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CHARACTERIZATION AND MECHANICAL PROPERTIES OF BIOCOMPOSITE OF CANTULA FIBER REINFORCED NANO-HA/MAGNESIUM/SHELLAC FOR BONE SCREW MATERIAL

KARAKTERISASI DAN SIFAT MEKANIK BIOKOMPOSIT NANO-HA/MAGNESIUM/SHELLAC YANG DIPERKUAT SERAT CANTULA UNTUK MATERIAL SEKRUP TULANG

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ABSTRAK

Kecelakaan merupakan salah satu penyebab utama terjadinya patah tulang di Indonesia. Salah satu metode pengobatan patah tulang adalah dengan menggunakan sekrup tulang yang didukung oleh pelat penyangga yang dipasang pada tulang yang patah. Saat ini, banyak material biomedis untuk sekrup tulang yang sedang dikembangkan dengan sifat biodegradable, sehingga tidak memerlukan pengangkatan kembali setelah proses penyembuhan tulang pascaoperasi. Tujuan dari penelitian ini adalah untuk mengetahui pengaruh penambahan serat cantula terhadap kekuatan tarik, laju keausan, dan kristalinitas biokomposit nano-HA/magnesium/Shellac sebagai bahan sekrup tulang. Material nano-HA/magnesium/Shellac/serat cantula dicampur menggunakan blender. Perbandingan antara magnesium dan hidroksiapatit (HA) adalah 70:30, dengan variasi penambahan serat cantula sebesar 0%, 10%, 20%, dan 30% dari total volume. Setelah pencampuran, material dikompaksi dengan tekanan 300 MPa selama 10 menit. Selanjutnya, dilakukan proses sintering pada suhu 140 °C selama dua jam. Hasil penelitian menunjukkan bahwa nilai kekuatan tarik tertinggi diperoleh pada variasi 30%, yaitu sebesar 7,86 MPa. Laju keausan terendah tercatat sebesar 0,31 x 10^{-3} mm^3/Nm pada variasi yang sama. Kristalinitas tertinggi berdasarkan hasil pengamatan XRD juga diperoleh pada variasi 30%, yaitu sebesar 79,65%.

Kata kunci*:* *Biomaterial; Magnesium; Serat Cantula; Tingkat Keausan; Kristalinitas*

ABSTRACT

Accidents are a major cause of fractures in Indonesia. One of 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 biocomposite 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 is added with variations of 0%, 10%, 20% and 30% of total volume. After that, the material mixture was compacted with a pressure of 300 MPa for 10 minutes. Then the sintering process was carried out at a temperature of 140 ^{o}C for two hours. The results showed that the highest tensile strength value was 7.86 MPa at 30% variation.

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The lowest wear rate was 0.31 x 10-3 mm3/Nm at 30% variation. The highhest crystallinity in XRD observations was obtained at 30% variation, which was 79.65%.

Keywords: *Biomaterial; Magnesium; Cantula Fiber; Wear Rate; Cristallinity.*

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 for the treatment of fractures. Magnesium was chosen as a biomaterial because it is biodegradable and exhibits mechanical properties similar to those of human bones (Liu et al., 2018). Hydroxyapatite was added to reduce the rapid degradation rate of magnesium (Rahman et al., 2020). Hydroxyapatite has the same mineral content as human bone. Hydroxyapatite is biocompatible, osteoconductive, and capable of stimulating bone formation (Mozartha, 2015).

Shellac is added to bind hydroxyapatite with other materials. Shellac is a natural polymer that possesses biodegradable, nontoxic, and renewable properties (Triyono et al., 2021). 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 (Raharjo et al., 2021).

In this study, a magnesium/HA/ Shellac biomaterial was combined with cantula fibre for the manufacture of bone screws. The results of this study are expected to provide an alternative material for bone screws, which will be beneficial for patients with bone fractures.

Method

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

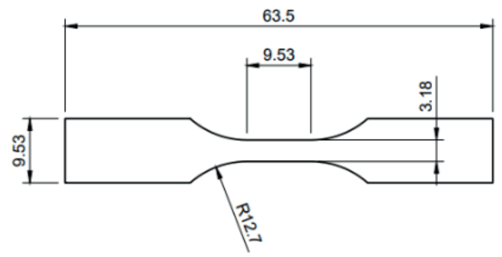
The preparation of a Shellac solution begins with crushing the secretions of Shellac lice using a mortar, followed by pulverizing them in a blender to make a powder. After that, the lacquer secretion powders were mixed with 96% ethanol using a magnetic stirrer. Mixing was carried out for 4 hours with a ratio of lacquer secretion powders to ethanol of 1:10. Then, the Shellac solution was allowed to precipitate for a while, and the filtering process was carried out. The result of this filtering was a Shellac solution.

The cantula fiber used in this study had previously been pretreated by soaking it in 2% NaOH solution for 6 hours. After that, the cantula fibers were washed with clean water and left to dry for 3 days. Then, the cantula fibre dried at 110°C for 45 minutes. After drying, the cantula fibre was cut into lengths 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.

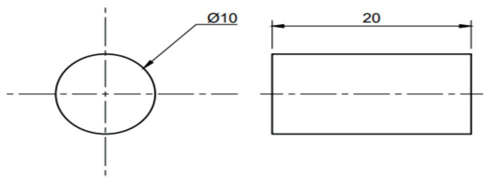
The preparation of the Mg/HA/Shellac mixture and cantula fibre was initiated by mixing the nano-hydroxyapatite and Shellac solution using a magnetic stirrer, which was carried out for 2 hours at a temperature of 100 °C and a speed of 200 rpm. The cantharidin in the mixture is in the form of a dry powder. Then, it was mixed with magnesium powder and calcium powder using a blender at three different speeds for 1 minute each. The mixed materials were then placed into the mould and pressed using a press machine with a pressure of 300 MPa. The formed specimen was then removed from the mould.

The compacted specimens were sintered in a furnace at 140 °C and held for 2 hours. This sintering process was suitable for strengthening the bond between the powder grains and reducing the porosity of the specimen. This specimen from the sintering process would be tested as bone screw material. This study presents the results of bone screw composite tests treated with two methods, without treatment and with heat treatment. Bone screws without heat treatment will be sintered at a temperature of 140°C and left for 2 hours, with variations in the addition of cantula fiber, starting from fiber volume fractions of 0%, 10%, 20%, and 30%. Furthermore, bone screws with the heat treatment method will be sintered with varying heat, namely, 100, 120, 140, and 160 (°C) for 2 hours with a ratio of magnesium, nano-HA-Shellac, and cantula fiber respectively, namely 5:2:3 (vt%).

In this study, several tests have been divided. Testing of untreated specimens involves tensile tests, wear tests, FTIR analysis, and XRD analysis. Tensile tests using UTM according to ASTM D638 type V with a load of 50 kg and a tensile speed of 10 mm/min. Wear tests were conducted using a pin-on-disc type tribometer machine, as per ASTM G-99, with a load of 2 kg, a friction speed of 1 m/s, and a path length of 501.58 m. FTIR (Fourier Transform Infrared) observations were conducted to identify the functional groups present in the specimen. To determine the crystallinity of the material, X-ray diffraction (XRD) analysis was performed.



(a)



(b)

Figure 1.
Test specimen; (a) tensile test ASTM D368 type V, (b) wear rate ASTM G-99
Source: Author's documentation (2025)

Furthermore, specimen testing with treatments involves bending tests, fracture toughness tests, and degradation rate tests. Bending test using UTM according to ISO 178 three-point bending method with a load cell of 50 Kg and a compression rate of 10 mm/min. The fracture toughness test is carried out using the SENB (Single Edge Notch Bend) method, as specified in ASTM D-5045, with a load cell of 50 Kg and a compression rate of 10 mm/min. A degradation test was conducted by immersing the specimen in a container containing 20 mL of PBS (Phosphate-Buffered Saline) solution, which was incubated at a temperature of 36-38°C. Furthermore, the specimen is observed for weight loss for 28 days.

RESULTS AND DISCUSSION

Untreatment

Tensile Test

Figure 2 shows that the tensile strength of magnesium/n-HA/Shellac/fiber specimens increases with the addition of cannula fiber content, as follows: 0%, 10%, 20%, and 30%, respectively, with values of 2.73 MPa, 5.48 MPa, 6.96 MPa, and 7.86 MPa. The result of those variations was comparable to the tensile strength of human bones, which is 2.54 MPa (Røhl et al., 1991). However, the tensile strength results from this study are significantly lower than that of 316L stainless steel, which is typically used as an implant for bone, at 465 MPa (Prakash et al., 2016).

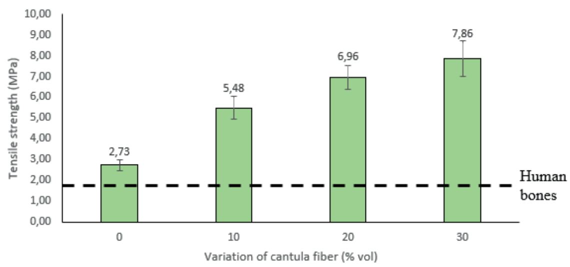


Figure 2.
Tensile strength test result
Source: Author's analysis (2025)

The fibers in the composite mixture resist most of the forces acting on the mixture. The role of the matrix in the mixture is to protect and bind fibers to work properly. The tensile strength value increases with the addition of cantula fiber. This happens because the cantula fibers used have a high aspect ratio, specifically 21.11. If the aspect ratio is high, the tensile strength will also increase because the fibers are able to withstand loads effectively. The more cantula fibers added, the greater the load they can withstand (Mahmuda et al., 2013). Alkaline treatment also enhances fiber-matrix bonding by cleaning and roughening the fiber surface, thereby promoting mechanical interlocking (Fathoni et al., 2017).

Wear Rate Test

Figure 3 shows that the specific wear rate of the magnesium/n-HA/Shellac/cantula fiber specimens decreased with increasing cantula fiber content, measured at 3.38, 1.16, 0.81, and 0.31 × 10^{-3} mm^3/Nm for 0%, 10%, 20%, and 30% variations, respectively. A previous study reported that the specific wear rate of HA/Shellac/cantula fiber implants was 0.51 × 10^{-3} mm^3/Nm (Rahardjo et al., 2022). The 30% variation in this study showed a lower wear rate than that reference value, while the other variations were higher. However, all four variations still exceeded the specific wear rate of human bone, which is 0.082 × 10^{-3} mm^3/Nm (Jawaiid et al., 2018).

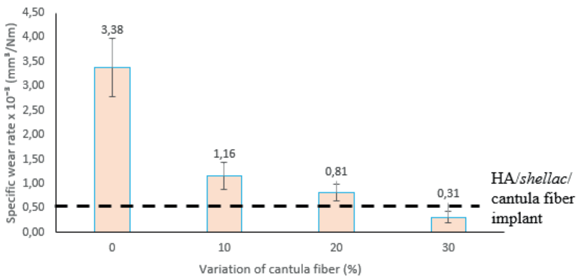


Figure 3.
Wear rate test result
Source: Author's analysis (2025)

Cantula fiber acts as a sound reinforcement because its addition decreases the specific wear rate of Mg/n-HA/Shellac/cantula fiber mixture. This is related to the increase in crystallinity of each variation, thereby increasing the hardness value of the material (Septiani et al. 200)]. Increasing crystallinity indicates the strength of the material structure is increasing. In addition, the increase in tensile strength value also affects a decrease in the wear rate of the specimen. Cantula fiber exhibits good resistance to wear characteristics of biocomposite materials as indicated by the reduced wear rate as shown in Figure 3 (Sunardi et al., 2015).

FTIR Observation

Based on Figure 4, at wavenumbers of 3445 cm-1 and 3429 cm-1, an OH- (hydroxyl) peak appears, which is identical to that of n-HA (Suryadi, 2011). 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 affects increasing intensity of OH- functional group as shown in Figure 4. The wavenumber at 2932 cm-1 corresponds to the CH2 (methylene) functional group, which is identical to that of natural fiber (Raharjo et al., 2018). In addition, that wavenumber was also influenced by the presence of Shellac.

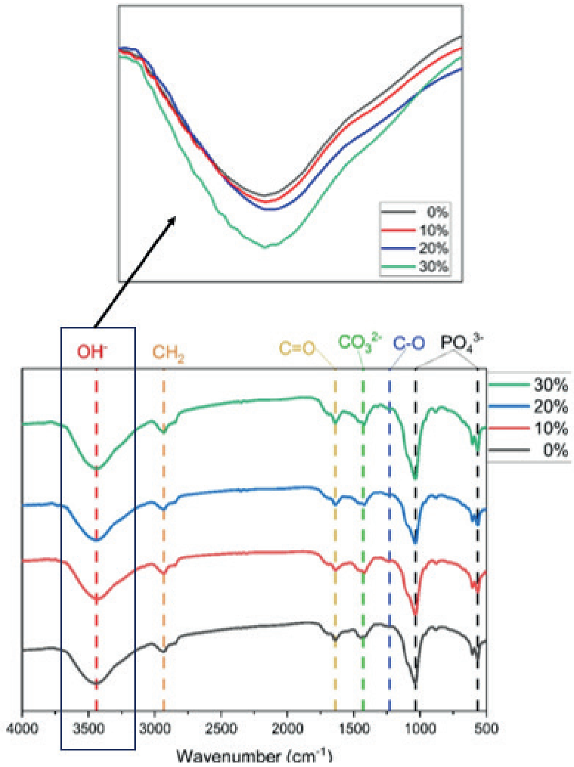


Figure 4.
FTIR observations result
Source: Author's analysis (2025)

Table 1.
Functional groups from FTIR observations

No	Functional Groups	Wavenumbers (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

Source: Author's analysis (2025)

Wavenumbers at 1693 cm-1 and 1686 cm-1 were identified as characteristic of the C=O functional group, which is typically associated with carboxylic acid or ester structures derived from Shellac (Wang et al., 2008). In wavenumbers 1448 cm-1 and 1421 cm-1, CO32- (carbonate) functional group appears due to the reaction between carbon dioxide and n-HA in free air (Suryadi, 2011). At wavenumber 1231 cm-1, C-O functional group appears due to cantula and Shellac fiber bonds (Wang et al., 2008). At wavenumbers of 1035 cm-1, 603 cm-1, and 564 cm-1, a sharp peak of the PO43- (phosphate) functional group is observed, resulting from the strong influence of n-HA (Suryadi, 2011).

The emergence of covalent bonds in the form of C-H, C-O, and C=O resulted in stronger bonds between atoms. This causes the wear resistance of the specimen to increase because the material is not easily eroded (Ridwan et al., 2022).

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).

Table 2.
XRD observations result

Variation of Cantula Fiber (%)	Crystallinity (%)
0	58,39
10	69,81
20	71,94
30	79,65

Source: Author's analysis (2025)

In this study, the highest crystallinity, at 79.65%, occurs at a 30% variation. The value of crystallinity at 0%, 10%, and 20% variation respectively is 58.39%; 69.81%; and 71.94%. The highest crystallinity that occurs at 30% variation indicates the atomic structure is more regular than other three variations (Arini et al., 2015). Increasing the volume fraction of cantula fiber yields good results in terms of tensile testing, wear rate, and crystallinity of the Mg/n-HA/Shellac/cantula fibre biocomposite.

The crystallinity value is directly proportional to the mechanical properties of the material (Sumiati et al., 2016). This study demonstrates that the tensile strength of the material increases in proportion to the increase in crystallinity. This is also by the results of the wear rate test in this study. The higher crystallinity value indicates that addition of cantula fiber volume fraction results in more rigid material, so wear rate value decreases which indicates the material does not wear out quickly. From this study it can be concluded that Mg/n-HA/ Shellac/cantula fiber biocomposite has potential to be further developed into bone screw material.

Treatment

Bending Strength Test

The bending strength test showed that the bending stress of the magnesium/HA/ Shellac/cantula fiber specimens increased with the heating temperature. The results of the bending test are shown in Figure 3, respectively, at temperatures of 100°C, 120°C, 140°C, and 160°C, with corresponding stresses of 11.19, 20.14, 25.76, and 27.67 MPa. At temperatures of 120°C, 140°C, and 160°C, the bending strength value of Magnesium/HA/ Shellac/Cantula Fibre exceeds that of human bone by 10-20 MPa, but it remains lower than that of stainless steel (White et al., 2009).

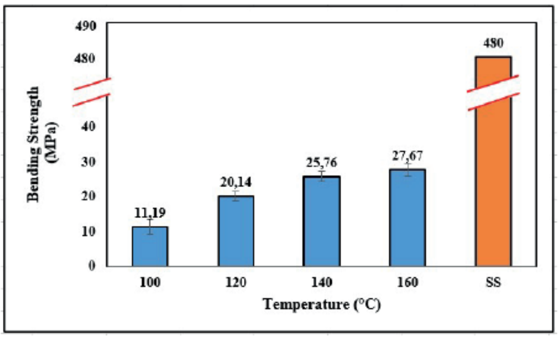


Figure 5.
Bending test result
Source: Author's Analysis (2025)

The lowest bending stress is 11.19 MPa at 100°C, where the bonding between particles has not occurred entirely or has not formed a bond. The highest value was obtained at 160°C, with a pressure of 27.67 MPa. This relationship is directly proportional to density and inversely proportional to porosity, as illustrated in Figures 1 and 2. As the density increases and the porosity decreases, the bending strength value increases with increasing heating temperature. The results of this test are supported by a study from Lucky (2020), that the higher the density and the lower the porosity, the greater the bending stress of the material (Erliyanti et al., 2020; Sukanto, 2009).

An increase in the heating temperature of the specimen causes an increase in the modulus of elasticity. The value of the modulus of elasticity for variations of 100°C,120°C, 140°C, and 160°C respectively is 1.78, 5.73, 9.78, and 15.02 GPa. Figure 4 shows that each variation in heating temperature will form a curve with a different slope. The slope of this curve affects the value of the modulus of elasticity. The higher the heating temperature, the smaller the deflection of the specimen, resulting in an increased slope of the curve and a higher modulus of elasticity.

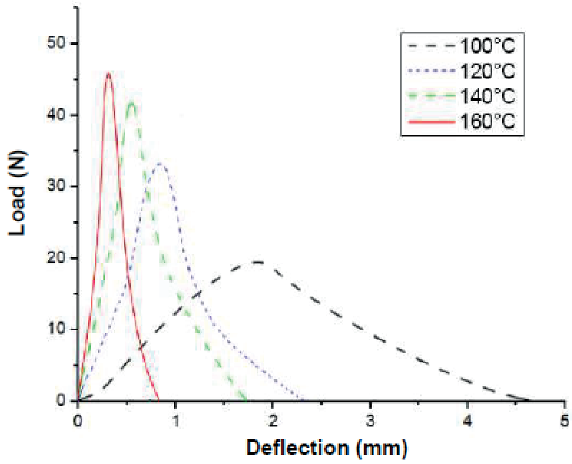


Figure 6.
Bending test load/deflection curve
Source: Author's analysis (2025)

Fracture Toughness Test

The fracture toughness test showed that as the temperature increased, the amount of fracture toughness increased sequentially, namely at 100°C, 120°C, 140°C, and 160°C, with values of 17.05, 19.57, 22.47, and 24.97 MPa/m, respectively. However, the fracture toughness of Magnesium/HA/Shellac/Cantula Fiber is lower than That of Stainless Steel at 95 MPa/m.

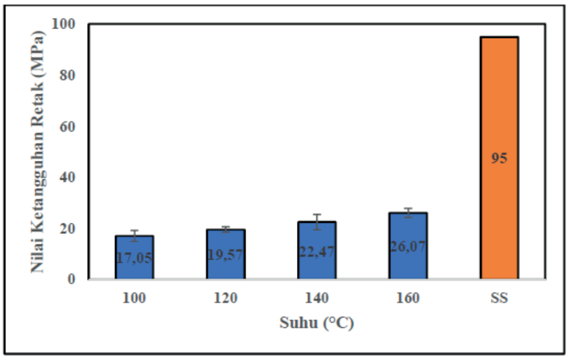


Figure 7.
Fracture toughness test results
Source: Author's analysis (2025)

Figure 7 shows the results that the fracture toughness increases as the heating temperature increases. This is because the increasing heating temperature causes the porosity to decrease. As the particles begin to bind, the gaps in the specimen start to fill, increasing the value of fracture toughness. These results are reinforced by a study by Tjokorda (2002), which shows that increasing the heating temperature results in lower porosity and increased fracture toughness (Nindhia et al., 2002).

Degradation Rate

The degradation rate test aims to determine the decay rate of Magnesium/HA/Shellac/Cantula Fiber specimens annually. The results of the biodegradation rate test are presented in Figure 8.

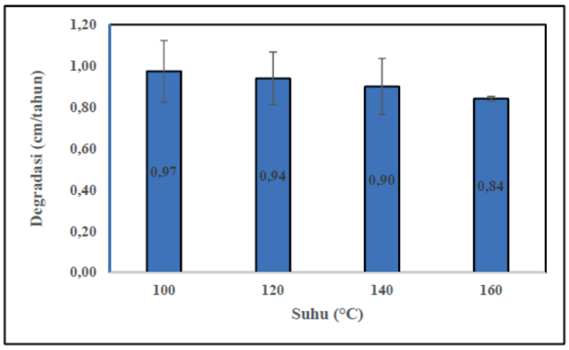


Figure 8.
The degradation rate of magnesium/HA/Shellac/cantula fiber
Source: Author's analysis (2025)

Figure 8 is a graph of the degradation rate of the Magnesium/HA/Shellac/Cantula Fiber specimen. The graph shows the average annual rate of degradation for each temperature variation of 100°C, 120°C, 140°C, and 160°C, which are 0.97, 0.94, 0.9, and 0.84 cm/year, respectively. As the temperature increases, the degradation rate slows down (decreases); it is directly proportional to the porosity value in each specimen. This occurs because the percentage of porosity increases and the density decreases, allowing the contact between the specimen's surface and the PBS solution to widen (Triyono et al., 2020).

In addition, the size of the crack in the specimen also affects the value of the degradation rate. Figure 7 shows that as the heating temperature increases, the size of the crack shrinks. At 100°C, the crack size tends to be larger due to the weaker bonds between particles. It causes the PBS solution to enter more easily and dissolve more readily.

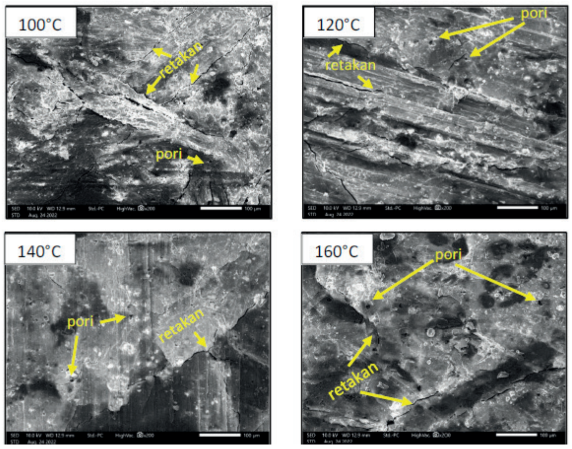


Figure 8.
Photos of Scanning Electron Microscope (SEM) 200x
Source: Author's documentation (2025)

Meanwhile, at 160°C, the cracks are more minor, and the density is low, which lowers the contact between the specimen surface and the PBS solution. Cracks occur due to the different thermal expansion values of each material that makes up the specimen. Rapid changes in the cooling temperature cause each material to shrink, resulting in cracks on the surface.

CONCLUSION

From this research, it was found that the highest tensile strength value occurred in 30% cantula fiber variation of 7.86 MPa. In the wear rate test, the lowest wear rate occurred at a 30% variation of 0.31 x 10^ (- 3) mm^3/Nm. From FTIR observations, the addition of cantula fiber affects the increasing intensity of OH- functional group which is identical to natural fibers. From XRD observations, the highest crystallinity value was obtained at a 30% variation, with a value of 79.65%.

Cantula fiber acts as a sound reinforcement because its addition affects the tensile strength value and wear resistance of the material, which increases in proportion to the increasing 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 the addition of cantula fiber affects the value of tensile strength, wear rate, and crystallinity of Mg/nano-HA/Shellac/cantula fiber.

Based on the testing process, observations, and the results of the discussions carried out in the study, the following conclusions can be drawn. Bending test on Magnesium/HA/Shellac/ cantula fiber specimens showed that the higher the heating temperature, the higher the bending stress value. Fracture toughness test on Magnesium/HA/Shellac/cantula fiber specimens showed that the value of fracture toughness increased with increasing heating temperature. Observation of the degradation rate on specimens of Magnesium/HA/ Shellac/ cantula fiber showed that the rate of degradation slowed down with increasing heating temperature.

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