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EFFECT OF NaOH SOAKING TIME VARIATION ON THE TENSILE STRENGTH OF HEMP FIBER COMPOSITE USING POLYURETHANE MATRIX

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Abstract

NaOH soaking treatment on fiber is very important in making composites. NaOH soaking can remove lignin and hemicellulose on the fiber surface so that the fiber is cleaner and can provide strong fiber and resin adhesion. The aim of this research is to find out how the best time for soaking in NaOH for hemp fiber compares to the tensile strength of hemp fiber composites. The materials used are polyurethane and hemp fiber. The research focused on variations without treatment, 2 hours, 3 hours, 4 hours, and 5 hours with a percentage of 5% NaOH and dried under direct sunlight after soaking. Composite manufacture was carried out using the compression molding method for 24 hours with a pressure of 5 bar. Tensile testing was also used in this research with reference to the ASTM D 638 standard. The fiber surface experiences changes in shape such as cleanliness, color and fiber structure which are different in each variation. It should be noted that the maximum tensile strength is 11,597 MPa at 4 hours of soaking, and the lowest tensile strength is 6,054 MPa at 5 hours of soaking. Then the lowest modulus of elasticity value is found in the variation without treatment at 0,049 GPa. The highest value of elastic modulus is found in the 4 hour variation with a value of 0,076 GPa.s

Key words: Composite, Polyurethene, Compression Molding, Tensile Strength, NaOH, Hemp Fiber

INTRODUCTION

Nowadays, the development of materials is progressing rapidly. This is due to the need for materials that are capable of withstanding both cold and hot temperatures. Friction resistant and tensile strength can be consideration of selecting materials. Otherwise, each material has different specification depends on utilize and need of material itself. The development of technology in Indonesia have plentiful using advanced material and good for safety and durability. However, the advantages of itself don't covered posibility with high prices. There are lot of method to increase durability and efficiency value of material using a substance named composite (Silvia et al., 2022).

The development science and technology about composite is main factor of innovation idea that for developing good material like strength properties, low density, flexibility, corrosion durability, eco-friendly, easily decomposing naturally, and affrodable prices. Natural fiber composite with plenty advantage that can be factor of application in many industry, like automotive (body, interior, etc), naval architecture, transportation, and biomedical (Rahman & Sosiati, 2020). One of the reference for utilizing a product that have some characteristic with requirement in industry this day is innovation of material substance composite.

Composite was refered to verb " to compose" that means set up and unite. To easily understanding composte itself is gathering of two or more substance that have different characteristic. Composite literally consist of two substance component like fiber as filler material and bonding fiber as matrix (Azhari et al., 2020). Composite is process of mixing substance that have classified refer to combination of each their properties for gain new material with better properties than material who without treatment and bonding surface between eachother (Azhari et al., 2020). Composite have matrix as countinous phase that can give the distribution against uniform load that the inside of it have reinforcing material or reinforcement. Composite is a material in which there are two or more components that have different properties. The process of combining two materials in this composite usually has different mechanical properties in each constituent material(Rahman & Sosiati, 2020).

Composite who compared to metal have advantages refer to characteristic like, designed with high process, so that give more specific stiffnes than metal (Hazhari et al., 2022). Composite have better noise, vibration, and sound resistant. There are lot of classification composite, type for this research is composite with fiber as filler inside. Natural fiber is fiber that gained from natural enviromental like plants, animals, and minerals. Fiber that from plants like sisal leaves, pineapple leaves, hemp leaves, residual sugarcane, etc. Fiber that from animals like fleece, skin, and silk.

Composites have advantages more efficient than other materials such as wood. The advantages of composites in good mechanical and physical properties make composite materials resistant to corrosion, weather resistant, and have a relatively low density. In addition, this composite material has advantages in terms of versatility (Wiranegara et al., 2022). The composition in the composite manufacturing process requires several components in it. Each of these constituent components has its own function and use in order to make composite products that have a variety of advantages. Some constituent components such as: reinforcing components (fibers and other particles), binding components (fiber binding matrix) this component serves to protect from external damage and forward the load that is forwarded to the fibers inside, then namely additional components (chemical substances, additives) (Sunardi et al., 2014).

Fiber composite is the process of combining between the adhesive matrix and fiber as reinforcement. In general, fiber usually used have bigger characteristic than adhesive matrix itself. On process making composite that use natural fiber that need to treatment like alkalization for natural fiber that would use for cleaning from oil dirt and other substance in order to mixing between adhesive matrix and natural fiber that can unite perfectly (Maryanti, Sonief, & Wahyudi, 2011).

The research that Fajar Paundra did with his friends in 2022 concluded that to find out the strength of composite material such as types of fiber, alkaline treatment, volume fraction, matrix variation, variation of size fiber, and combined of two types fiber (hybrid) (F Paundra et al., 2022). Adhesive material that usually used have high heat resistance, can fill up inner thecomposite. The matrix in the composition such as epoxy, polyurethane, catalist resin, etc. with some consideration of problems and needs from automotive industrial that existed, the show up the innovation with utilize of natural fiber that modified in making process in order to fulfill the needs with less cost. So with all the consideration, that have made the research with the topic was Effect of Variation of Soaking Time Fiber Hemp Composite Using NaOH Matrix Polyurethane On Tensile Strength (Sidik et al., 2024). Therefore, with this research, it is expected that hemp fiber can be useful in the manufacturing industry and household life, and can be used to make various useful materials. To improve the function of hemp fiber which is commonly used for textiles and folk crafts into engineering materials, it is necessary to research and develop it as a composite material that matches its physical and mechanical properties, so that a new composite material will be created.

METHODS

This research uses hemp fiber (*Agave Sisalana*) as reinforcement and polyurethane resin as the matrix. The hemp fiber is cut to a length of 5 cm with an average diameter of 0.10 mm. Subsequently, an alkali treatment process using NaOH is carried out. The variations in soaking time in the NaOH solution are: no treatment, 2 hours, 3 hours, 4 hours, and 5 hours. This is done to remove oil and dirt from the fiber. Before soaking in the NaOH solution, the fibers are first washed with water and dried. The composite material composition consists of 80% matrix and 20% hemp fiber.

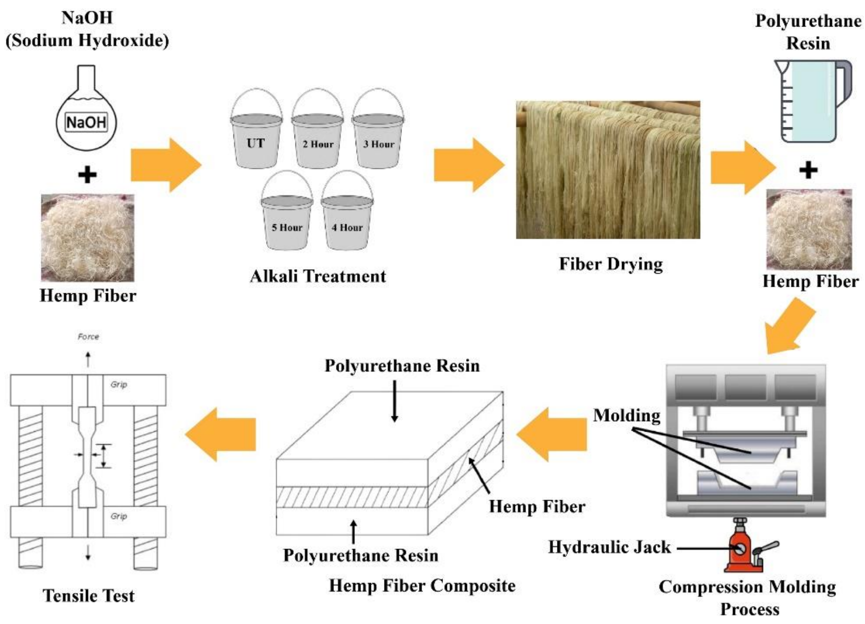


Figure 1. The Process Composite Manufacturing

Figure 1 is the process of making composites using the compression molding method. This process uses polyurethane as matrix and filler with hemp fiber which both united in the mold with help from matrix polyurethane. The compression molding method is a material manufacturing technique that involves placing composite materials in an open mold, which can and applied high pressure to form the final product. This method is suitable for sisal fiber and polyurethane resin based on composites. The mold used was 18 x 18 x 0.3 cm in size. The pressure from compression molding is used five bars. After the composite manufacturing process, fiber surface testing and tensile testing were conducted.

Tensile testing is a test that is used to find out the information about tensile strength of a material. The test is carried out on the specimens are subjected to tensile forcing on both face of specimen with periodic tensile strength (Margono et al., 2020). The information about is value of maximum tensile strength and elongation display in the computer beside of the tensile testing machine. In this testing, tensile testing refers to ASTM D 638 that shown in figure 2 (F Paundra et al., 2024).

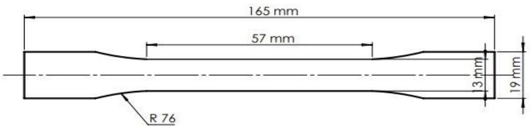


Figure 2 Tensile Test on ASTM D 638 standard

This machine have two variables that can be conducted. There are load control machine and movement control machine. In this machine, there equations that using for this research shown below.

$$\sigma t = \frac{P}{A} \qquad (1)$$

Description:
σt= tensile Strength (MPa)
P = Force (N)
A = Cross Sectional Area (mm^2)

Tensile strength generate some data such as modulus elasticity, stress, and strain. This research focused on stress and strain that can discussed in result. For equation the strain, following below.

$$\varepsilon = \frac{l_i - l_0}{l_0} \qquad (2)$$

Description:
ε = Strain (%)
l_i = following length (mm)
l_o = previous length (mm)

RESULTS AND DISCUSSION

This research is conducted to find out the result from the testing of tensile strength on composite with variation of soaking time with NaOH of fiber hemp fiber. The result is following at figure 3.

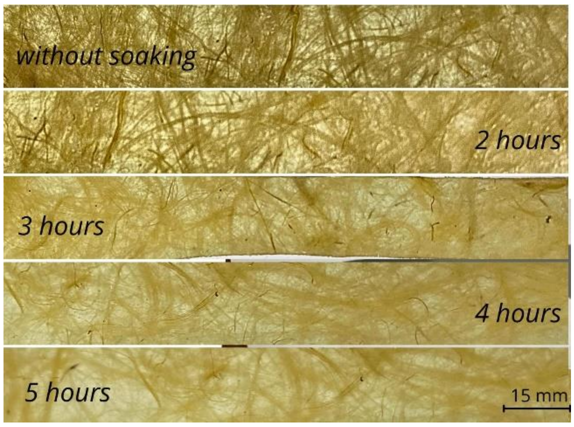


Figure 3. The result of manufacturing co mposite

Figure 3 shows the results of the composite manufacturing process using hemp fibers with polyurethane matrices with varying lengths of soaking time: without treatment, 2 hours, 3 hours, 4 hours, and 5 hours. From Figure 3, it can be seen that each soaking variation has a difference in the physical shape of the fibers in it. In the variation without soaking, it can be seen that the hemp fiber inside is brownish black because there is still a lot of dirt attached to the surface of the fiber. The 2 hours variation of soaking the specimen looks a little cleaner than the previous variation because it has gone through soaking in NaOH solution. The 3 hours variation looks clean and the fiber structure has begun to appear, it's just that there is still a little dirt attached (Purkuncoro, 2017).

The 4 hours variation looks very clean and there is no visible dirt attached to the fiber. In the 5 hours variation, the fibers look clean but the image of the fibers has begun to pale and there is brittleness at some points of the hemp fiber. By looking without tools alone the difference in specimen results is clear. With different variations of testing in each specimen, the results of analysis and observation are also different in the five specimen variations. Observations were made to see the condition of the fiber both from the physical form, the condition of the dirt attached to the hemp fiber and the fiber diameter and fiber fragility after soaking with NaOH. The longer the soaking of the fiber with NaOH solution, the cleaner it will look from dirt and the smaller the diameter of the fiber because the dirt attached has been cleaned (Purkuncoro, 2017).

Micro Structure on Hemp Fibers Face

The tests on the surface of hemp fibers were carried out with the aim of seeing the results of the influence of the NaOH solution soaking process on the surface of hemp fibers. Then the observation results can help in analyzing the final results of tensile strength on hemp fibers and resin. This test was carried out using a streo microscope and was carried out sequentially according to the variations in this study. This test was carried out after all variations were completed in the soaking process in NaOH solution. Soaking is very necessary if the research is related to natural fibers because through this soaking process it can help clean dirt which can later increase the attachment between hemp fiber and polyurethane resin. Soaking variations are carried out with no soaking, 2 hours, 3 hours, 4 hours, 5 hours. Figure 4 shows the results of testing the fiber surface with variations without soaking (Nesimnasi, Boimau, & ..., 2015).

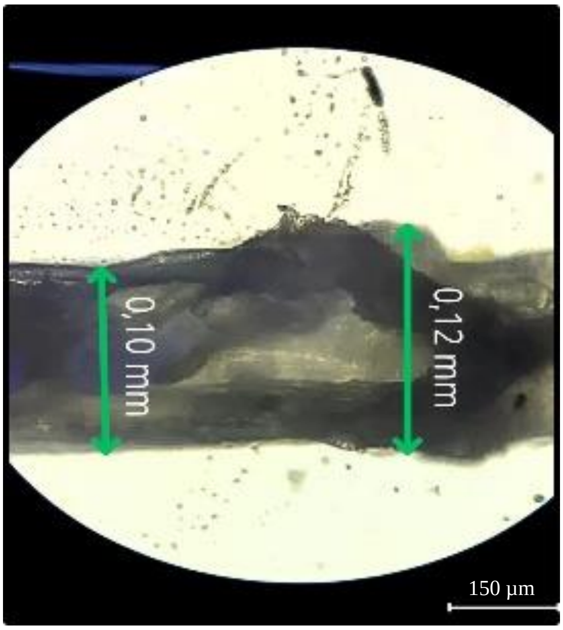


Figure 4. Photo of microstructure hemp fiber non treatment

Figure 4 is a hemp fiber that did not through the NaOH solution soaking process or without soaking. Hemp fiber that did not through the NaOH solution soaking process can be seen that the surface of the hemp fiber still has a lot of black clumps or dirt attached. The hemp fiber dirt when seen with the eye cannot be seen dirt attached to the surface of the fiber. The diameter of the fiber which should be 0,10 mm increases to 0,12 mm because there is a lot of dirt attached to each surface of the hemp fiber.

Such problems often occur if the fiber did not through the NaOH solution soaking process, therefore this soaking process is an important process in this research (Diharjo, 2006). Dirt on hemp fibers can cause fibers to not be able to provide maximum fiber strength because there is a barrier between the fiber and the resin used. Soaking is very necessary if the research is related to natural fibers because through this soaking process it can help clean dirt which can later increase the attachment between hemp fiber and resin. Figure 5 is the first comparison of all existing variations with a variation of NaOH soaking time for 2 hours (Purkuncoro, 2017).

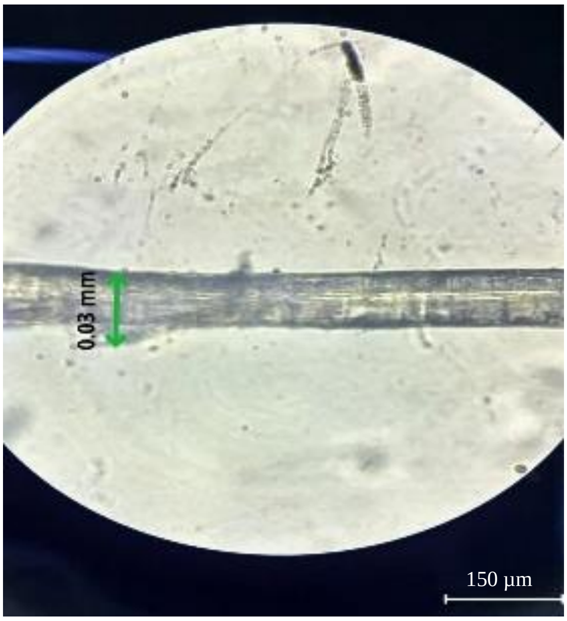


Figure 5. Photo of microstructure hemp fiber with 2 hours soaking

Figure 5 shows the results of testing the surface of hemp fibers that have been soaked for 2 hours. A clear difference can be seen on the surface of the fiber which is starting to clean but not perfect. In this 2 hour soaking there is still some dirt attached to the surface of the hemp fiber. The fiber diameter has decreased because the dirt and wax on the fiber surface have disappeared. Without soaking, the initial diameter of the fiber was 0,12 mm and decreased significantly to 0.03 mm after soaking for 2 hours. Soaking is very necessary if the research is related to natural fibers because through this soaking process it can help clean dirt which can later increase the attachment between hemp fiber and resin.

With fewer impurities, the hemp fibers show the possibility of increased attachment to the polyurethane resin, although it has not yet reached optimal conditions. Figure 6 shows the results of testing the surface of hemp fibers that have been soaked with a length of time of 3 hours (Diharjo, 2006).

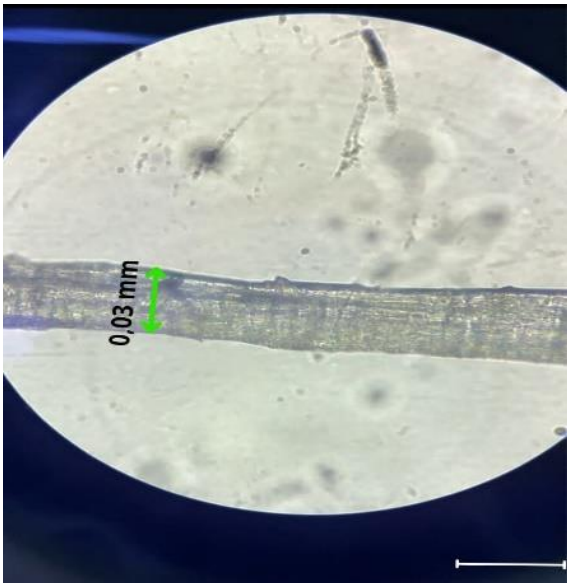


Figure 6. Photo of microstructure hemp fiber with 3 hours soaking

Figure 6 is a hemp fiber that has been soaked with NaOH solution for 3 hours. The hemp fiber looks cleaner and the fiber structure has begun to clear. The difference that occurs is not too significant some points there is still dirt attached to the surface of the hemp fiber. The fiber diameter in the 3 hours NaOH soaking variation is 0,03 mm. It can be seen that each difference in the length of NaOH soaking also has a significant difference on the fiber surface. The longer the soaking, the cleaner the fiber surface from various impurities and waxes. Figure 7 shows the results of testing the surface of hemp fibers with variations in the length of NaOH soaking for 4 hours.

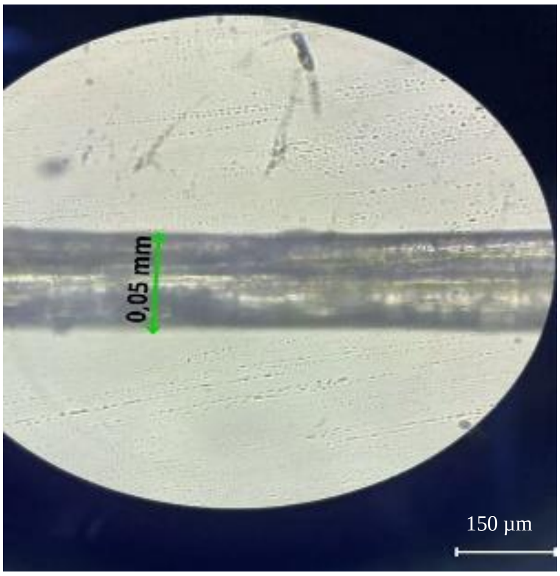


Figure 7. Photo of microstructure hemp fiber with 4 hours soaking

Figure 7 shows the results of surface testing with variations in NaOH soaking for 4 hours. Hemp fibers look very clean and the microfibrial structure is clearly visible fiber diameter in the variation of NaOH soaking for 4 hours of 0.05 mm. it can be seen from the variation of NaOH soaking for 4 hours is proven effective in cleaning the fiber from dirt and can produce maximum microfibrillar structure microfibrillar structure formed optimally increases the ability of the fiber to withstand loads and prevent deformation, making the composite more durable and efficient in various applications. Figure 8 shows the results of testing the fiber surface with variations in the length of

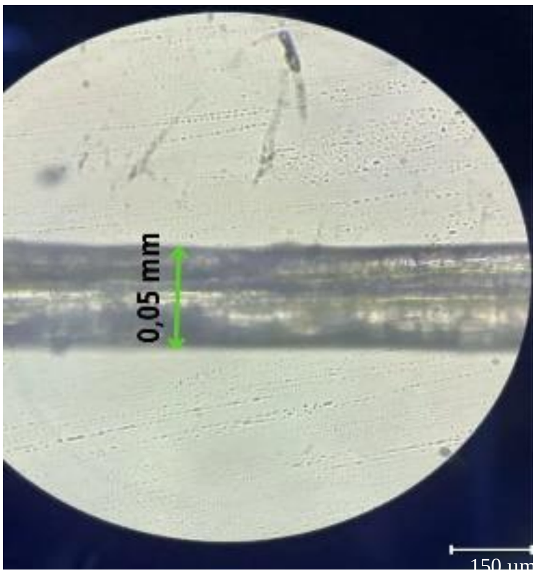


Figure 8. Photo of microstructure hemp fiber with 5 hours soaking

Figure 8 shows the results of testing the fiber surface with a variation in the length of NaOH soaking for 5 hours. The hemp fibers in this variation look very clean but the fiber structure shows the signs of brittleness. The effect of too long soaking in the NaOH solution is slightly cropped and split with other structures. In this variation, the diameter concentration of hemp fiber after NaOH soaking for 5 hours is 0,05 mm. This shows that too long soaking on NaOH can damage the shape of the fiber, causing brittleness even though the microfibrillar structure is still formed. Therefore, although 5 hours of NaOH soaking is effective in cleaning hemp fibers from various impurities, the negative side effects on tensile strength and brittleness indicate the need for balance in the NaOH solution soaking process. This confirms that the cleaning process of natural fibers must be adjusted to the characteristics of the fiber so that brittleness and even damage to natural fibers does not occur (Millenio et al., 2022).

Overall, from the discussion of hemp fiber surface testing, it can be emphasized that the hemp fiber soaking process is a urgent thing to do before making composites made from natural fibers such as hemp fiber. In testing the surface of hemp fiber, it was found that the soaking time of 4 hours was the best and did not damage the hemp fiber. From the results of testing the surface of hemp fibers, it is also confirmed that the length of time of NaOH soaking in hemp fibers greatly affects the final result of fiber conditions. It shows that cleaning and rearranging the microfibrils in the fiber can improve the ability of the fiber to bond with the resin. This is very important to obtain composites with good mechanical properties sufficienly (Millenio et al., 2022).

Tensile Testing

Tensile testing in this research was carried out using the Universal Testing Machine Zwick Roell All Round Z250R in the Mechanical Engineering material engineering laboratory of the Sumatra Institute of Technology. Tensile testing was carried out in order to determine the value of the tensile strength of polyurethane-matrixed hemp fiber composites with varying lengths of NaOH soaking time. Tensile testing specimens are made referring to the ASTM D 638 standard. Data that has been obtained through calculations with the formula σ = F / A, the results obtained in table 1.

Table 1 The results of tensile testing

No.	Soaking time (Hours)	Tensile Strength (MPa)	Yield Strength (MPa)	Elastic Modu-lus (GPa)
1	Non Soaking	7,607	7,0	0,049
2	2	7,880	7,1	0,051
3	3	8,179	8,0	0,052
4	4	11,597	11,4	0,076
5	5	6,054	5,4	0,048

Composite specimens. In this research, tensile testing was carried out in five variations and each variation produced different values of maximum tensile strength, yield strength and elasticity modulus. The factor that causes the difference in numbers is due to several factors, one of which is the length of time of the NaOH solution soaking process. The graph of the results of the comparison of the length of soaking with the maximum tensile strength can be seen in Figure 9.

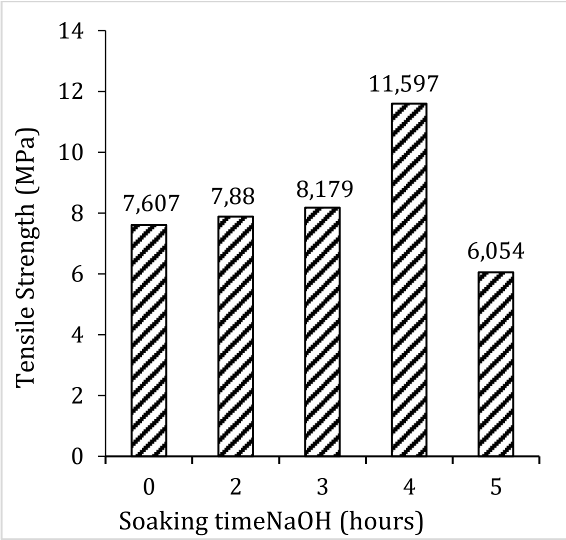


Figure 9. Graph of Tensile Strength

Figure 9 is a graph showing the comparison of the results of each test variation. The graph shows the difference in maximum tensile strength each variation. In the variation without soaking, the maximum tensile strength value is relatively small with a result of 7,607 MPa because the hemp fiber did not through the NaOH solution soaking process. Then the tensile strength value tends to increase but not huge for the increase in the NaOH soaking variation for 2 hours is 7,880 MPa. The increasing of maximum tensile strength is due to the loss of dirt attached to the hemp fiber which then has an effect on the strength of the attachment between the fiber and the polyurethane resin when had tested tensile sufficienly (Millenio et al., 2022).

The increase in maximum strength value also occurs in the variation of soaking in NaOH solution for 3 hours with a result of 8,179 MPa. The highest value of maximum tensile strength against the length of time soaking in NaOH solution for 4 hours with a maximum tensile strength value of 11,597 MPa. In the variation of soaking in NaOH solution for 5 hours there was a decrease of 6,054 MPa due to the longer soaking of fibers in NaOH solution in addition to giving a clean effect also gives the effect of fragility to the hemp fiber structure and the tensile strength also produced in this 5 hours variation tends to decrease (Millenio et al., 2022).

The soaking treatment of NaOH solution in Figure 9 is very influential on the final value of the maximum tensile strength of polyurethane matric hemp fiber composites. in the variation of soaking times of 2 hours, 3 hours, and 4 hours, there is an increase in the value of tensile strength that occurs due to hemp fibers that begin to clean and are able to provide maximum attachment between the matrix and the fiber because there is no barrier in the form of dirt and wax attached to the hemp fiber. However, it can also be seen that the 5 hours soaking variation experienced a fairly drastic decrease even though the fibers were clean from dirt and wax attached, the fiber structure had begun to break down and become brittle which caused the attachment of the fibers and the matrix to decrease. In addition to analyzing the maximum tensile strength value of hemp fiber composites, this research also has an elastic modulus value towards the soaking time in NaOH solution. Figure 10 shows the effect of soaking time in NaOH solution on the elastic modulus value (Lastri, 2023).

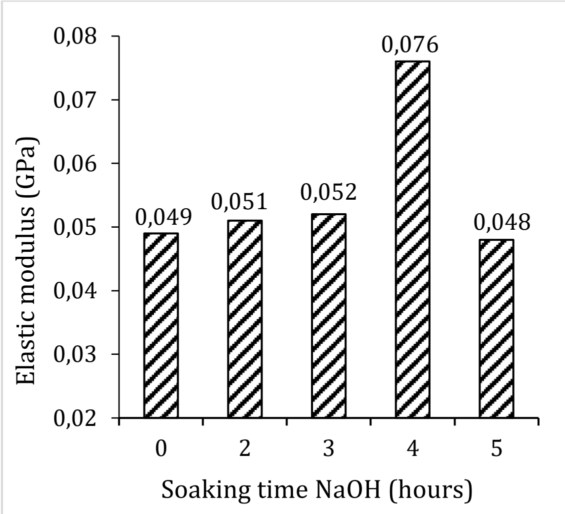


Figure 10. Graph of elastic modulus

Figure 10 illustrates the elastic modulus values in relation to the soaking time of hemp fiber composites in a NaOH solution, calculated using the formula $E = \sigma / \varepsilon$. For the soaking time variations of no treatment, 2 hours, and 3 hours, the elastic modulus showed a steady increase with values of 0.049 GPa, 0.051 GPa, and 0.052 GPa, respectively. The highest value, 0.076 GPa, was observed at the 4-hour soaking time. However, the lowest elastic modulus value, 0.048 GPa, was recorded at the 5-hour soaking time.

The fluctuation in the elastic modulus of the hemp fiber composite is directly related to the soaking duration in the NaOH solution. In composites without alkali treatment, the elastic modulus was lower due to the unclean fiber surface, which hindered proper adhesion of the resin. The increase in elastic modulus after soaking demonstrated that the alkali treatment effectively cleaned the fiber surface, enhancing the adhesion of the resin to the fiber. However, the elastic modulus decreased after 5 hours of soaking, likely because prolonged exposure to the hot alkali solution damaged the fiber surface. While NaOH treatment helps clean dirt and wax from the fibers, excessive exposure can lead to fiber brittleness and damage. Table 2 presents macro photos of fracture surfaces after tensile testing of hemp fiber composites, following the ASTM D 638 standard (Nugraha et al., 2020).

Table 2 Images of micro fracture on tensile testing for composite hemp fiber

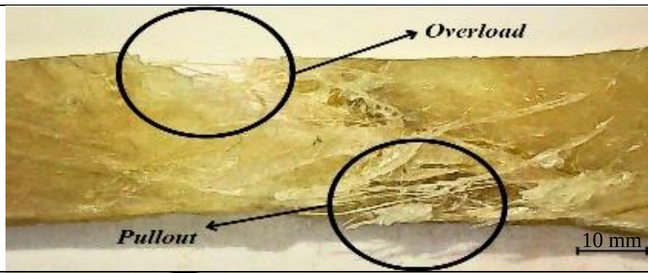
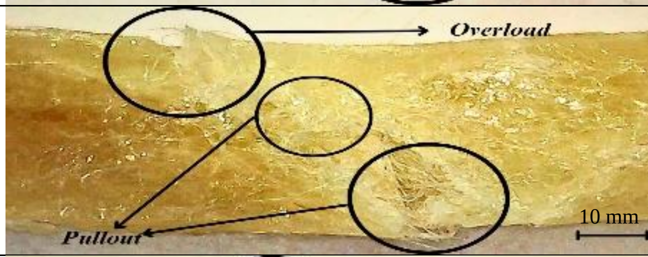
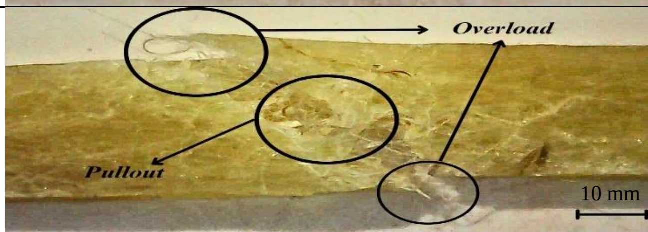
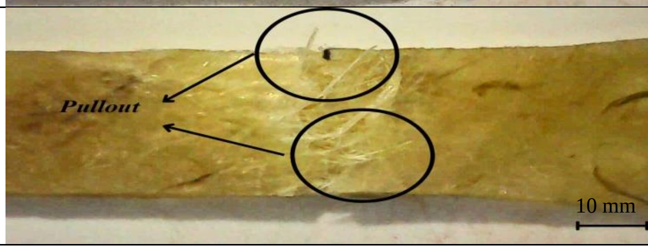
Soaking Time	Front View
Without Treatment	
2 Hours	
3 Hours	
4 Hours	
5 Hours	

Table 2 is the result of fracture analysis after tensile testing on polyurethane matrix hemp fiber composite specimens. The plastic deformation is large enough to indicate that the specimen is ductile in the form of fibers and coarse. Pullout and overload fractures dominate the type of fracture in polyurethane matrix hemp fiber composite specimens (Diharjo, 2006). From the five test variations have different shapes and location of the fault. Fracture with pullout type is a fracture that the combined between fiber and matrix is not strong so that the fiber comes out of the matrix connection. While overload is a fracture that can occur when the fiber breaks due to the attachment of fiber and matrix that is so strong (Lastri, 2023).

CONCLUSIONS

Based on the result of the discussion, the following conclusions are drawn:

1. The results of the tensile strength value in each variation have increased as in the variation without treatment in NaOH solution of 7,607 MPa, a significant increase in the 2 hour variation of: 7,880 MPa, variation of 3 hours soaking is: 8,179 MPa,

the highest variation in soaking for 4 hours with a value of: 11,597 MPa, and a decrease in the maximum tensile strength value in the 5 hour variation with a tensile strength value of: 6.054 MPa.

2. The value of elastic modulus with the maximum tensile strength value is directly proportional which increases in each variation of soaking time such as in the variation without soaking with an elastic modulus value of: 0.049 GPa, increased in the 2-hour variation with a value of: 0.051 GPa, in the 3-hour soaking variation there was also an increase with a value of: 0.052 GPa, the highest elastic modulus value is found in the variation of soaking in NaOH solution for 4 hours with a value of : 0.076 GPa, and the elastic modulus value decreased in the variation of soaking in NaOH solution for 5 hours with a value of: 0.048 GPa.

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