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Effect of Alkalization Treatment on The Tensile Strength and Interface Character Matrix-Fibber of Bamboo Petung (Dendrocalamus Asper) Reinforced Polyester Resin Composite

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Abstract. The purpose of this study is to determine the alkalization process effect of bamboo petung fiber (Dendrocalamus Asper) on the Tensile strength of Polyester composite reinforced with bamboo petung fiber. The alkalization process is expected to increase the bond between the automatic and the fiber which in turn can increase the tensile strength of the composite. In this study NaOH was used as an alkaline substance. NaOH is an alkali commonly used as a substance for the alkalization process in various natural fibers for natural composite reinforcing material. Alkalization was done by soaking the fiber into NaOH solution at room temperature and time variation for 1 hour, 2 hours, and 3 hours. After the alacalization process, the fiber was processed to be made into a composite with a polyester matrix with the direction of unidirectional fiber orientation. The process of making composites used vacuum infusion method. Before being used as reinforcing fibers, petung bamboo fiber composites were tested for single fiber tensile strength both after alkalization treatment and fiber before alkalization process. Composite tensile testing was conducted on all variations of both composites with fibers before Alkalization and composite after the alkalization process by using ASTM D638-03 standard. Scaning Electronics Microscope (SEM) was also conducted to observe the morphology of fibers before and after alkalization, besides that SEM was also used to determine the fracture shape of polyester composite reinforced with bamboo petung fiber. From the Tensile testing conducted on single fiber and composite, it was obtained results that either the single fibers strength or composites decreased with increasing NaOH soaking time.

Keywords; Bamboo Petung, natural fiber composite, Uniderectional fiber composite, Alkalization

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1. Introduction

Bamboo Petung plays an important role in rural communities, especially in Indonesia. Bamboo material is known by the society that has good properties to be utilized i.e. strong, resilient, straight, flat, hard, easy to be splitted, easy to be formed, easy to be worked on, as well as its light material therefore it easy to be carried. Besides that bamboo is also relatively inexpensive compared to other building materials because it is found a lot around rural settlements. Bamboo is a versatile plant for rural Indonesian people.

There are several bamboo types in Indonesia, namely Tali Bamboo (Gigantochloa apus), Java black bamboo/wulung bamboo (Gigantochloa atroviolacea), common bamboo/ampel bamboo (Bambusa vulgaris), giant bamboo/petung bamboo (Dendrocalamus asper), golden bamboo (Bambusa vulgaris var. Striata), and tulup bamboo. Petung bamboo has a significant percentage in Indonesia, which is around 22.8% [1]. Petung bamboo has a fairly good seedling growth percentage of 52% so that the bamboo fast growth becomes an ease when used as raw material. The qualified strength and the easy fibre processing become an advantage [2]. Holocellulose content (73.63%), lignin (27.37%) and fiber cell wall thickness (0.90 microns) of Petung bamboo is more than sero bamboo/golden bali bamboo (Schizostachyum brachycladum) (71.96%; 26.18%; 0.80 microns) and tui bamboo (Schizostachyum lima) (72.77%; 26.05%; 0.77 microns). Cell wall thickness will greatly affect the shrinkage; the thicker the cell wall, the greater the shrinkage will occur. In addition to cell wall thickness factors, another factor related to the water content in bamboo is the pore cells number. Pore cells contain more water than fiber cells.

In polymer composites reinforced with natural fibers, the interface character matrix-fiber needs to be considered [3]. This is related to the compatibility between fiber and matrix and fibers hydrophilic characters. Alkalization is one of natural fibers modifying methods to improve compatibility between matrixes [4]. Alkalization treatment on fibers causes hemicellulose partial loss; lignin found on the fiber surface, fiber surface tofography becomes coarser, resulting in better mechanical interlocking with the matrix. With the loss of lignin on the fiber surface, the reaction fields and chemical bonds between fibers and matrix increase which will improve their mechanical properties [5].

NaOH is one of alkali type which is popularly used for surface modification of natural fibers. The alkalization principle with NaOH is by binding OH- which is in the fiber (hemicellulose, cellulose, and lignin) so that the interface of the fiber and matrix is getting better which then can increase the mechanical strength of the composite [6] [7].

Composite is a material that is a combination or mixture of two or more materials on a macroscopic scale to form a third material that is more useful. In addition to petung bamboo fibers used as reinforcing materials, another thing that needs to be considered is the binding of fibers (matrixes) [8, 9]. On the other hand, the composite manufacturing process which is considered effective is the Resin Tansfer Molding (RTM) method or commonly called resin infusion, it is a composites creation method of using low pressure applications to regulate the resin sequence into lamina.

The matrix material choice is taken from materials that have resilient properties to be able to continue and endure the pressure and shear stress received. But for this research, fibrous composites with relatively brittle matrixes were developed, so polyester was used. Polyester as a matrix can make a connection between the absorption of pseudo-static energy from various fibers and composite dynamic energy absorption [10]. This is the reason for the use of extensive polyester matrix composites, because of low prices, high strength, and relatively simple manufacturing processes [11, 12]

The combination of bamboo fiber as reinforcement then using polyester as its matrix is believed to be a breakthrough in the composite materials creation innovation that are able to be applied to significantly influence the results of mechanical and physical strength. With the creation of this material, it is expected that a revolution in the environment, especially from the use in the field of previous materials that are less environmentally friendly, will be more easily decomposed, able to reduce waste, and lower the composite products prices.

The research aims to examine the composites characteristics using petung bamboo fibers as reinforcement for composite with polyester matrix. In addition, the effect of NaOH soaking on Petung Bamboo Fiber (Dendrocalamus Asper) on the tensile strength of the polyester matrix will also be studied.

2. Research Methodology

2.1. Raw Material Preparation
Petung bamboo (Dendrocalamus Asper) was collected from bamboo forests in Malang, East Java Province, Indonesia as shown in Figure 1. Bamboo was first cut to a size of 30 cm before being hammer to separate bamboo fibers with its binder so that bamboo fibers were obtained. The cutting bamboo was dried in a drying cabinet to get 3-5% moisture content. Meanwhile the polyester resin used was supplied from YUKALAC® 157 BQTN-EX polyester resin.

Figure 1. Petung bamboo, from Malang, East Java, Indonesia

In this study, the fiber making process was performed manually. Hammers, scissors, and cutters were used in separating fibers from bamboo sticks. Fiber obtained on average was obtained with a diameter of 0.7 mm. Fiber separation is indeed quite easy, but to obtain good fiber, it requires sufficient precision so that it is separated one by one.

2.2. Alkalization
Alkalization using NaOH solution was performed by soaking the fiber in NaoH solution of 5% Volume. The Alkali that used was NaOH from Sigma Aldrich with a purity of 98%. Dilution was done by mixing NaOH with destilated water until the concentration drops to 5%. In this study, the alkalization process was carried out at room temperature with variations in soaking for 1 hour, 2 hours, 3 hours. After the soaking process, the fiber was immediately cleaned by rinsing it with water then continued with running water to ensure that the Alkalization reaction process was completely stopped. After the cleaning process with water, the fiber was then dried using laboratory tissue and then put in a mixing cabinet to get 3-5% moisture content. After that the fiber was ready to be used as reinforcement in polyester composites with petung bamboo fiber reinforcement

2.3. Composite manufacturing process
Petung bamboo reinforced polyester composites were made using the vacuum infusion method as illustrated in Figure 2. The Vacuum infusion mechanism was to reduce the pressure on the mold chamber so that the resin mixed with catalyst in reservoir was attracted into the mold. This method is to

overcome the voids occurrence on the composite after drying. The composite mold size was based on the composite Tensile test size with ASTM D638-03 [13] standard for sheet-type as shown in Figure 3. The resin-fiber volume fraction applied in this study was 50%.

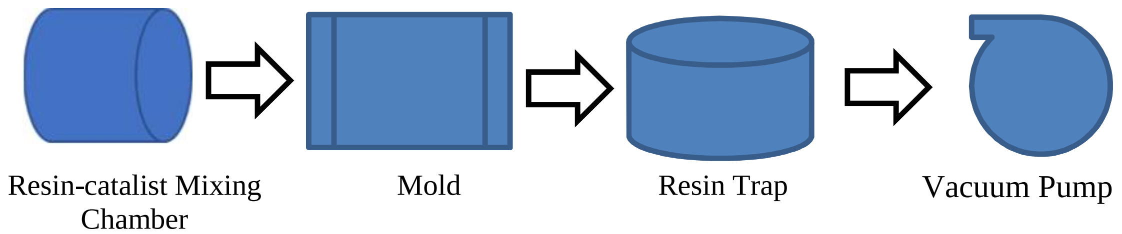

Figure 2. Composite creation mechanism with Vacuum Infusion

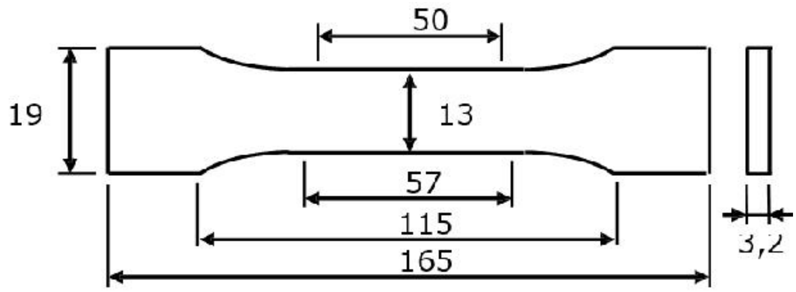

Figure 3. Tensile test specimen size based on ASTM D638-03, the dimension in mm.

2.4. Composite Fracture Observation

In this study to determine the fracture character of the composite, an observation by using Macro photos and SEM was performed. SEM used Phenom XL desktop Scanning Electron Microscope, and the macro photo used digital camera Kiss X7i Canon.

2.5. Tensile Testing

Tensile testing was conducted on Petung bamboo single fibers and Petung bamboo Polyester-Composites to determine the characteristics of their mechanical properties. Composite tensile testing based on ASTM D638-03 testing standards. Single fiber testing used the IMADA tensile tester, whereas the composite testing used Universal Tensile tester machine from SHIMADZU.

3. Results and Discussion

3.1. NaOH soaking effect analysis of petung bamboo single fiber on the single fiber tensile strength

Tensile test results of the Petung Bamboo single fiber with alkalization variation of 1 hour, 2 hours, and 3 hours shown in Figure 4. From Figure 4, it can be seen the single fiber strength respectively from the lowest to the highest was 3 hours NaOH soaking of 98.4 Mpa, 2 hours NaOH soaking of 112.5 Mpa, 1-hour NaOH soaking of 138.7 Mpa; single fiber without NaOH soaking of 191.3 Mpa has the highest tensile strength. The important role of the natural matrix in petung bamboo fiber itself is that it is able to strengthen the bond, so that the highest tensile strength values can be seen in untreated variations because the natural matrix of the polyester is not affected at all by alkali treatment. The natural matrix detached is because the NaOH treatment is expected to be replaced by a resin matrix which will be combined into a composite.

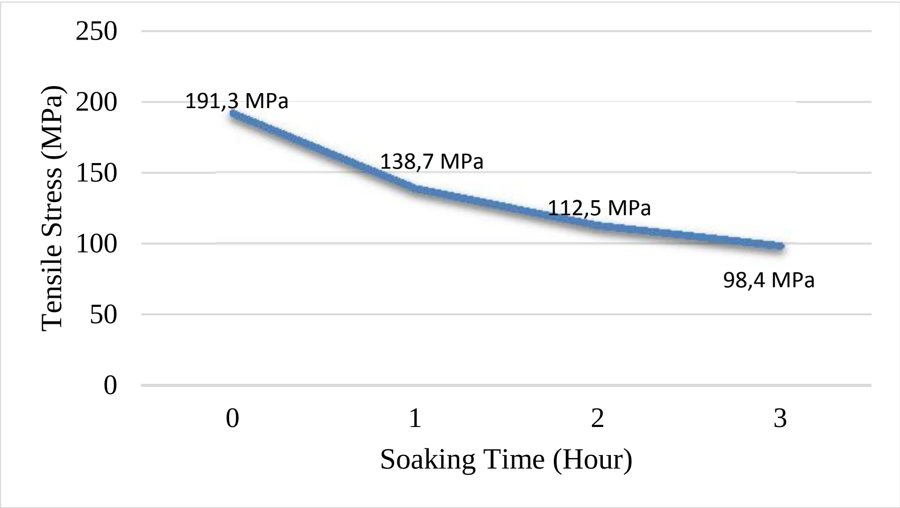

Figure 4. Graph of NaOH soaking effect on petung bamboo single fiber on the single fiber tensile strength

From the figure 4, it can be seen that there is a strength decrease on the fiber with NaOH soaking variations in 1 hour, 2 hours, and 3 hours. There is a considerable assumption that fiber chemical content is released during the soaking process so that the longer the soaking time, the more fiber chemical content will be released. So that in the 3hour soaking variation has the smallest single fiber strength, the fiber or composition functions as an reinforcement is also eroded due to the soaking time duration. Gradually the fiber strength decreases because the microfibril colection of fibers compiler which are joined by lignin will separate, so that the fibers are only in the form of fine fibers which are separated from one another. Because the substance content is lost, causing the fiber damage and lower this petung bamboo fiber tensile strength.

3.2. NaOH soaking effect analysis of petung bamboo fiber with polyester matrix composite toward composite tensile strength

The influence of soaking time of fiber on the tensile strength of the Bamboo petung reinforced polyester composite is shown in Figure 5.

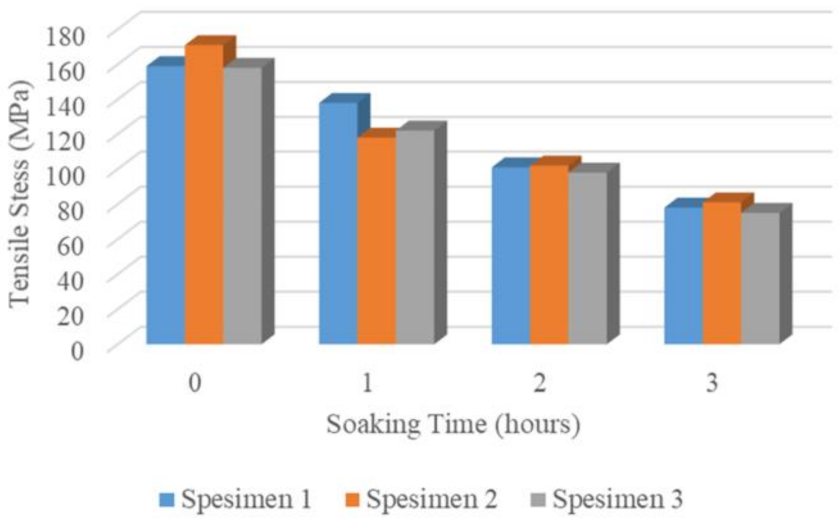

Figure 5. Graph of NaOH soaking effect analysis of petung bamboo fiber with polyester matrix composite toward composite tensile strength

Figure 5 Can be seen in each variation having three specimens that have been processed into composites. Polyester fibers that have been processed into composites if sorted from the highest to the lowest, then the highest tensile strength was polyester fiber without soaking of 171 Mpa, 1 hour soaking polyester fiber of 138 Mpa, 2 hours soaking polyester fiber of 102 Mpa, and the lowest tensile strength was in bamboo fiber with 3 hours soaking of 81 Mpa. With the order of tensile strength as above, it has similarities with the results of single fiber tensile testing that the value of the greatest tensile strength is in fibers without NaOH soaking and the tensile strength of a single fiber will decrease along with the soaking time duration. The chemical composition of fiber is one of the main factors in determining the tensile strength of fibers because in composite without NaOH soaking is blocked by the lignin layers presence on the fiber surface as on the theory basis that lignin is a chemical compound in rigid natural fibers, the release of the bonds between fibers with a matrix called as "fiber pull out" becomes the main cause when tensile tested failed. The lowest tensile strength occurence was at 3 hours soaking of 81 Mpa. There is damage or bond release of chemical compounds that make up natural fibers such as lignin and hemicellulose with the properties of those substances will greatly affect the tensile strength of the fiber when the compound is released from the bond due to long enough soaking time.

The matrix and fiber ability when holding the load and stretching is indeed still visible, but the bond that should have formed between the matrix and the fiber failed, so the composite formed failed early because the fibers contained were degraded. This can be clarified because on the 6% NaOh soaking as discussed earlier, hemicellulose and lignin gradually decreases because of the soaking duration, the natural fibers strength which naturally formed will decrease because microfibils fiber-forming collection is one of the reinforcing factors that are combined by compounds called lignin will separate, so that the existing fibers leave fine fibers that only have a little or tend to run out of the earlier forming compounds content so that they are separated from each other.

3.3. Scanning Electron Microscope Analysis on fracture

The results of SEM analysis on bamboo fiber composite fracture with polyester matrices are shown in Figure 6. The NaOH treatment in fiber causes a partial loss of hemicellulose, lignin, which exists on the fiber surface; the fiber surface becomes coarser, resulting in better mechanical binding with the matrix. With the loss of lignin on the fiber surface, therefore the reaction fields and chemical bonds between fibers and matrices increase which will improve their mechanical properties. But in the hemicellulose content of 6% NaOH alkalization, lignin gradually decreases, so the natural fibers strength will decrease because a collection of microfibril fibers which are joined by lignin will separate, so that the fiber is only in the form of fine fibers separated from one another. The 6% NaOH content is too large so that it erodes most of the lignin along with increasing soaking time. Because the essential content disappear, causing the fibers damage and reduce the tensile strength of this petung polyester fiber. And the magnitude of the stress and strain that can be held by the composite decreases along with the soaking duration.

Fiber swells because the longer the soaking time, the more water molecules absorbed by the fiber. Release of the fiber bonding surface with polyester or called debonding on the fiber which results in mechanical damage or decreased composite mechanical strength, as shown in the SEM photo fracture in the figure. In section of composite fracture without soaking (a). On that fracture, it seems clear that some points have defects or pull out failures, this can reduce tensile strength. In the next magnification stage, a factor that can affect strength is found, namely Debonding, the binding capacity release between fiber and the matrix.

In the test sample with 1 hour NaOH soaking (b) shows the type of failure that is debonding and fiber pull out. But the percentage of failures is dominated by the fiber pull out mechanism rather than the debonding failure. Debonding failure is more due to the weak bond between fiber and matrix. While

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the type of fiber pull out failure is more due to fiber breakage as a result of the fiber being unable to bear the load received. Debonding that occured in 1 hour NaOH soaking is less than petung polyester fiber composite without NaOH soaking.

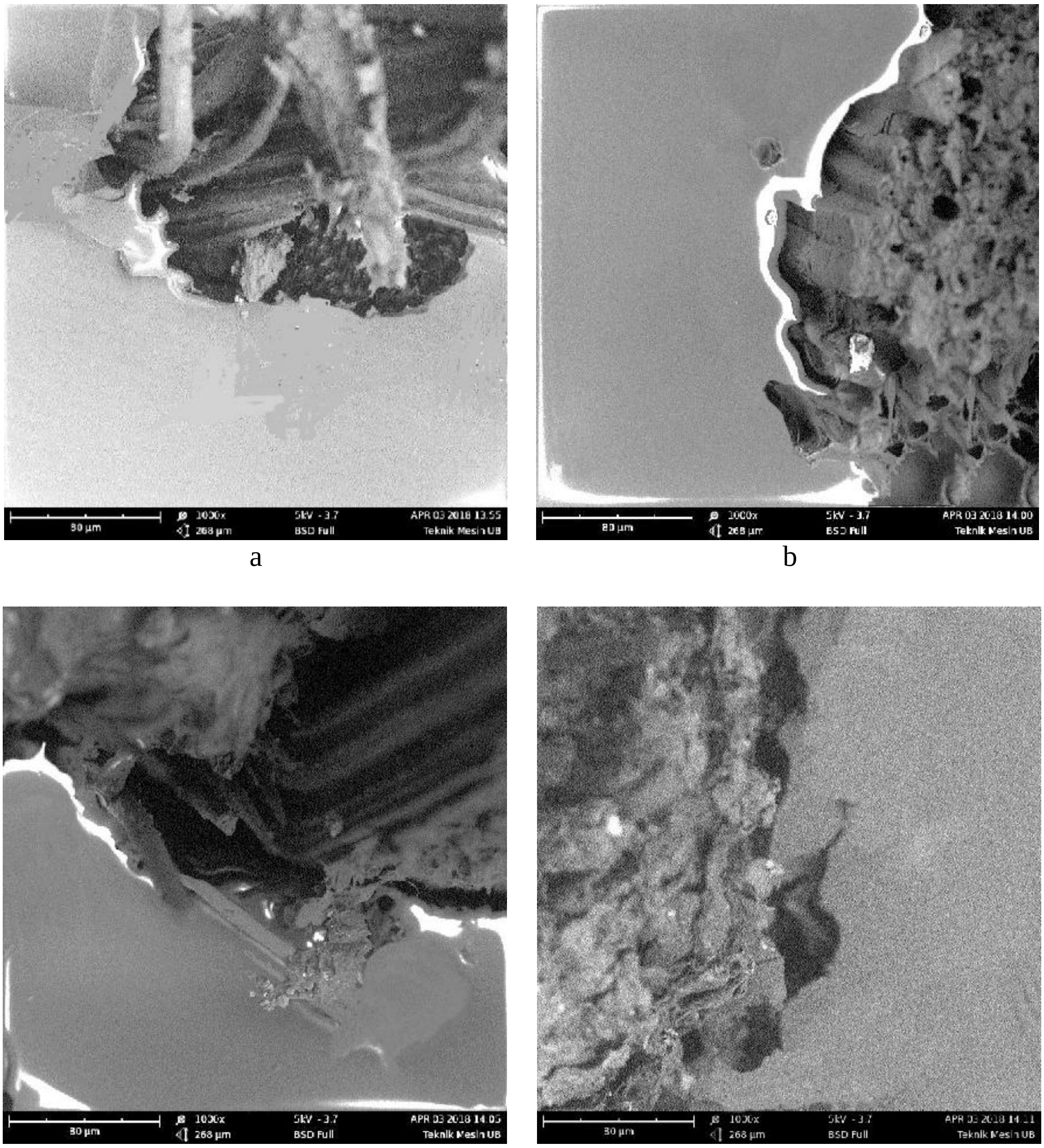

Figure 6. SEM photo results of petung bamboo fiber composite fractures with polyester, magnification of 500x and 1000x (a) Without NaOH Soaking (b) 1 hour NaOH Soaking (c) 2 hours NaOH Soaking (d) 3 hours NaOH Soaking

In the test sample with 2 hours soaking NaOH (c) showed the type of failure in the form of many visible fibers began to brittle more than without NaOH soaking, and 1 hour NaOH soaking this fragility greatly affected the tensile strength of the composite because if it is spreaded throughout fiber certainly will reduce the strength drastically. The reason is the fiber strength is lower than the strength of the fiber-matrix bond so that the fiber is fractured first. Figure (d) is a 3-hour composite fracture section which has the lowest tensile strength value. Because fiber looks very brittle and fragility in the fibers is evenly distributed when subjected to 3-hour soaking. So the breaking of the bond between fiber and matrix is caused by the fiber fragility. With the fiber fragility, it causes lignin and hemicellulose to almost disappear or only left a little.

3.4. Macro Photographs Analysis of composite fracture on petung bamboo fiber with polyester matrix
Figure 7 shown fracture the fracture evidence of the bamboo petung renforce polyester composite.

a b

c d

Figure 7. Macro Photo results of a composite fracture of petung bamboo fiber with polyeser matrix (a) Without NaOH Soaking (b) 1 hour NaOH Soaking (c) 2 hours NaOH Soaking (d) 3 hours NaOH Soaking

From figure 7 (b) it can be seen that composite products with fiber with 1 hour NaOH Soaking show combination failure type between debonding and fiber pull out. Fiber Pull Out that occurs in bamboo fiber composites with 1 hour soaking dominates more than the debonding that occurs. Debonding failure is more due to the weak bond between fiber and matrix. While the type of fiber pull out failure is more due to fiber breakage as a result of the fiber being unable to bear the load received. The debonding average in composite petung polyester fiber specimens with 1 hour NaOH soaking was 4.88%. And the tensile strength decreases to 138 MPa due to fiber alkalization of 1 hour NaOH fiber surface conditions that directly make the fiber begin to degrade the content of lignin and hemicellulose in petung polyester fibers which affect the composite tensile strength and bonding to the polyester matrix.

From Figure 7 (c), it can be seen that composite products with fiber with 2 hours NaOH Soaking show fibers were pulled out from the matrix that is more than soaking without NaOH, and 1 hour NaOH soaking and in this composite there was starved resin, it is a defect that occurs due to lack of resin which is given to fibers so that a resin deficiency occurs in the composite. The cause of fiber matrix pull out is the fiber strength is lower than the fiber-matrix bond strength so that the fiber experience fracture earlier, while the lack of resin occurs because the release of resin does not fully bind the fiber due to the load received by the composite material. In this composite product pull out also occurs. The fibers that come out of the composite are fibers that fail to break in the fracture area of the matrix during loading on the material, so that the fracture of the fiber is different. Inability to accept force evenly, resulting in Pull Out. Fracture that occurs is not due to debonding but is dominated by several fibers which begin to brittle and its tensile strength decreases to 102 MPa due to fiber alkalization of 2 hours NaOH surface conditions which directly make the fiber more degraded amorphous content on petung polyester fibers from composite variations without soaking and 1 hour NaOH soaking.

It can be seen that the composite product with 3 hours NaOH Soaking matrix experienced a material failure which affected its strength which is caused by brittleness and becoming slippery on the fiber surface when 3 hours NaOH soaking produced a 3 hour composite fracture that had Lowest tensile strength. With 3 hours soaking, more fibers are released from the matrix bond due to their inability to bond with each other compared to 2 hours soaking, 1 hour and without soaking. With the lowest composite tensile strength of 81 Mpa. The large percentage of NaOH which greatly influences the composition of fibers, especially in 3-hour soaking, is the longest alkalization of petung polyester fibers so that it greatly influences the condition of the fiber surface which directly makes the fiber degrade and the chemical content damage.

This happens because the alkali will erode the surface of this petung polyester fiber. So that the composite fracture is not caused by debonding but is caused by the fragility of the fiber when given NaOH soaking treatment. However, there is also Pull Out which decreases the strength of the composite.

4. Conclusions

From the results of the research conducted, we can conclude a number of things as follows.

1. The results of the tensile strength testing of petung bamboo single fiber without NaOH soaking were 191.3 Mpa, petung bamboo fiber with 1 hour NaOH soaking of 138.7 Mpa, petung bamboo fiber with 2-hour NaOH soaking of 112.5 Mpa, and the the lowest in 3-hour soaking bamboo fiber of 98.4 Mpa. This happens during the soaking process that the fiber chemical content is released; so that the longer the soaking time, the more fiber chemicals are released so that the variation of 3 hours soaking has the smallest single fiber strength, fiber or composition that functions as an reinforcement which is eroded due to the soaking time and the matrix of the fiber itself has not yet reached its maximum condition to fill the role as a natural binder.
2. It is obtained that the highest tensile strength of composite products with the vacuum infusion method was found in the petung bamboo fiber composite without NaOH soaking of 171 Mpa, petung bamboo fiber composite with 1 hour soaking of 138 MPa, composite bamboo petung fiber with 2 hours NaOH soaking of 102 MPa, the petung bamboo fiber composite with 3 hours NaOH soaking of 81 MPa.
3. It is obtained that the value of composite products debonding with the biggest vacuum infusion method is found in the petung bamboo fiber composite without NaOH soaking of 4.75%, the petung bamboo fiber composite with 1 hour NaOH soaking of 4.88%, bamboo petung fiber composite with 2 hours NaOH soaking there is no debonding, because the fiber is fracture so what happens to the fiber is only pull out caused by brittle fibers. Followed by 3-hour NaOH soaking which has more brittle fibers.

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