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Submission date: 08-May-2021 09:51AM (UTC+0700)

Submission ID: 1581072680

File name: Sirait_2021_J._Phys._Conf._Ser._1811_012016.pdf (1,008.08K)

Word count: 3906

Character count: 20896

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To cite this article: Makmur Sirait *et al* 2021 *J. Phys.: Conf. Ser.* **1811** 012016

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Thermal and Mechanical Properties a Membrane of the Mixing PVA Nanocomposite and Limestone Hydroxyapatite

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Abstract. Synthesis and characterization of membranes from a mixture of PVA nanocomposites and limestone hydroxyapatite have been carried out. The limestone is crushed, sieved with size of 200 mesh. The synthesis of hydroxyapatite was carried out by heating limestone powder using a furnace at a temperature of 600 °C for 4 hours. Furthermore, limestone was dissolved with distilled water and 0.3M H₃PO₄ solution using a magnetic stirrer with stirring at 300 rpm for 1 hour. The pH of the solution was made 10 by adding 1 M NH₄OH periodically. The solution was aged for 24 hours and the precipitate was dried using an oven at 120 °C for 5 hours. The hydroxyapatite powder was characterized by FTIR to see the functional groups and XRD characterization to see the hexagonal hydroxyapatite phase. The characterization results obtained that the main groups of hydroxyapatite compounds were calcium hydroxide (CaO), phosphate (PO₄³⁻), and hydroxyl (OH⁻) groups with a crystal size of 86.27 nm. Limestone hydroxyapatite was mixed with a solution of PVA-chitosan with a composition variation of 0%, 1%, 2%, 3%, and 4% weight using a magnetic stirrer at 40°C for 3 hours using the sol-gel method. The membrane obtained was characterized by DSC to determine its thermal properties and UTM to determine its mechanical properties. The maximum melting point obtained in the mixture (98: 2) % was 195.51°C and the highest tensile strength was at the percentage of 1% with a modulus of elasticity of 22.28 M.Pa and tensile strength of 13.23 M.Pa.

Keywords: Thermal, Mechanic, Nanocomposite, Hydroxyapatite, PVA.

1. Introduction

Nanocomposite polymers consist of nanometric particles dispersed in the polymer matrix. Combining a small number of nanometer-sized fillers can produce composites with better properties for industrial and technological applications [1]. Biopolymer-based packaging materials have not been widely used in the packaging industry, mainly because of the lack of good mechanical properties. One way to overcome such deficiencies is to combine them with other materials or fillers[2]. Recent studies have shown that homogeneous mixing between polymers with various types of filler nano-sized materials will result in improvements to physical properties, mechanics, barrier[3], and thermal properties when compared to micro-sized[4]. One of the many intensively studied biopolymers is polyvinyl alcohol (PVA) because of its nature which can shape the film well, soluble in water, easy in process, not toxic, biocompatible, and biodegradable[5].

Recently it has developed the PVA/chitosan composite membrane improving chemical stability, proton conductivity, and lowering methanol permeability. At the mixing of PVA/chitosan with a



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comparison of 9:1 that is tied cross with glutaraldehyde for 1 hour obtained a proton conductivity of $20 \times 10^{-3} \text{ s cm}^{-1}$ and methanol permeability of $16.24 \times 10^{-7} \text{ cm}^2 \text{ s}^{-1}$. This result showed a mixture of PVA/Chitosan which is cross-bound with glutaraldehyde is better than the Nafion® 115 which has a proton conductivity of $14 \times 10^{-3} \text{ s cm}^{-1}$ and methanol permeability of $18 \times 10^{-7} \text{ cm}^2 \text{ s}^{-1}$ [6].

Polyvinyl Alcohol (PVA) is a resin that is made from the merger of the molecules (polymerization) of vinyl acetate, by hydrolysis of a portion of the ester as a catalyst. The physical properties of polymerization depend on the degree of polymerization and the degree of hydrolysis [7]. PVA has good chemical stability, the ability to form a film, and the properties of hydrophilicity (molecular likelihood for water dissolved) high. Also, PVA is soluble in water, is biodegradable, non-toxic, and indicates minimal adhesive. PVA can form pores and able to interact with hydrophilic interfaces with body tissues [8]. PVA can be modified with a variety of methods such as the sol-gel method, UV radiation method [9]. PVA is a brittle polymer homo that in its use PVA combined with other polymers or ceramic materials to increase mechanical strength. Hydroxyapatite is one of the potential ceramic type that can be combined with PVA in fixing the mechanical strength of the membrane [10].

Hydroxyapatite $\text{Ca}_{10}(\text{PO})_4(\text{OH})_2$ is a biomaterial used in bone graft bone implants to repair damaged tissues and broken bones. Hydroxyapatite (HAp) has biocompatible and osteoconductive properties. Hydroxyapatite synthesis was carried out by reacting to Ca and P with a comparison of mol 1.67 [11]. Hydroxyapatite can be produced by using a natural substance that contains a lot of calcium in the form of calcium carbonate CaCO_3 . Calcium carbonate can be found easily around our living environments such as in limestone, bones, shells, and the protector of soft animals [12]. Margareta [13] uses limestone as a material for the manufacture of hydroxyapatite, the reason researchers use limestone because limestone contains pure calcium which is quite high at 98.2%.

HAp can be used in areas with relatively low mechanical strength, such as bone and tooth fillers, or coatings on implant devices. The bones of humans who often experience fractures are tibia and fibula that serve to support the weight of the body while standing, mechanical strength also plays an important role. To overcome these limitations, it has developed polymer-biologic composites in recent decades [10].

Degirmenbasi et al report the manufacture of biocomposites from hydroxyapatite – collagen-polyvinyl alcohol. Hydroxyapatite is mixed in situ in a mixture of collagen/PVA. The resulting biocomposites show material with high elasticity, which is increasing with cryogenic treatment. The amalgamation of collagen in PVA/Hap biocomposites resulted in internal porosity with interconnected pore sizes in the range of 50-500 nm [14].

2. Experimental Method

The method used in this study is the coprecipitation method and sol-gel methods. Limestone in ball mill and filtered with 200 mesh size, then calcined using a furnace with temperature 600°C within 4 hours to become CaO powder. The CaO suspension has added a solution of H_3PO_4 0,3 M as much as 200 ml with an additional rate of 5 ml/min and stirred using a magnetic stirrer for 1 hour at a speed of 300 rpm. Then heated solution using a hotplate with temperature 90°C and pH solution is set to 10 with the addition of solution NH_4OH 1 M periodically. The solution that has been heated to pH 10 is allowed for 24 hours (aging) at room temperature. Then the solution is filtered with a filter paper to obtain the precipitate. Deposits are dried using an oven with a temperature of 120°C for 5 hours.

The membrane making process is 24 grams PVA dissolved with 200 ml aquades using a magnetic stirrer at 80°C and 500 rpm speed and 24 grams chitosan dissolved with 200 ml acetic acid using stirrer to homogeneous. PVA and chitosan solution in stirrer up to homogeneous with a ratio of 70%:30% at a temperature of 40°C . Then Hydroxyapatite is mixed with PVA-Chitosan solution with a percentage of 0%, 1%, 2%, 3%, and 4% weight. The hydroxyapatite-PVA-Chitosan solution in a stirrer for 3 hours with a temperature of 40°C , then printed on the mold of glass to form a membrane. The membrane of the PVA nanocomposite mixture and hydroxyapatite are characterized using DSC, SEM, thermal test, and mechanical testing.

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3. Results and Discussion

Analysis of the FTIR spectrum on hydroxyapatite is done to know the function group. The cluster of functions on hydroxyapatite is characterized by the emergence of absorption in the number of waves (500–4000) cm⁻¹. The infrared spectrum of hydroxyapatite with alkaline deposition method is presented in Figure 1 and the analysis of the high peaks is presented in Table 1.

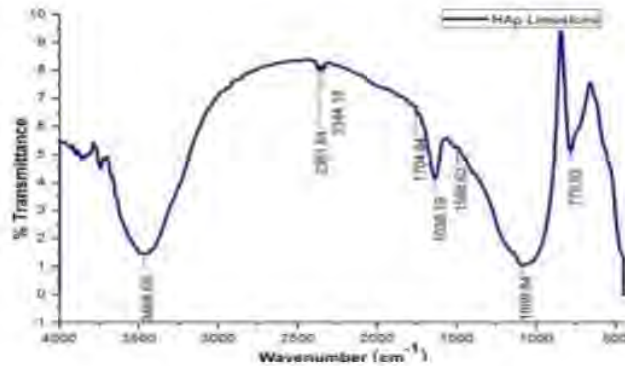


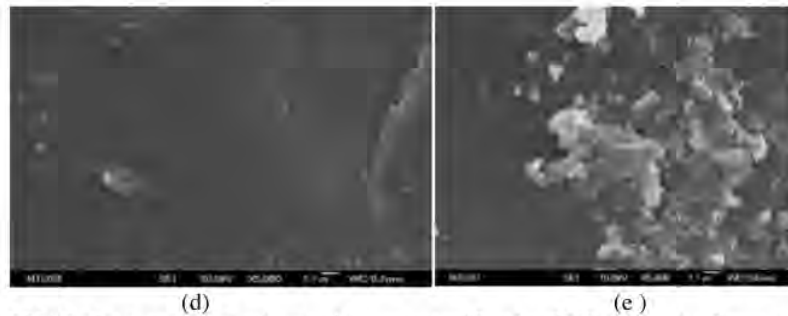
Figure 1. Limestone Hydroxyapatite Powder Spectrum

Table 1. FTIR graph peak comparison data of hydroxyapatite.

Functional Groups	Number of waves (cm ⁻¹)		
	Limestone HAp	Yellowfin Tuna Fish Bone HAp	200 Japan HAp (standard)
Phosphate (PO ₄) ³⁻	779.92; 1089.84	983.74;	560.21; 600.72; 1136.00
Carbonate (CO ₃) ²⁻	1508.63	1460.18; 1546.98;	1400.00
Hydroxyl (OH)	3468.68	3571.36	3425.58

The hydroxyapatite graph in Figure 1 and the comparative data of the hydroxyapatite peaks in Table 1 indicates the peak absorption of the phosphate cluster (PO₄)³⁻ on the hydroxyapatite of the limestone is characterized by uptake in the number of waves about 779.92 cm⁻¹ and 1089.84 cm⁻¹. The Carbonate-function cluster (CO₃)²⁻ which appears on the number of waves 1508.63 cm⁻¹ indicates a C-O vibration from the CO₃ cluster. The existence of hydroxyl cluster (OH) on the hydroxyapatite of limestone is indicated by the emergence of peak absorption of approximately 3468.68 cm⁻¹. There are a phosphate and hydroxyl group in the limestone hydroxyapatite, indicating that there has been a very good hydroxyapatite phase while the presence of clusters -CO₃ in the mixture of hydroxyapatite compounds with small intensity so that the possibility of forming amorphous phases.

The results of the X-ray diffraction pattern on the hydroxyapatite shown in Figure 2.



Figures 3. PVA hydroxyapatite membrane-chitosan magnification 5000 times. a) mixture of HAp 0%; b) mixture of HAp 1%; c) mixture of HAp 2%; d) mixture of HAp 3%; e) mixture of HAp 4%.

Figure 3 shows the morphology of the membrane's slightly wavy surface with a fairly homogeneous spread. Besides, there are white dots on the membrane signifying chitosan has not been perfectly mixed in solution. This less-than-perfect surface and mixture can occur due to the less conscientious manufacturing process, both stirring, unstable temperature, and less good drying process. From the image obtained that the more the percentage of hydroxyapatite increases, the more noticeable the pore spreads are not homogeneous. The size of the pore in the limestone hydroxyapatite membrane with a percentage of hydroxyapatite 0%, 1%, 2%, 3%, and 4% are calculated using the application ImageJ ranging between $39 \mu\text{m}$, $30 \mu\text{m}$, $43 \mu\text{m}$, $36 \mu\text{m}$, and $34 \mu\text{m}$.

Characterization of thermal testing of hydroxyapatite membrane PV Achitosan-limestone was carried out using TGA/DSC with the data obtained the percentage of mass in the mixture of hydroxyapatite 0%, 1%, 2%, 3%, and 4% as in Figure 4.

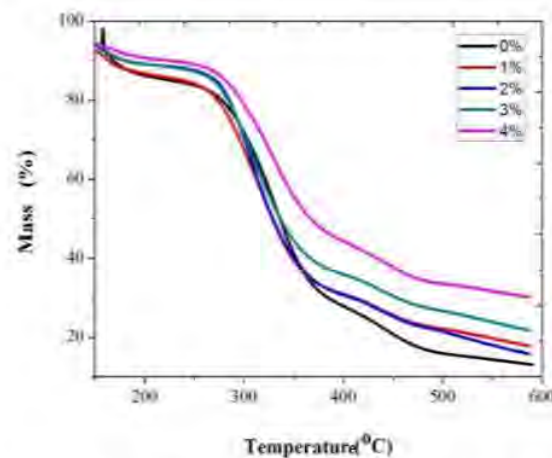


Figure4. TGA Thermogram Graph Membrane Hydroxyapatite-Chitosan-PVA

Figure 4 shows the influence of the HAp mixture on the thermal stability of the Hydroxyapatite-chitosan-PVA membrane. The thermal stability of each sample can be seen from the mass change occurring. Based on the results of the thermal characterization of the membrane obtained T_d value at ambient temperature (280-300) °C. The membrane suffered mass loss from temperature (150-160) °C, but after the temperature instability, there was a mass loss percentage. The

TGA Thermogram graph also shows that the percentage of HAp mixed in the membrane is directly proportional to the percentage of the mass membrane.

Figure 5 is the DSC result showing the endothermic process (heat absorption) which aims to determine the glass transition temperature (T_g) and the melting temperature (T_m) of the membrane HAp-chitosan-PVA. While Figure 6 shows an exotherm (heat release) process that aims to determine the temperature of the crystallinity (T_c) from membrane hydroxyapatite-Chitosan-PVA.

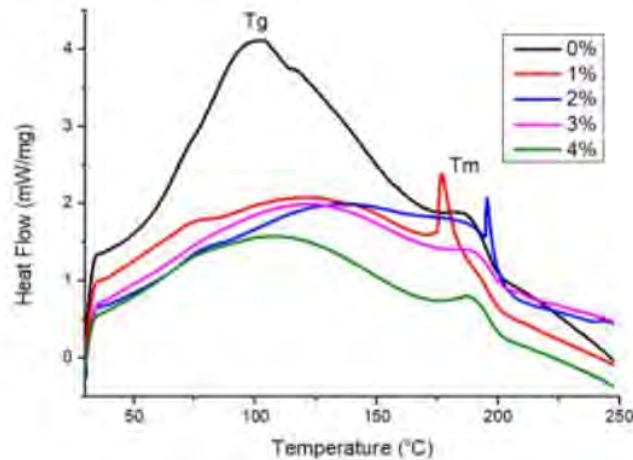


Figure 5. Thermogram DSC of Hydroksiapatit-Chitosan– PVAmembrane in the heating process

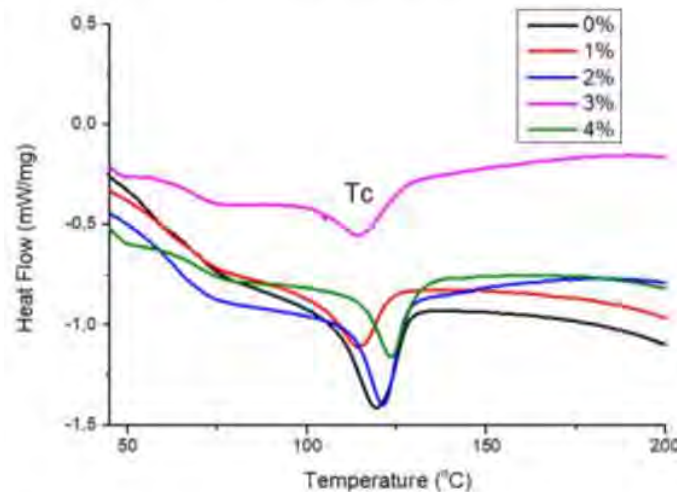


Figure6. Thermogram DSC of Hydroksiapatit-chitosan – PVAmembrane in the cooling process

From the thermal test results can be obtained some information about the thermal data showed in Table 2 as follows:

Table2. The thermal test result of membrane hydroxyapatite-PVA

Hydroxyapatite composition (%)	Tg (°C)	Tm(°C)	Tc(°C)	Td (°C)
0	100.96	185.67	76.17	300.05
1	124.47	177.00	74.34	285.43
2	130.14	195.51	73.68	288.28
3	122.80	191.34	75.67	288.78
4	111.29	186.33	76.50	298.22

From Table 2 can be analyzed that the addition of the composition of hydroxyapatite in the membrane will reduce the temperature value of the glass transition (Tg) and the melting temperature (Tm) in the membrane, this is seen in the sample with the composition of HAp (2-4)%. While the temperature value of crystallinity (Tc) has the highest temperature in the 4% hydroxyapatite mixture of 76.50°C. Glass transition expansion or the high temperature of the glass transition in the membrane occurs because of a very strong sample composite bond, which is a HAp ceramic with polymer PVA. When the membrane reaches crystallinity temperature, the evaporation of the liquid parts leaves a solid piece of the sample that begins to form a regular molecular arrangement (crystal lattice). Samples that are already in the form of solids get heating continuously with a temperature rise, resulting in a stronger vibration. This resulted in the sample being decomposed so that the sample resistance to low heat and formed a melting point (Tm)[16]. From Table 2 can be analyzed that the addition of the composition of hydroxyapatite in the membrane will reduce the temperature value of the glass transition (Tg) and the melting temperature (Tm) in the membrane, this is seen in the sample with the composition of HAp (2-4)%. While the temperature value of crystallinity (Tc) has the highest temperature in the 4% hydroxyapatite mixture of 76.50 °C. Glass transition expansion or the high temperature of the glass transition in the membrane occurs because of a very strong sample composite bond, which is a HAp ceramic with polymer PVA. When the membrane reaches crystallinity temperature, the evaporation of the liquid parts leaves a solid piece of the sample that begins to form a regular molecular arrangement (crystal lattice). Samples that are already in the form of solids get heating continuously with a temperature rise, resulting in a stronger vibration. This resulted in the sample being decomposed so that the sample resistance to low heat and formed a melting point (Tm)[16]. DSC and TGA characterization results are interconnected, which can be seen from the thermography graph in which the TGA graph at temperatures (150-160)°C begins to occur mass loss in the membrane, it is supported by the results of the DSC thermogram where the Tm value is at a temperature (177-195)°C. Such instability is likely due to the process of disconnection of branched chains on the membrane of the polymer into monomer-monomers due to high temperatures[17].

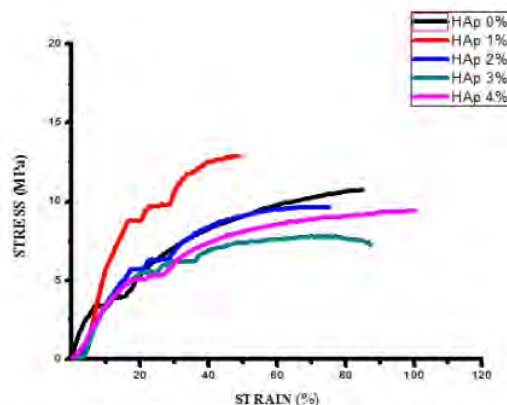


Figure 7. The Mechanical Test Results

Figure 7 shows the influence of the HAp mixture on the mechanical test. The result of the mechanical test of the high-limestone hydroxyapatite membrane is at a percentage of 1% with the modulus of elasticity 22.28 M.Pa and tensile strength 13.23 M.Pa. The addition of HAp 1% in the solution of Chitosan-PVA produces higher tensile strength and modulus value than others. The image can be seen as less good pull results with curves on each sample. This proves the mixture of the hydroxyapatite is less homogeneous and the stirring process is uneven. According to Warastuti[18], the increasing concentration of hydroxyapatite leads to a reduced tensile strength value and a drop extension. In the process of mixing PVA and chitosan on the membrane with the addition of hydroxyapatite less homogeneous, so that the interaction of active groups in PVA with active groups in the less maximum. It also corresponds to the SEM results where the morphology is less homogeneous.

4. Conclusion

The crystalline size of the hydroxyapatite limestone used as the PVA nanocomposite mixture is 86.6 nm with a hexagonal structure. Based on the results of a thermogram TGA/DSC membrane nanocomposite mixture of PVA and hydroxyapatite limestone obtained glass transition temperature and maximum melt temperature in the mixture (98:2) % namely 130.14°C and 195.51 °C, while the crystallization temperature and decomposition temperature in the mixture (96:4) % namely 76.50°C and 298.22°C. Result of mechanical characterization of the membrane with strong maximum tensile value at 1% hydroxyapatite mixture with modulus elasticity value 22.28 MPa and tensile strength 13.23 MPa. The addition of this hydroxyapatite mixture in the PVA nanocomposite is still less consistent with the value of thermal properties and mechanical properties, so it needs a more thorough manufacturing process and a more suitable mixture.

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Acknowledgments

The authors would like to acknowledge the Directorate of Research and Community Services, the Directorate General of Higher Education that has funded this Prime Research Universities with the

number contract of 190²P2H/AMD/LT/DRPM/2020¹³. Also, the author's gratitude is addressed to the Head of the University and Chairman of the Research Institute of the State University of Medan and other related parties that have supported the research until its completion.

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