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Characteristics of Chitosan-Potato Starch Biocomposite for Wound Dressing Applications with the Addition of Calcium Carbonate ($CaCO_3$) Filler

*Corresponding author: suryati@unimal.ac.id

Suryati[1]*, Sulhatun[2], Muhammad[3], Lukman Hakim[4], Riska Dwi Safira[5]
[123456] Universitas Malikussaleh, Aceh, Indonesia
meriatna@unimal.ac.id
sulhatun@unimal.ac.id
muhammad@unimal.ac.id
lukmanhakim@unimal.ac.id
riska.190140122@mhs.unimal.ac.id

ABSTRACT
An ideal wound dressing should be able to create moist conditions, control exudate, maintain body stability, and protect against infection. In addition, the need to reduce environmental pollution and the carbon footprint of synthetic materials encourages the development of environmentally friendly wound dressings. This study aims to examine the use of potato starch as a base material for the manufacture of biocomposites for wound dressing applications and analyze the effect of calcium carbonate (CaCO3) filler addition on the physical and chemical properties of biocomposites. The method used in this research consists of raw material preparation, biocomposite processing stage and testing stage. An investigation has been made to predict the effects of fillers of $CaCO_3$ with different concentration from 4 to 9 percents on the properties of Chitosan and Potato Starch Bio-composite for wound dressing applications. The procedure was to prepare the chitosan and starch preparation with equal proportion, fillers application on hydrogel, drying and characterization. The characterizations on synthesized bio-composite were its absorption percentage, swelling test and presented functional groups test. The stages of this research consist of raw material preparation, biocomposite processing, and testing phases. In the swelling test, the highest percentage swelling value was obtained with the potato starch-chitosan composition variation (w/v) of (40:60) with 6% $CaCO_3$, which amounted to 249.54%. In the absorption test, the biocomposite absorption percentage of the potato starch-chitosan composition showed the best results, specifically at the variation (w/v) of (40:60) with 6% $CaCO_3$, amounting to 72.09%. The results of functional group analysis (FTIR) contained in the biocomposite show functional groups O-H, C-H, and C-O, indicating that the wound dressing tends to be hydrophilic and these groups suggest that the wound dressing is easily degradable."

Keywords: Biocomposite, Calcium Carbonate, Chitosan, Potato Starch, and Wound Dressing

1. INTRODUCTION

The use of wound dressings is one alternative in the initial medical treatment of wounds (Oktaviani et al., 2019). As an ideal wound dressing, it must be able to create a moist atmosphere, control excess exudate, and maintain a stable body condition, and cannot be passed by microorganisms. To reduce environmental pollution from synthetic wound dressings, it is necessary to develop new materials from renewable sources (Kim et al., 2021).

2. LITERATURE REVIEW AND HYPOTHESIS

Potatoes generally have a starch content of around 22-28%. Compared to other starches, potatoes have *high swelling* power and viscosity values, both of which play an important role in the function of starch as *a gelling agent*. Many problems are found when using natural starch, including paste resistance, low stability and problems related to retrogradation. With this influence,

it is necessary to make modifications to starch to deal with the limitations of these problems both chemically and physically (Danimayostu, 2017). The physical properties of starch that are easily degraded provide more value in its use as a biodegradable material (Afiifah Radhiyatullah et al., 2015).

Chitosan has been widely studied for various applications due to its abundant and inexpensive biomass-derived properties, biodegradability, mucoadhesives, and derivatives of biomass. Chitosan also has antimicrobial activity, wound healing properties and hemostatic activity that make chitosan-based composites very useful in the biomedical field (Suryati, 2022).

Calcium carbonate ($CaCO_3$) is a type of calcium salt that can be found in natural materials such as alabaster, limestone, and eggshells. $CaCO_3$ material is used as an additional filling that is useful in overcoming the lack of strength of film properties (Hasanah &; Haryanto, 2017).

3. RESEARCH METHOD

The materials and equipment used in this study were *beaker glass, hotplate stirer, glass* mold, *oven*, digital balance, spatula, erlenmeyer, chitosan (shrimp shell DD: 90%), potato starch, $CaCO_3$ egg shell 90%, glycerol, *aquadest*, acetic acid 1%, NaCl and *Phospate Buffer Saline* (PBS). Research consists of three stages, namely preparation of raw materials, biocomposite processing and testing stage. In the last stage, tests are carried out on products including *swelling* tests, absorption tests, and functional group analysis.

4. RESULT AND DISCUSSION

In this study, based on the results of the analysis that has been carried out, it can be seen the influence of the addition of $CaCO_3$ materials on biocomposite characteristics, such as *swelling properties*, absorption properties, and functional group tests.

Swelling Test

Water resistance tests are conducted to determine the level of resistance of biocomposites to absorb liquids. The ideal environment for good wound healing is to keep the wound moist (Syahputra, 2018). A graph of the relationship between potato-chitosan starch variation and $CaCO_3$ on *swelling* properties can be seen in Figure 1 below.

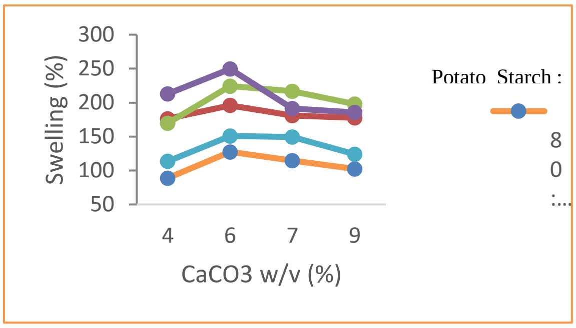

Figure 1 Graph of the relationship between variation in $CaCO_3$ composition and ratio of potato starch: chitosan (w/v) to *swelling* (%) of bio composites

In Figure 1 it can be seen that the % *swelling* value tends to decrease with increasing $CaCO_3$ composition. The highest percent *swelling* value was obtained in the variation in the composition of potato-chitosan starch (40:60) w/v% with $CaCO_3$ 4%, which is 213.157%. While the lowest percent *swelling* value in the composition of potato-chitosan starch (40:60) % w/v was obtained at $CaCO_3$ 9%, which was 185.53%. The results of research conducted (Haryanto, 2021), that with increasing chitosan concentration, the *swelling* ratio of hydrogel tends to rise. However, it should be noted that the effect of the amount of chitosan on the percent *swelling* value is not always linear. There are cases where when reaching a threshold, the addition of chitosan no longer increases the % *swelling* value significantly. For example, in variations in the composition (w/v%) of potato starch: chitosan (20:80) can be seen % *swelling* decreases. This happens because there is a limit to the capacity of chitosan to absorb liquids, which can reach a saturation point when the amount of chitosan reaches a certain level.

The results of the study (Zhou et al., 2019) also proved that the addition of $CaCO_3$ in chitosan acetate hydrogel increases the link between chitosan acetate chains and increases the crosslinking density so that water absorption capacity is reduced. As for according to (Saarai et al., 2011) the standard value of *swelling* which has been set as wound dressing is in the range of 200-500%. So that the results of degree *of swelling* from wound dressing prepared in study are close to the standard and still need improvement so that the value can increase.

Absorption test

The absorption test aims to determine the absorption capacity of potato starch-chitosan biocomposites and $CaCO_3$. One of the important parameters of wound dressings is their absorption ability. This is influenced by the higher the absorption capacity, the more it will increase its ability to absorb wound exudate (Nurlidar et al., 2013). In this test, the liquid used is a solution of

Phosphate Buffer Saline (PBS) with a pH 7.3 for 12 hours. PBS solution is made by dissolving the 1st PBS tablet in ml of aquades. Biocomposites are weighed before and after soaking to obtain percent absorption. The results of the absorption testing can be seen in Figure 2 below.

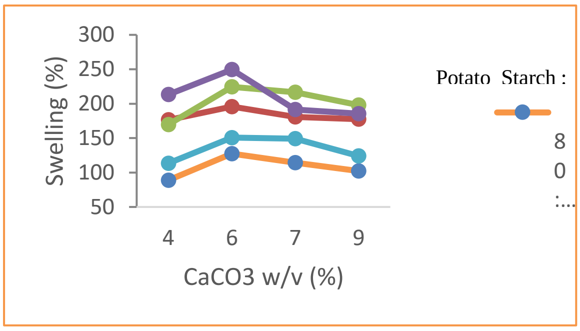

Figure 2 Graph of the relationship between variation in $CaCO_3$ composition and (w/v%) ratio of potato starch:chitosan to absorption ability of biocomposites

Figure 2 can be seen that the relationship between potato starch, chitosan and $CaCO_3$ affects the resulting absorption ability. The percent of absorption tends to decrease with increasing composition of $CaCO_3$. In this study, the highest percent absorption value was in a variation of (w/v%) (40:60) with $CaCO_3$ 6%, which was 72.09%, while the lowest absorption test result in the ratio of %w/v potato starch:chitosan (40:60) was obtained at $CaCO_3$ 9%, which was 68.72%. At high concentrations chitosan has stronger interactions with other materials in the system, such as fillers or other compounds (Zulfikar et al., 2009). Stronger interaction with other components can reduce the availability of chitosan to interact with the substance you want to absorb, thereby reducing the percentage of absorption.

As explained in research (Kasmuri & Zait, 2018) that eggshells as fillers are able to fill between polymer chains in bio composites, so as to reduce the speed of water absorption. An increase in the composition of $CaCO_3$ can result in an interaction or absorption competition between $CaCO_3$ and the substance to be absorbed (Suparwan et al., 2021). However, under certain conditions a higher % $CaCO_3$ concentration can increase the % absorption value, for example at the concentration of %w/v potato starch: chitosan ratio (20:80). Due to surface availability, an increase in the percentage of $CaCO_3$ in a system can result in an increase in the surface area of $CaCO_3$ available to interact with the substance to be absorbed.

Fourier transform infrared (FTIR) analisys

Fourier transform infrared (FT-IR), is one of the instruments widely used to determine molecular vibrations that can be used to predict the structure of chemical compounds. The results of

analysis of functional group were carried out to identify constituent compound, especially organic compound both qualitatively and quantatively. In study, the raw materials used are potato starch, chitosan with the addition of $CaCO_3$ as *filler*. Chitosan is a natural polymer that has three functional groups, namely amine acid, primary hydroxyl group and secondary hydroxyl group, while starch is starch from carbohydrates a polymer compound glucose consisting of three main components namely amylose, amylopectin and intermediates such as proteins and fats. The results of functional group analysis can be seen in Figure 3, Figure 4 and Figure 5.

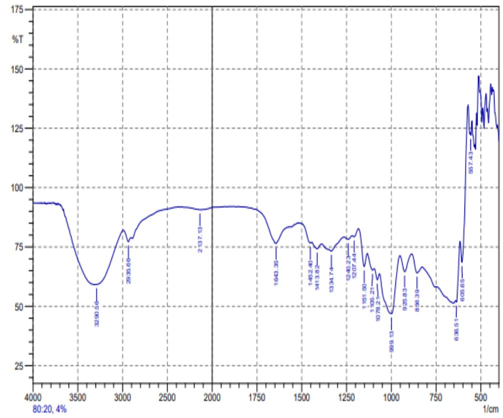

Figure 3 FTIR graph of potato starch-chitosan biocomposite (%w/v) (80:20) and 4% $CaCO_3$

Figure 3 shows the functional groups and compounds formed in the membrane layer. The results showed that the detection in samples (80:20) w/v % with the addition of $CaCO_3$ 4% there was an O-H group at wavenumber 3290.56 cm-1, C-H (Alkane) at wavenumber 2935.66 cm-1, C=C (Alkene) at wavenumber 1643.35 cm^{-1}.

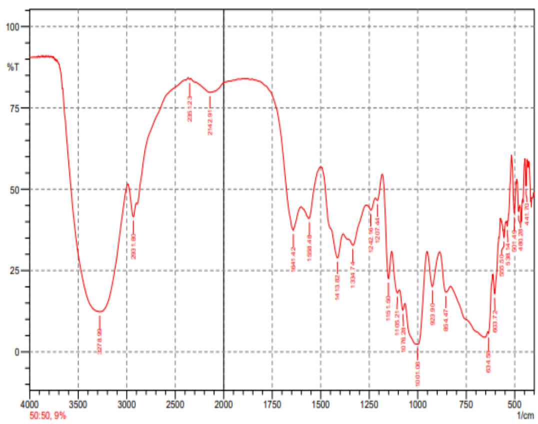

Figure 4 FTIR graph of potato starch-chitosan biocomposite (%w/v) (50:50) and 9% $CaCO_3$

Figure 4 can be seen the results of FTIR analysis in the membrane for samples with a concentration of (50:50) w/v % addition of $CaCO_3$ 9% shows the results that there are O-H groups at wavenumber 3278.99 cm^{-1}, C-H (Alkanes) at wavenumber 2931.80 cm^{-1}, double bonds with C=C functional groups identified at wavenumber 1641.42 cm-1, C-O functional groups identified at wavenumber 1151.50 cm^{-1}.

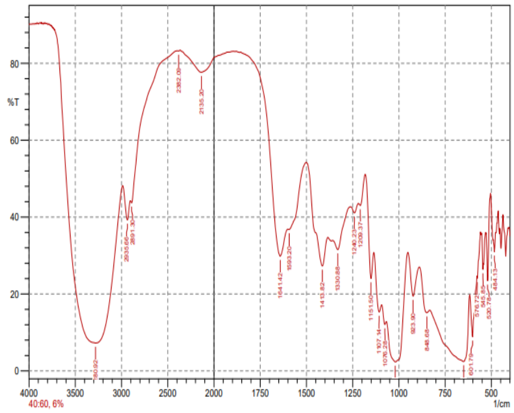

Figure 5 FTIR graph of potato starch-chitosan biocomposite (%w/v) (40:60) and $CaCO_3$ 6%

Based on Figure 5, it can be seen that the results of FTIR analysis at a concentration of (40:60) w/v% with the addition of $CaCO_3$ 6% show the results that there is an O-H functional group identified at wavenumber 3280.92 cm^{-1}, a C-H functional group is identified at wavenumber 2935.66 cm^{-1}, a C=C group is identified at wavenumber 1641.42 cm^{-1}.

The results of functional group analysis contained in biocomposites using a mixture of potato-chitosan starch and $CaCO_3$ showed that the resulting compound showed the presence of O-H hydroxyl groups derived from starch. Starch as a polysaccharide present in potatoes, has many hydroxyl groups that contribute to its hydrophilic properties and ability to form hydrogen bonds with water molecules (Sari et al., 2022). In FTIR analysis, the hydroxyl group (O-H) in starch will show typical absorption at about 3300-3600 cm^{-1}. This absorption peak reflects the *streching* and *bending* vibrations of the O-H group on the starch molecule. This hydroxyl group also plays a role in starch's ability to absorb and form complexes with calcium carbonate ($CaCO_3$) added to the mixture, thus forming a biocomposite containing both clusters.

The presence of C-O groups in starch and chitosan shows typical absorption at wavenumbers around 1000-1200 cm^{-1}. This absorption peak reflects C-O stretching vibrations in starch and chitosan molecules. This C-O group plays an important role in the chemical structure and functional properties of starch and chitosan, as well as in the interactions between these materials in biocomposites. These interactions can affect the physical, mechanical, and water absorption properties of biocomposites. The C-H group on the potato-chitosan starch biocomposite with the addition of $CaCO_3$ will show typical uptake at approximately 2850-3000 cm^{-1}. This absorption peak reflects C-H stretching vibrations in starch molecules, chitosan, and also possibly from $CaCO_3$ if there are C-H bonds in the compound.

According to the results of research conducted by (Rafif Putranta et al., 2019), chitosan-based wound dressings can stimulate wound closure, growth of new blood vessels, and regeneration of skin tissue in wounds. In addition, chitosan-based wound dressings have intrinsic antimicrobial properties that can prevent infection in wounds.

5. CONCLUSION

From the *swelling* test results, the addition of chitosan increases the percentage of water absorption due to the hydrophilic properties of chitosan and starch. The highest percent *swelling* value was obtained in the (40:60) w/v% potato starch-chitosan variation with 6% $CaCO_3$, which is 249.54%. In the absorption test, the absorption of biocomposite absorption perescentase made from potato-chitosan-$CaCO_3$ starch is the best in the (40:60) compost variation with 6% $CaCO_3$, which is 72.09%. From the results of analysis FTIR shows group contained in biocomposites made from potato starch-chitosan with the addition of $CaCO_3$ shows the results detected constituent components, namely O-H, C-H and C-O.

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