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ORIGINAL ARTICLE

Synthesis and Characterization of Sodium Carboxymethylcellulose from *Sansevieria trifasciata* as an Alternative Raw Material for Capsule Shell

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ABSTRACT

Objectives: Capsules are pharmaceutical preparations that are enclosed in hard or soft capsule shells made of gelatin. Because gelatin is typically generated from non-halal materials, there is a need for alternative raw materials for making capsule shells, one of which is carboxymethyl cellulose (Na-CMC), which is synthesized from cellulose.

Materials and Methods: This study aims to find alternative raw materials for the manufacture of capsule shells from cellulose-containing *Sansevieria trifasciata* leaves. Then, the isolated cellulose was treated with sodium Na-CMC using the alkalization and carboxymethylation methods.

Results: The cellulose that was produced fulfills the requirements for continuing the synthesis of Na-CMC. The yield of Na-CMC produced was 83.83%, with a pH of 6; dispersed in water and insoluble in ethanol and ether; a degree of substitution of 0.83; and a water content of 12%. The Fourier Transform Infrared Spectroscopy study results show the presence of functional groups such as O-H at 3332 cm^{-1}, C-H at 2930 cm^{-1}, C=C at 2050 cm^{-1}, O-Na at 1588.82 cm^{-1}, and C-O at 1020.16 cm^{-1}. The resulting capsule shell has the following properties: transparency, a slightly cream color, a somewhat firm texture, no odor, and a moisture content of 20%.

Conclusion: As a result, it is possible to establish that Na-CMC from the *S. trifasciata* leaves can be used as a raw material for capsule shells.

Keywords: Capsule shell, Na-CMC, *Sansevieria trifasciata*

INTRODUCTION

Capsules are solid preparations consisting of one or more drugs or other inert materials that are enclosed in a soluble hard or soft shell. Hard capsules are generally made of gelatin but can also be made of starch or other suitable materials.[1] Commercially available hard-shell capsules are generally made from gelatin, which is produced from the bone and skin of cows, pigs, or buffalo. Gelatin hard-shell capsules were introduced in 1931 by Arthur Cotton. One of the advantages of hard-shell capsules is that they can deliver both solid and liquid medicines. This indicates that the presence of hard-shell capsules is important as a drug delivery system (DDS).[2,3]

According to several sources, it is known that the most widely produced source of gelatin is derived from pork skin, namely 44.5% (136,000 tons), the second from cow skin, 27.6% (84,000 tons), the third from cow bones, 26.6% (81,000 tons), and the rest comes from another 1.3% (4,000 tons). The use of pork skin as the main source of gelatin manufacture started in 1930 in Europe. This is due to the limited availability of raw materials and high prices. In this case, pigs are abundant, while the

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consumers, Indonesian people, are mostly Muslim, and pigs are among the animals that are forbidden by Islam. Because of that, many studies have been carried out to find alternative materials, such as materials that are obtained from polysaccharides or natural polymers.[4]

Polysaccharides play a crucial role in plant life processes because they can form glycoconjugates with proteins and lipids, resulting in biological macromolecules. The primary wall's principal polysaccharides are cellulose, hemicellulose, and pectin. Cellulose is one of the most abundant natural biopolymers, found in the cells of many plants and algae.[5] Cellulose has three reactive hydroxyl groups that create inter- and intramolecular hydrogen bonds per anhydroglucose repeat unit. This second bond has a significant impact on cellulose's chemical reactivity and solubility.[6]

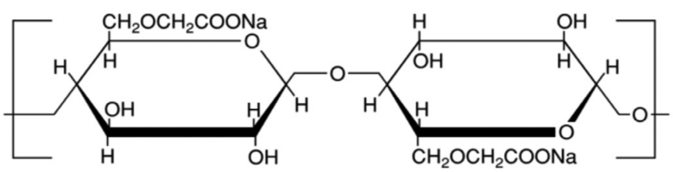

Figure 1. Sodium carboxymethyl cellulose (Na-CMC)[7]

Sodium carboxymethyl cellulose (Na-CMC) is a water-soluble organic polymer, which is produced by partial substitution of 2, 3, and 6 hydroxymethyl groups of cellulose by hydrophobic carboxymethyl groups. The chemical structure of Na-CMC, as illustrated in Figure 1, provides further insight into its composition and properties, highlighting the specific modifications that enable its solubility and functionality in various applications.

Na-CMC was chosen as an alternative to gelatin in the manufacture of capsule shells because it has a lower water content value than gelatin, which gives it a longer shelf life and makes it more resistant to damage due to physical, chemical, and microorganism factors.[8] It was demonstrated that the addition of CMC binder at 2.00% and 3.00% improved pellet water stability.[9]

Plants with a high cellulose content can be used to produce Na-CMC. Some plants, such as cassava peel, are used for cellulose isolation to serve as raw materials for Na-CMC, which contains 80-85% cellulose.[10] Meanwhile, the literature indicates that Pontederia crassipes plants contain 66.87% cellulose,[11] Elaeis guineensis fibers contain 59.6% cellulose,[12] and Imperata cylindrical stems contain 44.28% cellulose.[13]

Sansevieria trifasciata is a single-seeded or monocotyledonous plant that is one of the most popular ornamental plants, and it can be used as an alternative source of cellulose.[14] It contains chemicals such as flavonoids, saponins, and tannins.[15] Among other advantages, *S. trifasciata* survives extreme conditions of temperature and light, and it is resistant to various types of pollutants. The results of research by the United States Space Agency have proven that the mother-in-law's tongue plant can reduce sick-building syndrome naturally. Room conditions are affected by pollutants due to increased concentrations of carbon dioxide gas, cigarette smoke containing nicotine, and the use of air conditioners.[16]

Based on the above description of the cellulose content of *S. trifasciata*, the researchers wanted to use the mother-in-law's tongue as an alternative raw material for capsule shells derived from cellulose.

MATERIALS AND METHODS

Tools and materials

Tools

The equipment used in this research includes Fourier Transform InfraRed (FTIR) (Perkin Elmer Frontier C90704 Version 10.6.1), Magnetic Stirrer (DLAB MS7-H550-5), Instrument Moisture Content Analyzer, oven (Memmert), hot plate (IKA HS7-C-MAG), Grinder (Miyako), 40 mesh sieve, scale (Fujika), mortar and stamper, pH paper, plastic container, filter, tea-bag, and glassware (Iwaki).

Materials

The materials used in this study were *S. trifasciata* (5 kg wet), sodium hydroxide (NaOH) (Merck), nitric acid (HNO_3) (Merck), hydrogen peroxide (H_2O_2) (Brataco), hydrochloric acid (HCl) (Merck), acetic acid 70% (Merck), isopropyl (Merck), glycerin (Brataco), sodium chloroacetate (Brand), methanol 70% (Merck), methanol absolute (Merck), ethanol (Merck), ether (Merck), aquadest (Smart Lab).

Works procedure

Sampling

The study of *S. trifasciata* leaves was carried out in Jorong Kubang Bunguk, Situjuah Batur, Situjuah Limo Nagari District, Lima Puluh Kota Regency, West Sumatra, Indonesia.

Identification of S. trifasciata

Identification of the *S. trifasciata* was carried out at the Herbarium of Biology Department of Andalas University.

Sample preparation

S. trifasciata was weighed to obtain the wet weight. Then it was washed and dried. The drying process is carried out with the help of indirect sunlight. After drying, it was reduced in size with a grinder and sieved using a 100 mesh sieve. The *S. trifasciata* powder was weighed to determine the dry weight.

Cellulose isolation

50 g of *S. trifasciata* powder was hydrolyzed using 400 mL of 4% HNO_3 at 80 ˚C for 2 hours on a magnetic stirrer. The sample was then filtered and bleached with 200 mL of 15% H_2O_2 at 80 ˚C for 1 hour. Then delignification was carried out using 200 mL of 2N NaOH at 80 ˚C for 1 hour,[17] and the mixture was then filtered and bleached using 200 mL of 15% H_2O_2 at 80 ˚C for 1 hour. Then, the samples were placed in the oven at 70 ˚C for 24 hours.[18]

Synthesis of Na-CMC

A total of 6 grams of *S. trifasciata* cellulose was alkaliized by adding 100 mL of isopropyl alcohol and 20 mL of 20% NaOH in a beaker and heated at 30 ˚C for 90 minutes. Six grams of sodium chloroacetate were added to the mixture, and then stirred for 3.5 hours at 70 ˚C. The mixture was filtered and neutralized with 50 mL of acetic acid, followed by rinsing with methanol, filtered again, and then oven-dried at 50 ˚C.[19]

Characterization of Na-CMC

Organoleptic

Examination organoleptic examination was carried out under the Indonesian Pharmacopoeia 3rd edition, which included an examination of shape, smell, taste, and color. The examination involved direct observation using multiple senses.

Examination of pH

One gram of Na-CMC was dissolved in 100 mL of distilled water, and then the mixture was heated at a temperature of 60 ˚C and stirred until dissolved. The heated mixture is cooled at room temperature, and then the pH is determined with a pH meter.[20]

The examination of the solubility of

Na-CMC obtained was observed for its solubility in water, ethanol, and ether.

Inspection of moisture content

The determination of water content is carried out using the gravimetric method. In the gravimetric procedure, weigh 1 gram of sample in a dish whose weight is known, and heat it in an oven at 105 ˚C for 5 hours. After that, the cup containing the sample is put in a desiccator for 15 minutes and then weighed. Repeat the weighing every 1 hour and 30 minutes until the weight remains constant.

Swelling ratio test

Measurement of the swelling of Na-CMC in water was conducted to calculate the absorption capacity expressed in the swelling ratio. Measurements were carried out using distilled water at a temperature of 22 ˚C with a tea bag as part of the process. Before immersion, the weight of the tea bag (Wo) and dry Na-CMC (W1) were measured. Then, it was immersed in distilled water, and every 10 minutes, the weight was measured (W1). Before weighing, the tea bags were hung for 15 minutes to remove the remaining water that was not absorbed by the Na-CMC.[21]

Determination of the degree of substitution (DS)

Nought point five grams of Na-CMC was added to 10 mL of 2 N HNO_3 solution, and allowed to stand for 2 hours; then filtered, and the residue was baked in an oven at 60 ˚C to dry. Samples that have been in the oven are combined with 100 mL of distilled water and 25 mL of 0.3 N NaOH solution, then titrated with 0.3 N HCl.[22]

FTIR

FTIR analysis characteristics were used to determine the functional groups of Na-CMC. The first step of this analysis is to make a pellet by mixing Na-CMC with KBr. The fibers were ground with KBr until they became homogeneous, forming a fine powder. After being homogeneous, a certain amount of the powder is taken and then inserted into the pellet-making tool. The pellet that has been formed is put into an infrared spectrometer. After all the spectra were formed, they were analyzed and matched with data from the literature.[22]

Preparation of a of capsule

Capsule shells were made by dissolving 0.78 grams of Na-CMC in aqua dest, then adding 5 grams of carrageenan, which had also been dissolved in aqua dest. Then, the capsule shell was printed using a manual mold. The molded capsule shell is allowed to dry, then removed from the mold and trimmed.

Evaluation of capsule shell

Organoleptic

Organoleptic examination was carried out using the five senses, which included examination of shape, smell, taste, and color.

Water content test

One gram of capsule shell is weighed and put in a tared container. Dry at 105 ˚C for 5 hours. Continue drying and weighing at intervals of 1 hour until the weight remains constant.[21]

RESULTS

The isolation of cellulose from *S. trifasciata* was aimed at obtaining an active compound for the synthesis of Na-CMC. The characterization was conducted based on standardization parameters, as shown in Table 1. Table 2 presents the yield

Table 1. Characterization of Na-CMC *Sansevieria trifasciata*

Parameters	Terms FI	Na-CMC
Form	Powder	Powder
Color	White or white ivory ivory	White
Odor	Odorless or almost odorless	Slightly smelly acid
Moisture content	<10%	12%

Na-CMC: Carboxymethyl cellulose

Table 2. Yield and moisture content of *Sansevieria trifasciata*

Powder of *S. trifasciata*	Cellulose	Yield	Water content
50 grams	20 grams	40%	7 %

Table 3. Yield and pH

Parameters	
Yield	pH
88.83%	6

Table 4. Swelling test results

Time	10 minutes	20 minutes	30 minutes	40 minutes	50 minutes	60 minutes
Weight	6.38 mg	7.32 mg	7.29 mg	7.69 mg	8.13 mg	8.33 mg

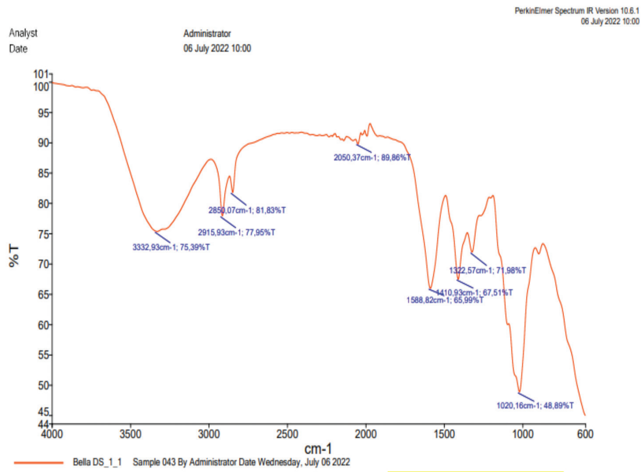

Figure 2. A spectrum of Na-CMC FTIR analysis *Sansevieria trifasciata*

Na-CMC: Carboxymethyl cellulose, FTIR: Fourier Transform InfraRed

and moisture content of cellulose, while the pH after the alkalization process is shown in Table 3. The parameter used to evaluate and calculate the adsorption capacity of Na-CMC was the swelling ratio (Table 4). The analysis of functional groups in Na-CMC was performed using IR spectroscopy that showed at Figure 2.

DISCUSSION

Cellulose isolation is the separation of cellulose and non-cellulose components such as lignin and hemicellulose.[22] The bleaching process aims to whiten the cellulose that is still brown and, at the same time, remove the remaining lignin.[18]

Alkalization is a process using NaOH 20% NaOH, aiming to activate the OH groups on the cellulose molecule. This process facilitates the development of cellulose and substitution reactions involving the sodium chloroacetate reagent. The expansion of cellulose causes the breaking of hydrogen bonds in the cellulose structure. In the alkalization stage, a substitution reaction occurs between the hydroxyl group and NaOH, which produces cellulose alkali in the form of a light brown, viscous solution. At this stage, an organic solvent is used as an inert reaction medium, where the alkalization and carboxymethylation processes can react simultaneously and can also increase the DS.[23]

The quantity of reagents employed in the carboxymethylation process has a significant impact on the quality of the Na-CMC produced.[22]

The reactions that occur in the alkalization and carboxymethylation processes are as follows:

$$\text{RcellulosaOH} + \text{NaOH} + \text{ClCH2COONa} \rightarrow \text{RcellulosaOCH}_2\text{COONa} + \text{NaCl} + \text{H}_2\text{O}$$

In addition to the reaction for the formation of Na-CMC, there is also a reaction between NaOH and sodium chloroacetate to form side products, sodium glycolate, and sodium chloride, based on the reaction as follows:

$$\text{NaOH} + \text{ClCH}_2\text{COONa} \rightarrow \text{HO-CH}_2\text{COONa} + \text{NaCl}$$

This reaction occurs between NaOH and ether, and the conditions of this reaction must be optimized to minimize the by-products formed.[18] Because of its strong hydrophilicity, the resultant Na-CMC water content still contains many water molecules, which may be determined by gravimetric measurements and validated by mass structure analyses.[24]

Na-CMC and glycerin can affect physical stability, as measured using a viscometer, and already comply with the Indonesian National Standard number 12-3524-1995. However, if the concentration of glycerin is reduced further, it will not significantly affect the thickness of the paste. However, if the concentration of Na-CMC is too high, it will result in poor spreadability of the toothpaste gel, which can affect other parameters such as pH and organoleptic properties.[25] The average amount of hydroxyl groups in the cellulose structure substituted by carboxymethyl or sodium carboxymethyl groups at carbons 2, 3, and 6 is referred to as the DS,[26] and is one of the parameters in determining the quality of Na-CMC. The higher the DS produced, the better the quality of Na-CMC because its solubility in water is greater. Meanwhile, the more by-products produced, the lower the quality of Na-CMC.[22] The DS obtained depends on the time and raw materials used in the synthesis process, but the value of the DS generally varies between 0.6 and 0.95[27] and ranges from 0.4 to 1.5.[24] After removing the water, the swelling test was performed until the equilibrium development value was attained, which can be estimated using the formula:[28]

$$\text{Swelling ratio} = \frac{wt - wo - w1}{w1}$$

The swelling Na-CMC can be affected by the hydrophilicity of the carboxylate groups in the hydrogel structure. Because of the limited space available for free water to enter the hydrogel network, the swelling capacity of Na-CMC is diminished. The swelling process can be initiated by the passage of water molecules through the matrix, the relaxing of polymer chains by hydration, and the expansion of the polymer network following relaxation. When Na-CMC is submerged in deionized water, the hydrophilic polymer chains generate osmotic pressure inside the hydrogel, causing the hydrogel matrix to form.[28] Functional group analysis using FTIR was carried out to prove the presence of specific functional groups in Na-CMC. Figure 2 shows a peak at a wavelength of 3332.93 cm-1, which indicates the presence of OH, so hydrogen bonding groups are formed between hydrogen atoms in one hydroxyl group in one glucose monomer and oxygen atoms from another hydroxyl group in a glucose monomer in cellulose polymer chains. The O-Na group was seen at a wavelength of 1410.93 cm-1, some clusters at a wavelength of 2915.93 cm-1, and the presence of a peak at a wavelength of 2850.07 cm-1 indicated a CH strain. At a wavelength of 1020.16 cm-1, the CO strain and the structure of the cellulose component are shown; at a wavelength of 1588.82 cm-1, the C=C group symmetrical stretch of the aromatic ring of lignin is shown.[24] Hendradi et al.'s[29] FTIR analysis revealed the production of covalent imine linkages between chitosan and glutaraldehyde. Furthermore, adding glutaraldehyde or genipin as crosslinkers reduced the mechanical strength of the implant. The humidity of the capsule normally fluctuates between 13 and 16%. If it is stored in a relatively high-humidity environment, the humidity rises, and the stiff capsule shell becomes warped.[30] The development of hard capsules to replace gelatin in DDSs is moving forward. Hard capsules are manufactured in six basic steps: dipping, spinning, drying, stripping, trimming, and joining.[2]

CONCLUSION

After conducting basic research to identify substitute ingredients for capsule production, it was determined that Na-CMC from *S. trifasciata* could be used as a raw material. Its characteristics include a powder-like shape, a bone-white color, a strong smell, a pH of 6, a water content of 12%, and a substitution degree of 0.83. This Na-CMC from *S. trifasciata* needs to be thoroughly developed and tested by further researchers before it can be manufactured and sold as a formulation material for capsule shells, such as average molecular weight must be determined for the cellulose obtained, and compare the capsule from gelatin cost, production method, equipment used, materials, etc.

Ethics

Ethics Committee Approval: This study did not involve human subjects, animal experimentation, or the collection of personal and identifiable data.

Informed Consent: Patient consent was not applicable.

Footnotes

Authorship Contributions

Concept: A.R., Design: A.R., B.V.O., I.A.S., Data Collection or Processing: B.V.O., Analysis or Interpretation: A.R., B.V.O., Literature Search: A.R., Writing: A.R., B.V.O., I.A.S.

Conflict of Interest: The authors declare no conflicts of interest.

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