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SYNTHESIS AND CHARACTERIZATION OF CRAB SHELL CHITOSAN SOLID ELECTROLYTE POLYMER (SPE) MEMBRANE

Muayanah✉[1], Multazam[1], Sun Theo C.L. Ndruru[2]

[1] Program Studi Tadris Kimia, Universitas Islam Negeri Mataram, Mataram, 83116.
[2]Pusat Riset Kimia Maju, Badan Riset dan Inovasi Nasional, Banten, 15314
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ABSTRACT

Efforts to prevent a global energy crisis by using renewable and environmentally friendly energy. Batteries are an alternative to replace non-renewable energy. However, in the battery, there is an electrolyte component which when finished using the battery will cause waste that causes the soil to become toxic. This study aims to determine the characterization of chitosan solid electrolyte membranes, in crab shells. The characterization carried out is the characterization of crab shell chitosan using an FTIR spectrophotometer, ion conductivity analysis of solid electrolyte membranes using an LCR Meter, and tensile tests using Tensilon. The method used in this research is experimental. Isolation of chitin and chitosan from crab shells was carried out in several stages, namely deproteination, demineralization, and deacetylation. The chitosan-lithium membrane was prepared by casting method with the addition of 15% lithium salt. The value of the degree of deacetylation in crab shell chitosan is around 39.39%. Based on the characterization, the chitosan-lithium SPE membrane conductivity value is 1.11 x 10-7 S.cm^{-1} with a tensile strength value of 27.07 MPa.

ABSTRAK

Upaya dalam mencegah terjadinya krisis energi secara global yakni dengan cara menggunakan energi yang terbarukan dan ramah lingkungan. Baterai merupakan salah satu alternatif untuk menggantikan energi yang tidak terbarukan. Akan tetapi, dalam baterai terdapat komponen elektrolit yang ketika selesai digunakan baterai tersebut akan menimbulkan limbah yang menyebabkan tanah menjadi beracun. Penelitian ini bertujuan untuk mengetahui karakterisasi membran elektrolit padat kitosan, cangkang rajungan. Karakterisasi yang dilakukan yaitu karakterisasi kitosan cangkang rajungan menggunakan spektrofotometer FTIR, analisa konduktivitas ion membran elektrolit padat menggunakan LCR Meter, dan uji tarik menggunakan Tensilon. Metode yang digunakan dalam penelitian ini adalah eksperimen. Isolasi kitin dan kitosan cangkang rajungan dilakukan dengan beberapa tahap yaitu deproteinasi, demineralisasi, dan deasetilasi. Membran kitosan-litium dibuat dengan metode *casting* dengan penambahan garam litium sebesar 15%. Nilai derajat deasetilasi pada kitosan cangkang rajungan sekitar 39,39%. Berdasarkan karakterisasi, nilai konduktivitas membran PEP kitosan-litium sebesar 1,11 x 10^{-7} S.cm^{-1} dengan nilai kuat tarik sebesar 27,07 MPa.

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✉Correspondence Author:
muayanahh34@gmail.com
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INTRODUCTION

The biggest challenge facing humans today is the energy crisis. This is due to the depletion of natural energy reserves in the form of fossil fuels that are used continuously. Efforts to prevent a global energy crisis are by using renewable and environmentally friendly energy. Batteries are one alternative to replace non-renewable energy. Batteries are electric cells, in which an electrochemical process takes place that can be reversed (reversible) and has high efficiency (Afif & Pratiwi, 2015). The reversible electrochemical process in question is the process of converting chemicals into a discharge process (electrical power) in the battery, and vice versa from the discharge process to chemical energy, recharging is carried out by regenerating the electrodes used, namely by passing an electric current in the opposite direction in the cell (Aditya, 2016). However, in batteries, there are electrolyte components that when used, the battery will produce waste that causes the soil to become toxic. In batteries, electrolytes function as conductors of lithium ions from the anode to the cathode or vice versa (Perdana, 2020). The use of liquid electrolytes still has several disadvantages including the risk of leakage, flammability when exposed to sparks, and toxicity (Gonggo et al., 2017). In contrast, solid electrolytes have advantages including being free from leakage, easy to refill, free from self-discharge, safer to use, easy to apply (Mufida et al., 2015), non-volatile, non-flammable, and have good chemical stability (Arlita, 2017). In addition, the ideal solid electrolyte membrane must have high thermal stability, high flexibility, relatively low cost, and abundant availability of materials in nature (Pratiwi, 2018). Currently, the polymer widely used in batteries is polyethylene oxide (PEO) as a matrix with inorganic salts dissolved in it. However, the high level of crystallinity of PEO limits its use in batteries and can only be used at temperatures above the melting point of the crystalline phase, which is around 60^{o}C. Therefore, it is necessary to find alternative polymers to replace PEO (Gonggo et al., 2017). Alternative polymers that can be used are environmentally friendly natural polymers. Based on this perspective, the use of biodegradable polymers such as chitosan is the right answer. Chitosan is a type of natural polymer that has the potential as a solid electrolyte material. Chitosan has several specific properties, both biological and chemical, including linear polyamines, bioactive properties, biocompatible, amine groups, reactive hydroxyls, chelators for transition metal ions, and antibacterial. Chitosan is very abundant in nature and can be renewed and has good properties such as being non-toxic, can decompose naturally in a relatively short time (biodegradable), can be adjusted (biocompatibility), and has high absorption (Multazam, 2014). Chitosan is a natural biopolymer derived from the chitin deacetylation process. Chitin is the main organic material found in crustaceans, six-legged insects, fungi, mollusks, and arthropods (Pratiwi, 2018). Crabs are animals whose shells can be used to make chitosan. Crabs are one type of marine biota that is widely favored by the public because they have good nutritional content for health (Dali et al., 2016). Crabs are one of the mainstay export commodities for fisheries in Indonesia (Al Faruqi, 2020). In the industrial sector, crabs are only taken for their meat, while their shells are simply thrown into the

environment, which can cause environmental problems, such as odor pollution, transmit disease, and disrupt public comfort (Supratman & Umroh, 2018). Shrimp shell waste and crab shells have the potential to be used as a mixing material or feed supplement, fertilizer, chitin, chitosan, food products, and others (Azizi et al., 2020). Crab shell waste also has chemical content that can be utilized, including 30-40% protein, 30-50% minerals, and 20-30% chitin (Natalia et al., 2021). Therefore, crab shell waste needs to be processed into high economic value and environmentally friendly materials because the waste contains chemical compounds that can be used as more useful materials, namely chitosan.

METHOD

Tools

The equipment used in this study includes common glassware. Samples and materials are weighed with an analytical balance of the $KERN_{ABJ-NM/ABSN}$ brand. For stirring, a Hotplate Stirrer of the Thermo Scientific brand. Memmert Experts in Thermostatics brand oven was used to dry the samples. To determine the functional groups contained in chitin using a Bruker Alpha type FTIR Spectrophotometer. To measure the conductivity of chitosan membranes and chitosan-lithium membranes using an LCR Meter of the Hi-tester Hioki 3532-50 brand (with a range of 42Hz - 1MHz). Tensile testing using a Tensilon RTG-1310 tool.

Materials

The materials used in this study were crab shells, sodium hydroxide (NaOH, Technical), hydrochloric acid (HCl, Technical), acetic acid (CH_3COOH, Pro Analyst), lithium acetate (CH_3COOLi, Pro Analyst), and distilled water.

Research Procedures

Isolation of Chitin and Chitosan from Crab Shells

This stage begins with Sample Preparation. The crab shells are washed clean to remove any remaining meat, dried, and ground. Then sieved using a 60 mesh sieve (Bahri et al., 2015), then dried again using an oven at a temperature of 60°C for 4 hours (Alawiyah & Hadi, 2016). The second stage is Deproteination. The crab shells that had been ground, were then dissolved in 4% NaOH with a ratio of crab shells: 4% NaOH 1:10 (w/v) at a temperature of 60-65°C for 2 hours while stirring. Then neutralized with distilled water, filtered, and dried at a temperature of 60°C for 15-20 hours (Sukma et al., 2014), (Alawiyah & Hadi, 2016). The third stage is Demineralization. The deproteinized crab shells were dissolved in 1M HCl with a ratio of crab shells: HCl 1:15 (w/v) at room temperature for 1 hour while stirring until CO_2 gas was no longer formed. Then neutralized with distilled water, filtered, and dried at 60°C for 15-20 hours (Mashuni et al., 2021). The last stage is Deacetylation. The demineralized chitin was dissolved in 50% NaOH with a ratio of crab shells:50% NaOH 1:20 (w/v) at 80°C - 100°C for 3 hours. Then neutralized with distilled water, filtered, and dried at 60°C for 15-20 hours (Azizi et al., 2020).

Synthesis of Chitosan-lithium Solid Electrolyte Membrane

0.59 grams of crab shell chitosan and 0.09 grams of lithium acetate were put into a 250 mL beaker, then the chitosan was dissolved in 30 mL of 2% acetic acid solution (v/v). The solution was poured into an acrylic mold, then the solvent was evaporated at room temperature until a thin film was formed (Multazam, 2014; & Pratiwi, 2018).

RESULT AND DISCUSSION

Chitosan Isolation from Crab Shell

Chitin isolation is carried out through two stages, namely deproteination and demineralization. On the other hand, chitosan is obtained through one stage namely deacetylation. The deproteination stage aims to remove the protein in the crab shell so that the bond between chitin and protein can be released or broken so that a crab shell that is free of protein is obtained (Multazam, 2014). This demineralization process aims to remove mineral content such as calcium carbonate ($CaCO_3$) and calcium phosphate ($Ca_3(PO_4)_2$) contained in the crab shell in small amounts (Agustina, 2015). Chitin obtained from the isolation process has a yield weight of 37.34%. Furthermore, the deacetylation stage is carried out by using a high-concentration base solvent. The deacetylation stage aims to break the covalent bond between the acetyl and the acetamide groups of chitin into a deacetylated amine group (Bahri et al., 2015). The yield weight of chitosan obtained from the crab shell is 21.14%.

a) b)

Figure 1. a) Chitosan powder from crab shells, b) chitosan membrane

Chitosan Characterization Using FTIR Spectroscopy

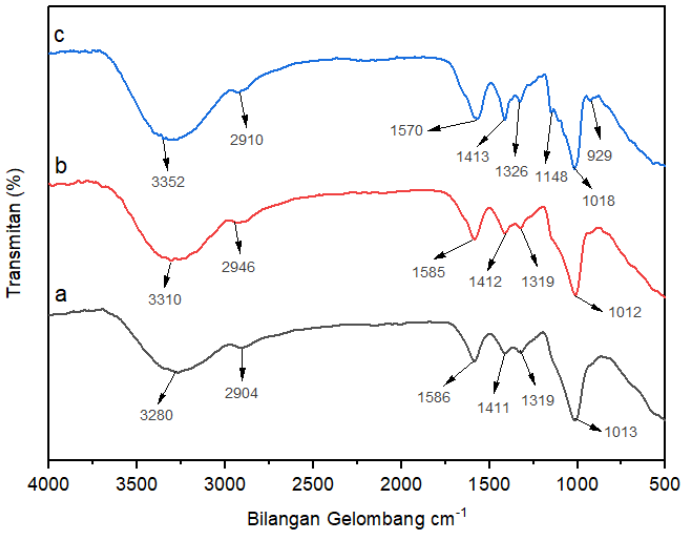

Figure 2 FTIR spectrum of a) chitosan, b) chitosan membrane, c) chitosan-lithium membrane

Based on the FTIR spectrum of the crab shell chitosan compound in Figure 2, the black line is observed to have absorption at 3280.8 cm^{-1} to 3345.5 cm^{-1} providing information on an -OH group (hydroxide) presence. The absorption wave number of 2904.5 cm^{-1} to 2965.4 cm^{-1} provides information about -NH_2- (amine group)

presence. The absorption intensity of the wave number of 1586.2 cm^{-1} to 1595.3 cm^{-1} provides information on the presence of a -C=O group (carbonyl group). According to Wulandari et al., (2020) stated that the characteristics of chitosan lie in the amide group and hydroxyl group. The location of the typical absorption of the amide and hydroxyl groups can be seen in Table 2 below.

Table 2. Comparison of functional groups of chitosan from research results with literature

Functional Groups	Wave Number (cm^{-1})	
	Literatur (Rusnaenah, 2020)	Research
-OH *(bending)*	3258	3280
-CH *(symmetric stretching)*	2875	2904
$-NH_2$ *(stretching)*	1553	1586
-CN *(stretching)*	1416	1411
$-CH_3$ *(symmetric stretching)*	1319	1319
-C-OH *(stretching)*	1065	1013

Degree of Deacetylation

The degree of deacetylation (DD) is obtained from the loss of acetyl groups in the acetamide group of chitin or free amino groups obtained after the deacetylation process (Wahyuni et al., 2020). Based on the calculation of the degree of deacetylation from the FTIR spectrum using the baseline method, the degree of deacetylation value obtained in the crab shell chitosan sample was around 39.39%. The low degree of deacetylation of chitosan is caused by several factors including the duration of stirring and the temperature used (Agustina, et al., 2015). The degree of deacetylation of chitosan ranges from 56-99%, while on an industrial scale for food production, the degree of deacetylation used ranges from 70% and above. The lowest degree of deacetylation in chitosan ranges from 40-60%, while commercial chitosan generally has a degree of deacetylation value of 70-90% (Harianingsih et al., 2019). Reaction time is a factor that affects the success of the deacetylation process, where the longer the reaction time, the more perfect the deacetylation process can be. This is because the addition reaction of hydroxy NaOH requires sufficient time to release the eliminated acetyl group (Luthfiyana et al., 2022). The use of long periods with high temperatures in the deacetylation process will cause a decrease in yield and molecular weight but can increase the degree of deacetylation (Wahyuni et al., 2020) so the quality of chitosan also increases.

Optimization of the temperature used in the deacetylation process ranges from 80^0C to 130^0C with a reaction time of 1 to 3 hours. According to Citrowati (2017), it shows that deacetylation using a combination treatment of 55% NaOH and a temperature of 100°C is a fairly good treatment to produce chitosan from hatchet shells. Based on the results of research conducted by Wahyuni, et al., (2016) showed that the degree of deacetylation increased along with the longer time of the deacetylation process, namely at 60 minutes it produced a degree of deacetylation of 55.6%, 90 minutes produced a degree of deacetylation of 62.4%, 120 minutes produced a degree of deacetylation of 70.3%, and 150 minutes produced a degree of deacetylation of 84.3%. Thus, it can be concluded that the longer the time used in the deacetylation process of chitin from snail shells, the degree of deacetylation can increase.

Ion Conductivity Analysis of Lithium Chitosan Membrane

Ion conductivity is one of the important parameters of a solid electrolyte

membrane (Safitri & Supu, 2020). This analysis aims to determine the resistance that occurs in chitosan and lithium chitosan membranes (Multazam, 2014). Figure 3 is a Cole-cole plot showing that the chitosan membrane and lithium chitosan membrane. The semicircle at high frequencies can be associated with a parallel combination of large resistance and large capacitance. Based on Figure 3, which forms an almost semicircular pattern, namely the chitosan membrane. With the addition of lithium salt, the semicircular shape becomes increasingly disappearing as shown in Figure 3 for the lithium chitosan membrane (b).

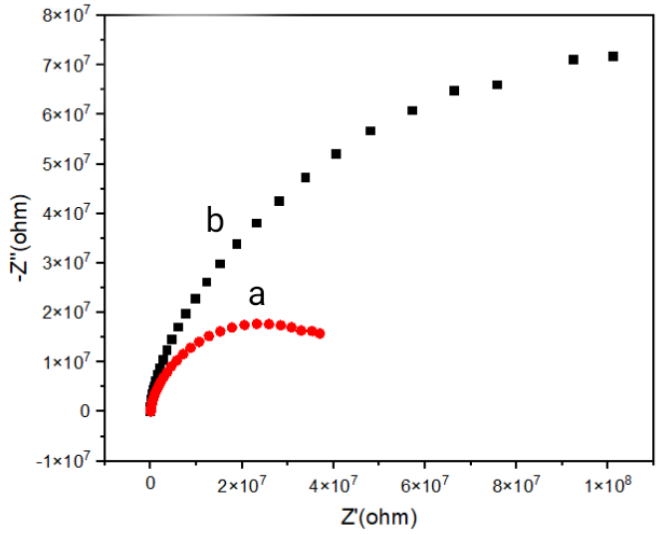

Figure 3 Cole-cole plots of a) chitosan membrane and b) lithium chitosan.

Table 2. Ion conductivity values of PEP membranes

No	PEP membranes	Ion conductivity
1	Chitosan	9,01 x 10^{-8} S cm^{-1}
2	Chitosan+lithium	1,11 x 10^{-7} S cm^{-1}

Based on the ion conductivity analysis in Table 2, chitosan and chitosan-lithium membranes were carried out using the Hioki 3532-50 LCR Hi-tester (range of 42Hz - 1MHz). In the chitosan and chitosan-lithium samples, the conductivity values were 9.01 x 10^{-8} S.cm^{-1} and 1.11 x 10^{-7} S.cm^{-1}, respectively. The ionic conductivity of solid electrolyte polymers that can be used in batteries is 10^{-9} to 10^{-3} S/cm. The addition of lithium acetate salt aims to increase the conductivity value of the material so that there is an increase in the ion conductivity due to the increasing number of ions and the movement of the ions contained in the electrolyte membrane (Safitri & Supu, 2020). Lithium-ion battery performance is based on ionic conductivity. In this case, Li^+ ions generally have their conductivity influenced by two things, namely the concentration of ions as charge carriers and the mobility of these ions. The greater the number of Li^+ ions in the polymer, under the same ion mobility conditions, the conductivity also tends to increase (Hidayat et al., 2016). According to research conducted by Darmawan, et al., (2023) that the ionic conductivity value of carboxyl-methyl cellulose (CMC)-based polymer electrolyte membranes increases with the addition of lithium acetate ($LiCH_3COO$). Ionic conductivity increases with an increasing weight percentage of lithium acetate salt ($LiCH_3COO$) and the optimum is obtained at 30% wt $LiCH_3COO$ of 2.47 x 10^{-5} S.cm^{-1}. In general, ionic

conductivity is influenced by the mobility and diffusion of Li ions which are getting faster. The degree of deacetylation can affect the ion conductivity in batteries. The greater the degree of deacetylation produced, the better the ion conductivity of the membrane. According to research conducted by Pratiwi (2018) using chitosan with a degree of deacetylation of 98% obtained the highest conductivity from the membrane with a ratio of chitosan: LiOH (85:15)% w/w, namely 1.301 x 10^{-1} S.cm^{-1}. Then in a study conducted by Putri, et al., (2021) using a degree of deacetylation of 87.28% and having the highest conductivity obtained from a membrane with a composition of 20% $LiClO_4$, namely 10^{-4} S.cm^{-1}. In this study, chitosan with a degree of deacetylation of 39.39% was used and obtained the conductivity of the chitosan membrane without the addition of Li^+ is 9.01 x 10^{-8} S.cm^{-1}, while the conductivity of the chitosan membrane with the addition of Li^+ was 1.11 x 10^{-7} S.cm^{-1}. The increase in conductivity in the membrane is assumed to be due to the addition of Li^+ ions which increase the number of ions and the mobility of ions in the membrane. Meanwhile, according to Nurhandini (2021), the increase in conductivity value is also caused by the interaction between Li^+ ions and the NH_2 groups of chitosan which act as hoping sites that move Li^+ ions in the polymer electrolyte membrane mixture.

Tensile Strength Test

The tensile strength test is one of the tests used as a parameter in testing the quality of a membrane. Tensile strength is the maximum force held by the membrane until the membrane is torn or broken with a certain surface area. In addition, tensile strength also plays a major role in the mechanical properties of the membrane.

Table 3. Tensile strength values of PEP membranes

No	PEP membranes	Tensile strength (Mpa)
1	Chitosan	26,42
2	Chitosan+lithium	27,07

Based on Table 3, the tensile strength value of the PEP chitosan membrane is 26.42 Mpa and chitosan-lithium is 27.07 Mpa. The tensile strength of the chitosan-lithium membrane is greater than that of the chitosan membrane without lithium. The addition of lithium salt affects the tensile strength value of a membrane. This is in accordance with research conducted by Pawestri & Marfuatun (2016) which states that the more lithium salt is added to cellulose acetate, the tensile strength value tends to be greater. The tensile strength values of the two PEP membranes meet the SNI 7818: 2014 standard, which is above 24.7 Mpa so the chitosan membrane from crab shells can be used as a separator in batteries.

CONCLUSION

Solid electrolyte polymer membranes based on chitosan from crab shells have been successfully made. The membrane was made using the casting method with the addition of lithium acetate salt. The deacetylation degree value of chitosan from crab shells was not sufficient to meet SNI, which was around 39.39%. However, the conductivity value of the chitosan-lithium PEP membrane was 1.11 x 10^{-7} S.cm^{-1} so that it could meet the standards for application in batteries. The tensile strength value of the PEP membrane was 27.07 MPa.

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