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Improving Mechanical Properties of Kenaf/Polyester Composites Using Sodium Hydroxide Treatment and Fabricated by Vacuum Infusion Process

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Abstract: The use of natural fibers is expanding as an alternative to synthetic fibers in many different industries. Theuse of natural fibers which once was a dream for environment a lists 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 also hydrophilic and polar in nature. On the other hand, thermoplastic and the rmoset 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 treatment of natural fibers with chemical agents. One method of making polymer matrix composite which has attracted the attention of manufacturers and researchers in the past two decadesis making composite by using Vacuum Infusion Process (VIP). The VIP is 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 tothe hand-lay up process. In this study, fibers surface are pre-treated for 1 h in a solution of sodium hydroxide with different concentrations of 5, 10 and 15%. Then, the tensile strength and energy absorbing of the kenaf/polyester composites made by VIP are studied. The tensile test results demonstrate that the ultimate strength of the kenaf/polyester compositepre-treated with 10% sodium hydroxide is 207.95 MPa which exhibits a significantly increase of 31.7% compared to the composite without surface treatment. To comprehensively studying the influences of surface treatment on fibers, Thermal Gravimetric Analysis (TGA) and Fourier transform spectroscopy (FR-IR) tests are prepared. Furthermore, Scanning Electron Microscope (SEM) is employed to investigate the fracture mechanism of bio-composite as well as the effect of sodium hydroxide on thekenaf fibers treatment.

Key words: Kenaf fibers, bio-composite, vacuum infusion process, mechanical properties, thermal gravimetric

INTRODUCTION

Todays, the demand for high performance materials is achievable goal for the engineering industry. There is a need for the advanced materials that have simultaneously 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 mechanical properties of a composite depend 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.

In this regard, Edeerozey *et al*. (2007) studied tensile properties of fibers through surface treatment of kenafusing 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 would be greater compared to the case of not using sodium hydroxide. The reason for increased tensile strength according to SEM pictures was the reduction of impurities of fibers by sodium hydroxide. They also showed that increasing sodium hydroxide concentration

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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 fibers and polyester resin. In this study, three types of surface treatment; 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, 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. The results of thermo-gravity analysis showed that the thermal resistance of fibers increases compared to the case without using chemical agents due to reduction of the hydroxide groups.

Fiore *et al*. (2016) used sodium bicarbonate to modify the surface of natural fibers. In this study, the fibers were 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 can double the tensile strength. Mohammad *et al*. (2016) studied the tensile properties, morphology and physical structure of two natural fibers of kenaf and pineapple. They used two chemicals of sodium hydroxide and silane as surface treatments. The results of SEM showed that the use of the chemical agents reduces the impurities in the fiber.

There are several studies which are focused on the properties and behavior of natural fiber composites. In this context the goal is improving themechanical properties based on changes in fabrication methods and types of surface treatment of natural fibers with chemical agents. Rajkumar studied the factors affecting adhesion of natural fibers with polyester matrix. Eslami-Frasany (2015) investigated the effects of sodium hydroxide on tensile, flexural and charpymechanical properties of palm tree fibers. Considered the polymer structure of natural fibers to investigate the effects of various chemical agents on the reduction of natural fiber polarity. Manalo *et al*. (2015) examined the tensile and flexural properties of natural fiber composite with polyester matrix wherein the fibers pre-treated with sodium hydroxide to obtain the ultimate strength and strain with temperature change.

In a study, Ho *et al*. (2012) cited the factors affecting the fabrication of composites having 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) reviewed the mechanical and thermal properties of natural fibers in thermoset and thermoplastic resins. Taib *et al*. (2016) showed that surface treatment of kenaf fiber composites and the corresponding effects using X-ray photoelectron spectroscopy and atomic force microscopy. Krishna and Kanny (2016) investigated the mechanical properties of kenaf/epoxy composites and showed that the use of surface treatment with amino acids can improve thermal stability of kenaf fibers. Ticoalu *et al*. (2010) studied 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.

Recently, VIP has attracted the attention of different industries and researchersas a closed-mold method that reduces the toxic and volatile substances for workers in comparison with the hand-layup process. This method is widely used in the manufacture of large composite parts with high quality surfaces such as wind turbines, pressure vessels, ferry panel, structure of bridges (Atafar *et al*., 2013; Kedari *et al*., 2011) designed an experiment to investigate the influences of different parameters on producing fiber glass/polyester composites using VIP. They showed that by increasing output pressure, the amount of cavity is reduced in fabrication of composite. Tried to control the flow of resin by reducing the viscosity and velocity of the resinwith heating the mold. Tahmassebpour (2016) investigated the strength of materials made by VIP and reported some benefitsand difficulties of this method. Atas *et al*. (2011) compared the impact response of composites made by two methods of hand-layup and VIP. They showed that due to the high volume percentage of fiber, composites made by VIP exhibit greater strength and higher impact resistance. In an applied research, Dweib *et al*. (2004). Maderoof sandwich panels by natural cotton fiber, paper pulp and chicken feathers using VIP. They investigated the flexural properties of each of bio-composites used in sandwich panel. Kuentzer *et al*. (2007) studied the effects of injecting additional resin and resin flow resistance on the cavities created in the fabrication of composite madeby VIP. Francucci *et al*. (2012) measured capillary pressure of injected resin during fabrication of jute/vinyl estercomposite. They found that the capillary force in natural fiber of jute is greater than synthetic fibers.

In this study, surface treatment with different concentrations of sodium hydroxide made on kenaf fibers. Then the effect of sodium hydroxide on kenaf fibers was investigated by two tests of IR and TGA. Finally, the mechanical properties of the resulted composites are examinedby producing polyester kenaf composites using vacuum infusion process.

MATERIALS AND METHODS

Fabrication method: For kenaf fiber surface treatment, 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% 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 with drawal 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.

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 reacts with the oxygen of hydroxyl agent. Reaction of sodium hydroxide with kenaf fibers:

$$\text{Fiber-OH} + \text{NaOH} \Rightarrow \text{Fiber-O}^{-}\text{Na}^{+} + H_2O$$

Water is a byproduct of the process. The chemical process schematicis shows above (Li *et al*., 2007). After weaving four layers of kenaf fibers, zone of the vacuum infusion process is covered with a layer of separator material and then the fibers are put in the zone. Both ends of the fiberis glued to fix them. Due to absorb additional resins and improve the quality of composite surface, Dacronis put on the fibers. Then, a Distributed Mesh (DM) layeris used for appropriate distribution and faster movement of the resin. The initial layout of the VIP can be observed in Fig. 2. After that, polyester resinis combined with 1% of the hardener material and then bubbles of mixing the resin and hardener is destroyed by putting it in degassing chamber. The resulting resin with suitable vacuumis injected in prepared matrix. The parts are not separated from the surface for 3 days and after that dacron fabric and DM layer are separated from the parts to post-cure the parts. In order tocure, fragments are put at 80°C for 3 h.

For tensile test, parts are trimmed according to the standard of ASTM D3039 while the standard of ASTM D638 is used for tensile test of raw resin. Three specimens of each materialwere tested and the mean valueis calculated and reported as the final data. The ISO 178 standard is used for three-point bending test. Furthermore, according to ISO 179-1, Charpy tests arecarried out on composite specimens reinforced with kenaf.

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

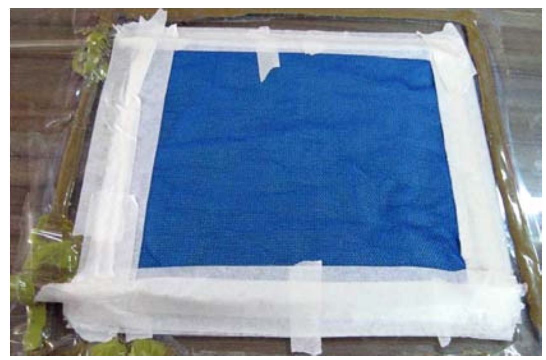

Fig. 2: Fabrication of specimen by VIP

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 is an appropriate method for analyzing chemical changes on the chemical structure of kenaf fiber. The FR-IR analysis for fibers pre-treated with sodium hydroxide (KN) and fibers without surface treatment (K) are shown in Fig. 3. The tests are conducted in the laboratory of Chemistry Department in Shiraz University.

The fluctuations in different bands for the pre-treated fibers and fibers without treatment are listed in Table 2. The fluctuations are related to cellulose, hemicellulose and lignin.

According to the analysis of FR-IR of non-treated fibers, it appears that the hydroxide groups are very high within the band 3443. The presence of hydroxide groups

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

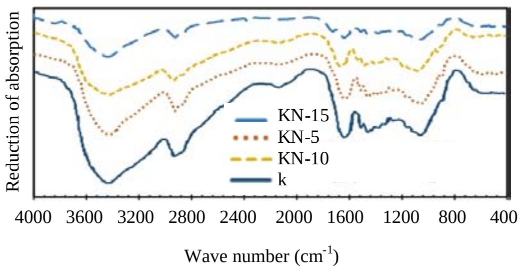

Fig. 3: Infrared spectroscopy analysis for surface pre treated fibers and fibers without treatment

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

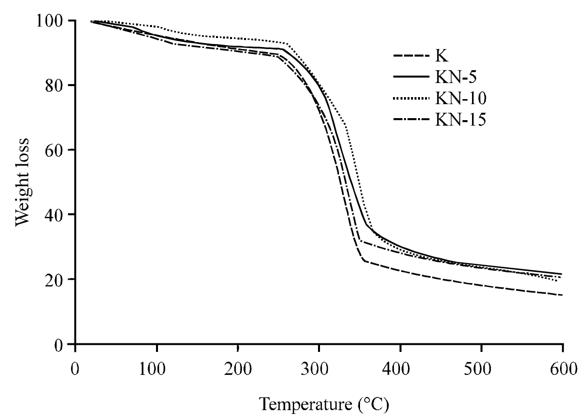

Fig. 4: Thermo-gravimetric analysis of fibers under alkaline surface treatment

means the polarity of the substance. Figure 3 shows that the more the concentration of sodium hydroxide, thelesserthe absorption within band 3443 and this means the reducing of fiber polarization. The presence of hydroxide groups means the presence of hydrogen bonds. 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 picks is reduced. The 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 increasein 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 fibersis reduced. Also, the absorption in band 891 relates to the glycoside bond. Glycosideis bonded to a non-carbohydrate part in the fiber then glycoside is reduced in the fiber with increasing the concentration of sodium hydroxide.

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 samplesis carried out in a controlled atmosphere while the temperature of the sample is rising at a certain rate. Figure 4 and 5 shows the thermo-gravimetric analysis of kenaf fiber pre-treated with sodium hydroxide (KN) as well as kenaf fibers without surface treatment (K). The tests are 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. According to Fig. 4, the destruction of the fibers occurs in 3 range of 50-120, 220-300 and 300-400°C. 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. According to Fig. 4, by modifying kenaf fibers with sodium hydroxide, the hydroxide groups in the fibersare decreased. Reducing the hydroxide groups, moisture absorption will also be reduced which causes the pre-treated fibers show less weight loss in this temperature range. 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 most part of fiber

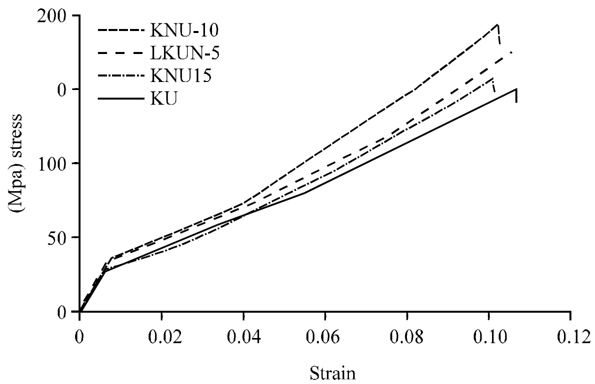

Fig. 5: Stress-strain diagram of kenaf/polyester composite

cellulose is destroyed. In general, the more the amount of fiber cellulose, the higher the heat resistance of the fiberis observed. Doing alkaline surface treatment 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-treated case. According to samples KN-10 and KN-15 with increasing sodium hydroxide concentration from 10-15%, the thermal resistance of the fiber is reduced. Increasing the concentration of sodium hydroxid eleads to damage and fragment of the cellulose polymer chain. Consequently, the thermal resistance of the fiberis significantly reduced. Surface treatment with 10% sodium hydroxide in the sample KN-10 increases the thermal resistance of the fibers by 7% t compared to non-treated fibers in the sample K. Mainly, the less the amount of hydroxide groups in the material, the more thethermal resistance. As already mentioned in the spectroscopy analysis, sodium hydroxide reduces the hydroxide groups in the fiber.

Tensile test of pre-treated kenaf/polyester composites: This study examines the tensile properties of kenaf/polyester composites. After weaving four layers of kenaf fibers around a steel frame and doing surface treatment with different percentages of sodium hydroxide, kenaf/polyester composite is made by VIP. After wards, tensile tests are carried outon the standard specimens. The stress-strain diagram of composite can be seen in Fig. 5.

According to Fig. 5, the ultimate stress of KU is equal to 156.41 MPa. The ultimate strength of KNU-10 is 207.95 MPa which is increased by amount of 31.7% compared to KU sample. This increase in the strength is due to the stronger and better linking of fibers with the polymer matrix. As mentioned in the infrared spectros

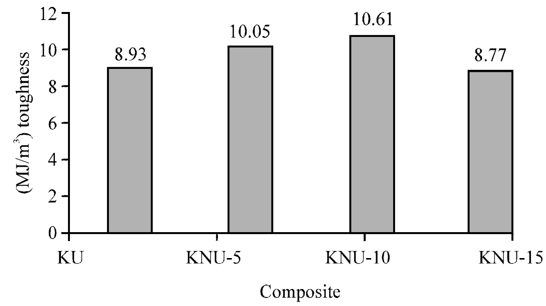

Fig. 6: Variations of toughness by increasing the concentration of sodium hydroxide

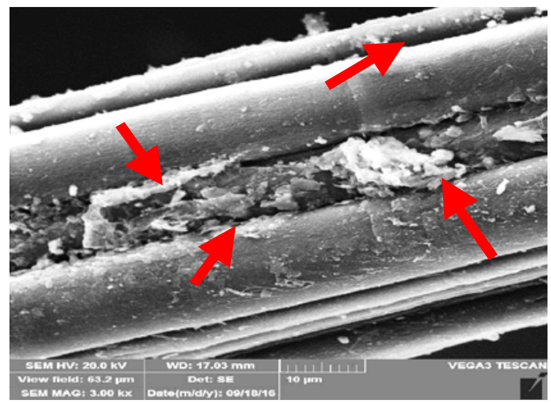

Fig. 7: SEM image of K sample

copy with surface treatment of natural fibers by sodium hydroxide, fiber polarity is reduced and this leads to a stronger bond of the fibers with non-polar matrix. It is also seen that the ultimate strength 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 treatment material, fiber is destroyed. Given the initial scope 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 improved, micro-cracks in the matrix occur in higher stress levels.

Figure 6 demonstrates the diagram ofvariations of toughness specimens with different ranges of surface treatment. The highest toughnessis observedin the sample KNU-10 which shows as increase of 19% compared to the sample KU. The toughness increase means the increase in the absorbed energy up to fracture. In the KNU-10 sample, due to a stronger link of fiber with the matrix, the composite toughness increases compared comparing Fig. 7 and 8, it can be concluded

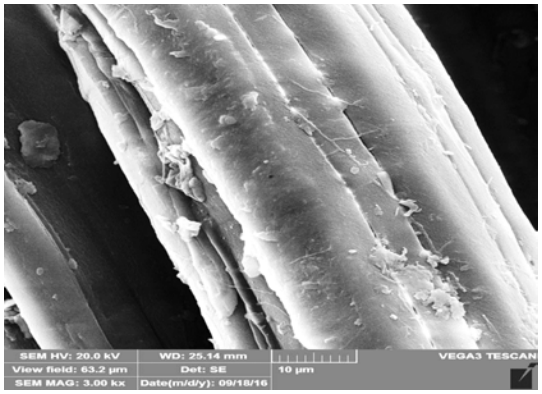

Fig. 8: SEM image of KN-10 sample

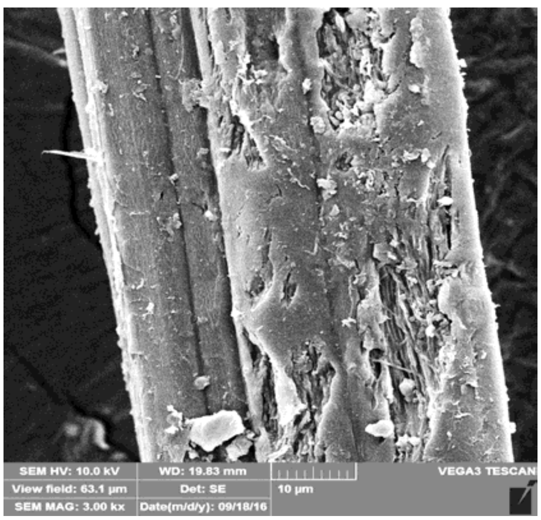

Fig. 9: SEM image of KN-15 sample

that surface treatment reduces impurities and flaws. As previously mentioned inthe fiber tensile test, the impurities act as stress concentration points and reduce the amount of ultimate strength and Youn's modulus. Figure 9 is the SEM image of KN-15. As observed in the image, increasing the time of modifier material concentration yields to damage of the fiber. The destruction of the fiber decreases mechanical strength.

Fracture mechanism of the kenaf/polyester composite: Figure 10 depicts the SEM image offracture surface of KU sample in different magnifications. The image sclearly exhibit the fiber pull-out phenomenon. In fact, due to the lack of adhesion between the matrix and fibers, transmition of stress from the matrixtothe fiber is decreased. Therefore, the possibility of composite fracture is decreased and fibers often tend to be pulled out from the matrix. In other words, fiber prefer to leave the matrix. In addition to the fiber pull-out, a limited number of fiber

Fig. 10: KU offracture at different magnificatios

fracture is observed which reflects simultaneous presence of the fracture and fiber pull-out. However, the predominent mechanism is fiber pull-out. Figure 11 shows scaning electron microscope image of KNU-15 specimen. As is clear from these images, fiber pull-out is almost not dominent and in this case fibers prefer to be fractured. In fact, due to strong adhesion between the matrix and fiber, stress transferred from the matrix to the fiber and the fiber pull-out is reduced. In addition to the fracture of the fibers, some fibers came out that indicates the

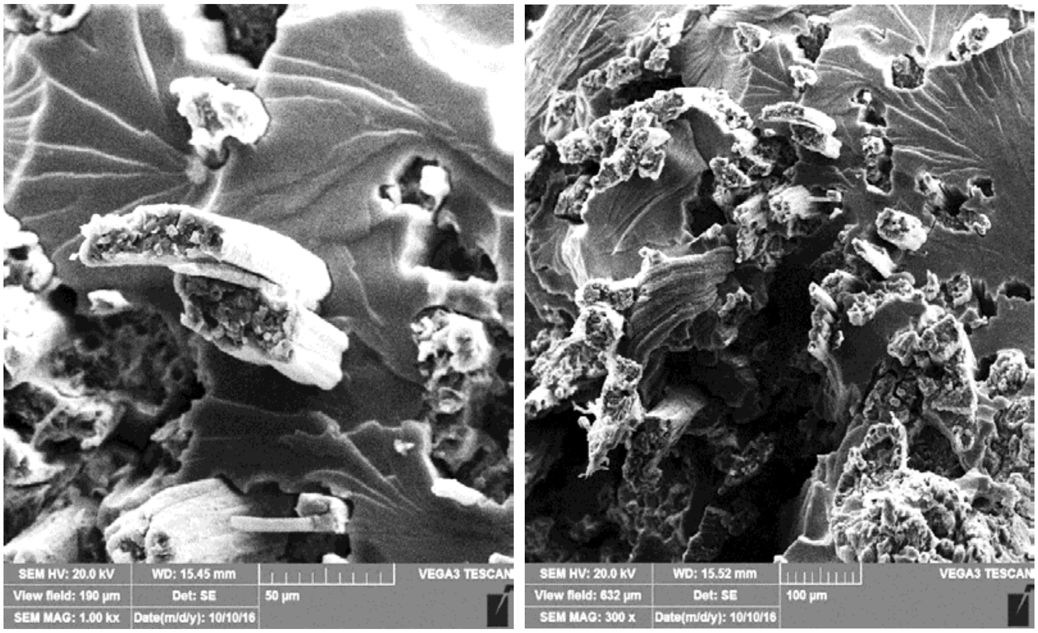

Fig. 11: SEM image of KNU-15 specimen at different magnifications

simultainious presence of two mechanisms of fracture and fiber pull-out. However, it can be said that the fiber fracture is the dominant mechanism.

CONCLUSION

To exhibit a practical application of bio-compsites with improved mechanical properties, a kenaf/polester composite made by vacuum infusion process as a closed-mold method is successfully fabricated. In this research, to create better links between the natural fibers with resin, kenaf fibers were initially put in sodium hydroxide solutions with concentrations of 5, 10 and 15% for 1 h. Then, to observe the effect of the surface treatment both tests of IR and TGA were used. The IR analysis showed that the hydroxidebondwas established. The TGA test showed that by creating hydroxide bond on kenaf fibers the fibers werte destroyed by heat later. Afterwards by conducting the tensile and Charpy impacttests on standard samples of kenaf/polyester composites, the ultimate strength and the amount of energy absorbed by the specimens were obtained. The tensile test results demonstrated that the tensile strength of pre-treated kenaf/polyester composites with 10% sodium hydroxide was 207.95 MPa which presents a significantly enhance of 31.7% compared to the samples without surface treatment. The results of this study indicated that kenaffibers pre-treated by 10% sodium hydroxide show better mechanical properties and have better adhesion to polyester resin. These two factors significantly increased the mechanical properties of kenaf/polyester composite and the introduced procedure can be employed in different industries for developing low cost and natural composites.

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