Department of Chemistry in Shiraz University.

Fluctuations in different bands for the modified fibers and fibers without surface modification have been listed in Table 1. The fluctuations are related to cellulose, hemicellulose and lignin.

According to the analysis of Fourier Transform spectroscopy (FR-IR) of non-modified fibers, it appears that the hydroxide groups are very high within the band 3443. The presence of hydroxide groups means the polarity of the substance. Reduction in the hydrogen bond reduces the C-H bond in the fibers which reduces the absorption in bands 2925 and 2858 compared to the case with non-modified surface. In the range of 900-1750

the severity of many peaks is reduced. Reduction of absorption in this range is due to the reduction of impurities in the fibers as well as the reduction of hydroxide groups in the hemicellulose and lignin. The absorption at wave number 1600 reduces with the increase in the concentrations of sodium hydroxide. This absorption relates to C=C bonds or lignin aromatic chains. By increasing the concentration of sodium hydroxide, lignin of fibers is reduced. Absorption in band 891 relates to the glycoside bond. Glycoside has bonded to a non-carbohydrate part in the fiber which increasing the concentration of sodium hydroxide, glycoside is reduced in the fiber (Table 2).

The results of thermo gravimetric analysis: Thermo gravimetric analysis is done for the physical changes and chemical reactions and then quantitative measurement of sample weight changes. In other words, continuous weighing of samples is done in a controlled atmosphere while the temperature of the sample is rising at a certain rate. The test was conducted in the laboratory of Shiraz University of Medical Sciences.

By changing the chemical structure of the fibers using sodium hydroxide, heat-fiber degradation will be changed. The first weight loss is in the temperature range of 25-70°C. In this temperature range, humidity absorbed by the fiber will be brought out. Reducing the hydroxide groups, moisture absorption will also be reduced which causes the fibers modified in this temperature range to show less weight loss. The second fiber decomposition temperature is in the range of 220-300°C. In this temperature range, hemicellulose and a part of lignin of the fiber is broken. The third fiber decomposition temperature range is between 300 and 400°C. In this temperature range, the much of fiber cellulose is destroyed. In general, the more the amount of fiber cellulose, the higher the heat resistance of the fiber. Doing alkaline surface modification on the fiber, the share of cellulose is increased due to the reduction of impurities and hemicellulose. So, the thermal resistance of the fibers increases compared to the non-modified case. According to samples KN-10 and KN-15 with increasing sodium hydroxide concentration from 10-15%, thermal resistance of the fiber is reduced. Increasing the concentration of

Table 1: Number of specimens and corresponding abbreviations

Surface operation	Abbreviation	Material	Row
No operation	K	Kenaf fiber	1
Modified kenaf with 5% sodium hydroxide	KN-5	Kenaf fiber	2
Modified kenaf with 10% sodium hydroxide	KN-10	Kenaf fiber	3
Modified kenaf with 15% sodium hydroxide	KN-15	Kenaf fiber	4
Kenaf composite/polyester without surface modification	KU	Kenaf composite/polyester	5
Kenaf composite/polyester modified with 5% sodium hydroxide	KNU-5	Kenaf composite/polyester	6
Kenaf composite/polyester modified with 10% sodium hydroxide	KNU-10	Kenaf composite/polyester	7

Table 2: The wave number and type of bonds

Bond	Wave number (cm ⁻¹)
O-H (cellulose and hemicellulose)	3500
C-H (cellulose and hemicellulose)	2925
C=O (carbonyl)	1744
Aldehyde CHO (lignin)	1650
Ketone-C=O (lignin)	1637
CH ₂ (lignin)	1458
C-H (cellulose)	1379
Glycoside chains	893

sodium hydroxide will damage and fragment the polymer network of the cellulose. Separating the polymer network of the cellulose will reduce the thermal resistance of the fiber. Surface modification with 10% sodium hydroxide in the sample KN-10 will increase the thermal resistance of the fibers by 7% compared to non-modified fibers in the sample K. In general, the less the amount of hydroxide groups in the material, the more the thermal resistance. As already mentioned in the spectroscopy analysis, sodium hydroxide reduces the hydroxide groups in the fiber.

The results of tensile test of kenaf/polyester composite using sodium hydroxide for surface modification: This section examines the tensile properties of kenaf/polyester composite. After weaving four layers of kenaf fiber around a steel frame and doing surface modification with different percentage of sodium hydroxide, kenaf/polyester composite is made through vacuum infusion process and its tensile is tested.

The ultimate stress of KNU-10 is 207/95 MPa which has increased by 31/7% compared to KU. This increase in the strength is due to the stronger and better linking of fibers with the polymer matrix. The infrared spectroscopy with surface modification of natural fibers by sodium hydroxide, fiber polarity is reduced and this leads to a stronger bond of the fibers with non-polar matrix. The ultimate stress of KNU-15 is 31/166 MPa. The reduction of ultimate stress with the increase of sodium hydroxide concentration may be due to the destruction of cellulose polymer structure. As seen in the microscopic photos of the fibers with increasing the concentration of the modifier material, fiber is destroyed. Given the initial slope of the stress-strain curve (range 30-45 MPa) of samples KU, KNU-5, KNU-10 and KNU-15, it is determined that if the bond of the fiber with the matrix is better, micro-cracks in the matrix occurs in more stress. The simultaneous effect of elastic modulus, tensile strength and fracture strain of composite material can be seen in the area under the stress-strain curve or so called toughness of the material.

The highest toughness is in the sample KNU-10 which has increased by 19% compared to the sample KU. Toughness increase means the increase in the absorbed

energy up to fracture. In the KNU-10 due to a stronger link of fiber with the matrix, the composite toughness has increased compared to KU. By increasing the concentration of sodium hydroxide in KNU-15, the toughness is reduced due to the fiber damage.

SEM image of the kenaf/polyester composite to determine the fracture mechanism: In fact, due to the lack of adhesion between the matrix and fibers, transmitting stress from the matrix to the fiber is decreased. Therefore, fracture probability is decreased and fibers often tend to be pulled out from the matrix. In other words, fiber prefer to leave the matrix to be fractured. In addition to the fiber pull-out, a limited number of fiber fracture is observed which reflects simultaneous presence of the fracture and fiber pull-out. However, the predominant mechanism is fiber pull-out.

CONCLUSION

In this research, to create better links between the natural fibers with resin, polyester/kenaf fibers were put in sodium hydroxide solutions with concentrations of 5, 10 and 15% for 1 h. Then, to observe the effect of the surface modification, both tests of IR and TGA were used. The IR analysis showed that the hydroxide bond has been established. The TGA test showed that by creating hydroxide bond on kenaf fibers, the fibers are destroyed by heat later. Then, using the tensile test and Charpy impact on two samples of kenaf/polyester composite, ultimate strength and the amount of energy absorbed by the impact were obtained. Tensile test results showed that the tensile strength of kenaf/polyester composite modified with 10% sodium hydroxide was 207/95 MPa which has increased by 31/7% compared to the case without surface modification. The results of this study indicated that kenaf fibers modified by 10% sodium hydroxide show better mechanical properties and have better adhesion to polyester resin. These two factors increase the mechanical properties of kenaf/polyester composite.

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