Mekanika: Majalah Ilmiah Mekanika

Analysis of Tensile Strength, Wear Rate, and Crystallinity of Bio-composite Nano-HA/Magnesium/Shellac Reinforced Cantula Fiber for Bone Screw Material

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Abstract

Accidents are a major cause of fractures in Indonesia. One of the treatments for fractures is bone screws with support plates that are placed on broken bone. Currently, many biomaterials for bone screws are being developed which have biodegradable properties so that post-operative bone healing is not required. The purpose of this study was to determine the effect of cantula fiber addition on tensile strength, wear rate, and crystallinity of nano-HA/magnesium/shellac bio-composite for bone screw materials. Nano-HA/magnesium/shellac/cantula fiber materials were mixed using a blender. The material was mixed with a magnesium/hydroxyapatite ratio of 70/30 and cantula fiber was added with variations of 0%, 10%, 20% and 30% of total volume. After that, material mixture was compacted with a pressure of 300 MPa for 10 minutes. Then sintering process was carried out at temperature of 140 °C for two hours. The results showed that the highest tensile strength value was 7.86 MPa at 30% variation. The lowest wear rate was 0.31 x 10^{-3} mm^3/Nm at 30% variation. The highest crystallinity in X-Ray Diffraction observations was obtained at 30% variation, which was 79.65%.

1 Introduction

Bone fracture is a complete or partial break in the bone continuity due to injury. In recent years, magnesium-based biomaterials have been developed to treat fractures. Magnesium was chosen as a biomaterial because it is biodegradable and has mechanical properties similar to human bones [1]. Hydroxyapatite was added to reduce the rapid degradation rate of magnesium [2]. Hydroxyapatite has the same mineral content as human bone. Hydroxyapatite is biocompatible, osteoconductive, and capable of stimulating bone formation [3]. Shellac is added to bind hydroxyapatite with other materials. Shellac is a natural polymer which has biodegradable, non-toxic and renewable properties [4]. To improve the mechanical properties of material, cantula fiber was added to Mg/HA/shellac mixture. Cantula fiber is natural fiber that has biodegradable properties. Cantula fiber has low density but high strength [5].

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Current bio-composite materials often face challenges such as insufficient mechanical strength, poor integration with surrounding tissues, or inadequate degradation profiles. There is a need for systematic studies to understand the effects of various factors, such as composite composition, fiber orientation, and manufacture processes, on the performance of bio-composite bone screws. Additionally, the development of standardized testing protocols and evaluation methods specific to bio-composite bone screws is essential for accurate comparison of different materials and formulations. In this study, magnesium/HA/shellac biomaterial was added with cantula fiber for the manufacturing of bone screws. The results of this study are expected to be an alternative material for bone screws and beneficial for patients with bone fractures.

2 Experimental Methods

2.1 Materials

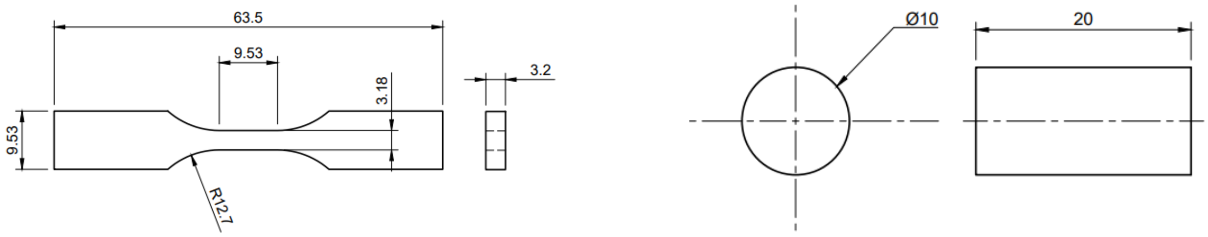
The materials used in this study were magnesium, nano-hydroxyapatite, shellac solution, and cantula fiber. Magnesium is used as matrix. Shellac-coated nano hydroxyapatite is used as a filler to reduce the porosity of material. Cantula fiber is used as reinforcement to increase the strength of material.

2.2 Methods

The preparation of shellac solution began with crushing the secretions of shellac lice using mortar, then pulverizing using a blender to make it into powder. After that, the lacquer secretion powders were mixed with 96% ethanol using a magnetic stirrer. Mixing was carried out for four hours with the ratio of lacquer secretion powders to ethanol was 1:10. Then the shellac solution is precipitated for a while and the filtering process is carried out. The result of this filtering is a shellac solution.

The cantula fiber used in this study had previously been pretreated by soaking it in 2% NaOH solution for six hours. After that, the cantula fibers were washed with clean water and left to dry for three days. Then the cantula fiber was dried in an oven at 110 °C for 45 minutes. After drying, the cantula fiber was cut into a length of 10 mm and then crushed four times using a crusher machine. Cantula fiber was sieved with a mesh of 60 to obtain cantula powder with an average aspect ratio of 21.11. The preparation of the Mg/HA/shellac mixture and cantula fiber was initiated by mixing the nano-hydroxyapatite and shellac solution using magnetic stirrer which was carried out for two hours at temperature of 100 °C and speed of 200 rpm. The result of mixture was in the form of dry powder. Then, it was mixed with magnesium powder and cantula powder using blender with three variations of speed for one minute each. The mixed materials were then put into the mold and then pressed using press machine with a pressure of 300 MPa. The formed specimen was then removed from the mold. The compacted specimens were sintered using a furnace at 140 °C and held for two hours. This sintering process was useful to strengthen the bond between the powder grains and reduce the porosity of the specimen. This specimen from sintering process would be tested as bone screw material. This study was carried out by the addition of cantula fiber with 0%, 10%, 20% and 30% variations of fiber volume fraction.

In this study, tensile test was carried out using Universal Testing Machine (UTM) according to American Society for Testing Materials (ASTM) D638 type V with load cell of 50 kg and pulling speed of 10 mm/minute. Wear rate test was carried out using tribometer machine type pin on disc according to ASTM G-99 with loading of 2 kg, frictional speed of 1 m/s, and track length of 501.58 m. Fourier Transform Infra-Red (FTIR) observations were carried out to determine functional groups that appeared in the specimen. To determine crystallinity of material, X-Ray Diffraction (XRD) observations were carried out with diffraction scan angle in the range of 10°-50°.



(a) (b)

Figure 1. Test specimen: (a) Tensile test ASTM D368 type V, and (b) Wear rate ASTM G-99

3 Results and Discussion

3.1 Tensile test

Figure 2 shows that tensile strength on magnesium/n-HA/shellac/fiber specimens cantula increase along with addition cantula fiber content on specimen 0%, 10%, 20%, and 30% respectively is 2.73 MPa; 5.48 MPa; 6.96 MPa; and 7.86 MPa. The result of tensile strength from those variations are on top the tensile strength of human bones, that is 2.54 MPa [6]. However, the tensile strength result from this study is far below the tensile strength of 316L stainless steel that generally used as implant bone, that is 465 MPa [7].

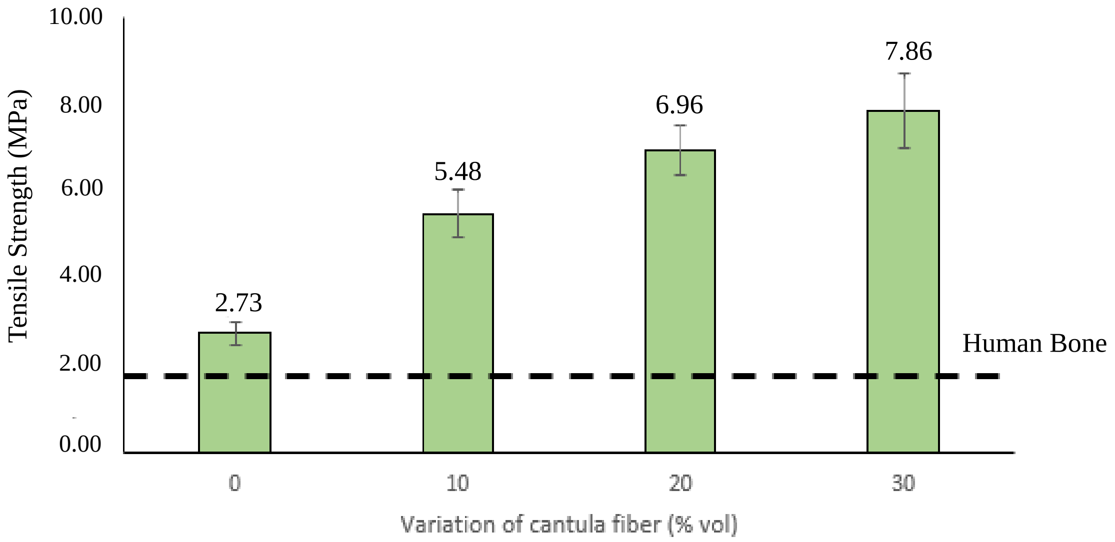


Figure 2. Tensile strength test result

The fibers in the composite mix resist most of the forces acting on the mixture. Role of matrix in the mixture is to protect and bind fibers to work properly. Tensile strength value increases with the addition of cantula fiber. This happens because cantula fiber used has a high aspect ratio, which is 21.11. If the aspect ratio is high, tensile strength will also increase because fibers are able to withstand loads well. The more cantula fibers added, the more load they can withstand [8]. In addition, cantula fiber has been given an alkaline treatment so the fiber surface is cleaner and rougher which allows a strong bond with the matrix. Formation of a strong bond is due to the occurrence of mechanical interlocking between fiber and matrix [9].

3.2 Wear rate test

Figure 3 shows the specific wear rate on magnesium/n-HA/shellac/cantula fiber specimen decreased with the addition of cantula fiber content in specimen sequentially 0%, 10%, 20%, and 30% of 3.38 x 10^{-3} mm^3/Nm; 1.16 x 10^{-3} mm^3/Nm; 0.81 x 10^{-3} mm^3/Nm; and 0.31 x 10^{-3} mm^3/Nm. A study showed that the specific wear rate of HA/shellac/cantula fiber implants was 0.51 x 10^{-3} mm^3/Nm. The 30% variation in this study was below the specific wear rate of HA/shellac/cantula fiber implants, while the other three variations were above this value [10]. The specific wear rate of the four variations in this study is still above the wear rate of human bones, which is 0.082 x 10^{-3} mm^3/Nm [11].

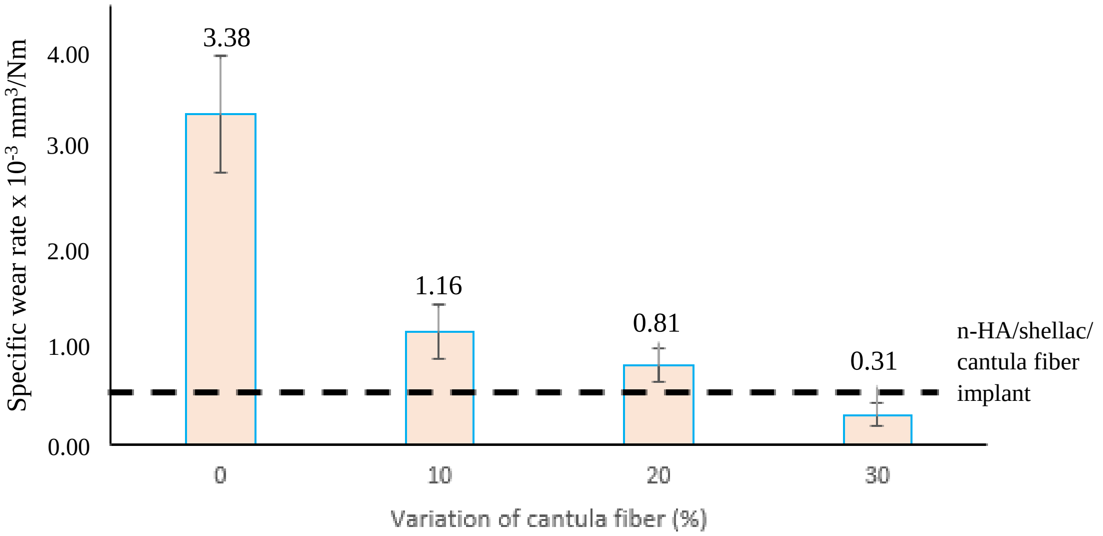


Figure 3. Wear rate test result

Cantula fiber acts as a good reinforcement because its addition has an effect on decreasing specific wear rate of Mg/n-HA/shellac/cantula fiber mixture. This is related to the increase in crystallinity of each variation thereby increasing hardness value of material [12]. The increase in crystallinity indicates the strength of material structure is increasing. In addition, increasing of tensile strength value also affects a decrease in the wear rate of specimen. Cantula fiber has good resistance to wear characteristics of bio-composite materials as known by the reduced wear rate as shown in Figure 3 [13].

3.3 FTIR observations

Based on FTIR results, at wavenumber of 3445 cm^{-1} and 3429 cm^{-1} an OH^- (hydroxyl) peak appeared which was identical to n-HA [14]. In addition, appearance of OH^- functional group at this wavenumber can also indicate the presence of cellulose from cantula fibers. The addition of cantula fiber volume fraction had an effect on increasing intensity of OH^- functional group, as shown in Figure 4. The wavenumber 2932 cm^{-1} (see Table 1) appeared as CH_2 (methylene) functional group which was identical to natural fiber [5]. In addition, that wavenumber was also influenced by shellac. Wavenumbers 1693 cm^{-1} and 1686 cm^{-1} were identified as C=O functional group which is characteristic of carboxylic acid or ester structure derived from shellac [15]. In wavenumbers 1448 cm^{-1} and 1421 cm^{-1}, CO_3^{2-} (carbonate) functional group appears due to the reaction between carbon dioxide and n-HA in free air [14]. At wavenumber 1231 cm^{-1}, C-O functional group appears due to cantula and shellac fiber bonds [15]. In wavenumbers 1035 cm^{-1}, 603 cm^{-1}, and 564 cm^{-1} there is a sharp peak of PO_4^{3-} (phosphate) functional group that appears due to the strong influence of n-HA [14]. Emergence of covalent bonds in the form of C-H, C-O, and C=O resulted in the stronger bonds between atoms. This causes the wear resistance of specimen to increase because material is not easily eroded [16].

Table 1. Functional groups from FTIR observations

No	Functional Groups	Wavenumber (cm^{-1})
1	OH^-	3445, 3429
2	C-H (CH_2)	2932
3	C=O, CO ester, carboxilyc acid	1693, 1686
4	CO_3^{2-}	1448, 1421
5	C-O	1231
6	PO_4^{3-}	1035, 603, 564

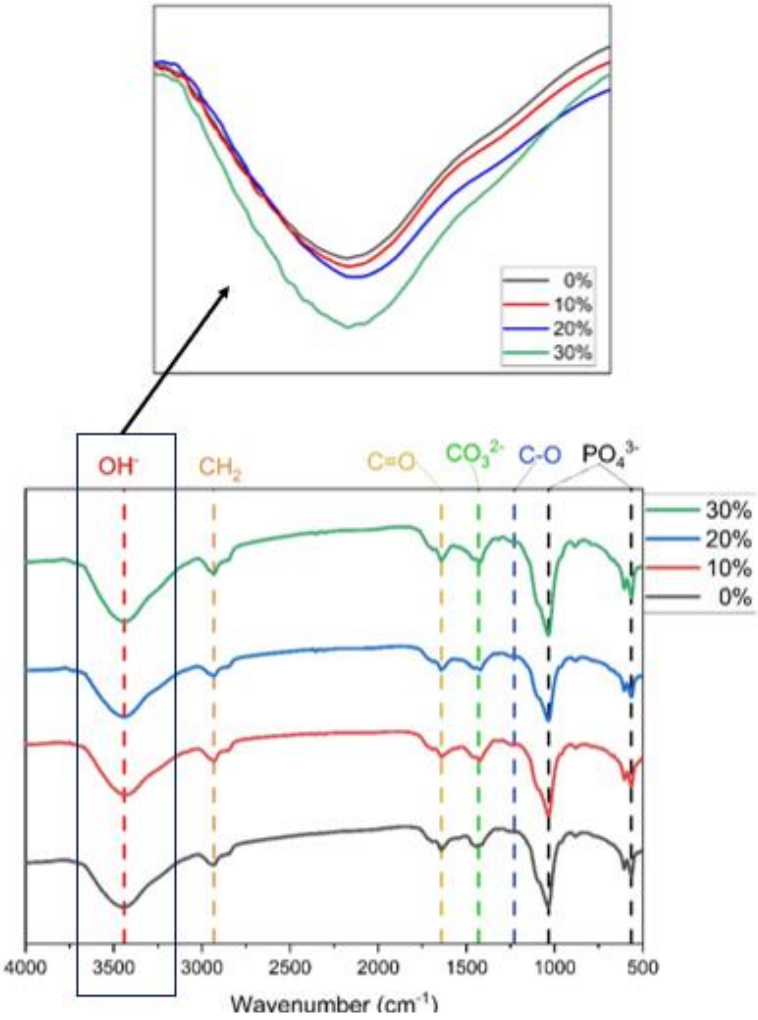


Figure 4. FTIR observations result

3.4 XRD observations

Crystallinity is a value indicating the amount of crystal intensity in a material. It is obtained by comparing crystalline area with total area (crystalline + amorphous). This study's highest crystallinity was 79.65%, occurring at 30% variation. The value of crystallinity at 0%, 10%, and 20% variation respectively was 58.39%; 69.81%; and 71.94% as summarized in Table 2. The highest crystallinity that occurred at 30% variation indicates the atomic structure was more regular than the other three variations [17]. The increasing volume fraction of cantula fiber shows good results in tensile testing, wear rate, and crystallinity of Mg/n-HA/shellac/cantula fiber bio-composite. Crystallinity value is directly proportional to mechanical properties of material [18]. This study has proven that the tensile strength value of material increases in proportion to increasing in crystallinity value. This is also accordance with the results of wear rate test in this study. The higher crystallinity value indicates that addition of cantula fiber volume fraction results in harder material, so wear rate value decreases, indicating the material does not wear out quickly. From this study it can be concluded that Mg/n-HA/shellac/cantula fiber bio-composite has potential to be further developed into bone screw material.

Table 2. XRD observations result

Variation of Cantala Fibre (%)	Crystallinity (%)
0	58.39
10	69.81
20	71.94
30	79.65

4 Conclusions

The highest tensile strength value occurred in 30% cantula fiber variation of 7.86 MPa. In wear rate test, the lowest wear rate occurred at 30% variation of 0.31 x 10^{-3} mm^3/Nm. From FTIR observations, the addition of cantula fiber has an effect on increasing intensity of OH^- functional group which is identical to natural fibers. From XRD observations, the highest crystallinity value was obtained from 30% variation of 79.65%. Cantula fiber acts as a good reinforcement because its addition affects the tensile strength value and wear resistance of material which increases in proportion to the increase of crystallinity value. The higher crystallinity value indicates that addition cantula fiber volume fraction results in increasing material structure strength and material getting harder, so that wear rate value decreases indicating that material does not wear out quickly. This shows that addition of cantula fiber affects the value of tensile strength, wear rate, and crystallinity of Mg/nano-HA/shellac/cantula fiber. Future work in engineering bio-composites for bone screws can be focused on refining material formulations, enhancing fabrication techniques, and developing advanced testing protocols to optimize mechanical strength, biocompatibility, and degradation profiles, ultimately aiming to improve the performance and clinical outcomes of these implants.

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