Synthesis of New Biodegradable Beauty Mask with Sodium Alginate and Agar

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Abstract. Nowadays, with the improvement of living standards, the consciousness of people over their skin status and hope for finding ways to pursue its perfect condition has become one of the most concerned topics. In this investigation, a wide range of sodium alginate, agar, and glycerol blends was rationed to find the most suitable mixture to produce beauty masks. The blend of 3 g pure sodium alginate, 1.25 g agar, and 250 mL of 4% (w/v) glycerol was used. The film without cross-linking could withstand twice as much weight compared to the film with CaCl₂, making it 13 times higher when comparing its stress. The water content is higher for the film without CaCl₂. When the two film was tested for glycerol and alcohol absorbance, sodium alginate with no cross-linking absorbance is preferred when testing in glycerol since it is less soluble. However, when testing with alcohol, the film with CaCl₂ is less soluble, thus preferred. The above qualities in which the film sample with CaCl₂ possess therefore enables it as a perfect paste for a beauty mask, being tensile, high absorbance of serum to provide a holistic care.

Keywords: Biodegradation; cross-linking; sodium alginate; agar.

1. Introduction

Biofilms are normally used as bioplastic and are developed to be applied in industries such as food packaging, construction, textiles, consumer products, transportation, and many more. The use of biofilms reduces plastic pollution and increases the sustainability of certain products. In particular, the cosmetics industry, which contributes to more than 120 billion units of plastic waste annually, has a lot of space to make a difference. Beauty masks, occupying 42% of the global beauty market, are yet non-degradable [1, 2]. Though made of non-woven fabrics, they do not contain natural ingredients such as cotton or wood fiber due to their high cost of production and transportation. Instead, they are made of artificial chemical fabrics, which can no longer be biologically decomposed. Therefore, the study aims to synthesize a biofilm with organic components as an alternative to non-biodegradable masks and investigate its properties in depth.

When preparing the films discussed throughout this report, glycerol functioned as a plasticizer, while the main constituents, agar and sodium alginate are both polysaccharides. Sodium alginate, a polysaccharide obtained from algae of the class Phaeophycean, is composed by L-guluronic acid linked by glycosidic bonds with the residues of D-mannuronic acid [3]. Being a natural gift from the planet, it can revitalize and moisturize skin, improve circulation, tighten loose skin, reduce pore size, lessen puffiness, and calm down skin after aggressive skin treatments [4]. The extraction of agar starts by washing the seaweed, heating it in water to dissolve the agar, then filtering the solution to remove the residues, later, cooling the filtrate to form a gel, and lastly removing the water from the gel via pressure or a freeze-thaw process. It is comprised of a combination of homogeneous polysaccharides, namely Agarose and Agarpectin [5]. Agarose is a strong gelling, non-ionic polysaccharide and it consists of alternating β-D-galactose and 3,6-anhydro-L-galactose units of agarobiose in its chemical structure (Fig. 1). Agarpectin is less clearly defined, a more complex polysaccharide with sulfate groups attached to it, that strongly influences the solution’s properties, gelling kinetics and gel features coming across for the formation of the membranes in this conducted research [6, 7].
In addition to the two biobased polymers, for the flexibility of the films, a plasticizer is vital for increasing the elasticity of a material. It is an essential characteristic for the cosmetic industry, especially for masks because the mask should be able to adjust according to the size of the consumer’s face. In this paper, glycerol was used as the plasticizer, which is important to overcome the brittle nature of the film due to extensive intermolecular forces [8, 9]. Moreover, being able to bend or crease without breaking is a characteristic that masks must possess to retain their shape when folded in the bag to be organized for commercial use. This softening and compatible feature of glycerol is extremely important to ensure the safety and comfort of the consumer when applying the material to the skin [10].

This research was undertaken to find the effect of varying the proportions between sodium alginate, agar, and glycerin, and find the most suitable concentration for each ingredient for the composition of the film. Moreover, this report will also explore the difference between the absorbance, strength, transparency, water content, and thermal stability of the sodium alginate solution. In addition, it also investigates the effect of Ca$^{2+}$ ions when modifying the network structure through cross-linking in the various compositions of sodium alginate. The main function of this solution is to create an antibacterial mask suitable for delivering high-quality serum in the face mask to the skin that maximizes the absorbance and is safe to be kept through the shelf-life period.

2. Methodology

2.1. Film Preparation

A solution of pure sodium alginate (3.0 g), agar (1.2 g) and glycerol (250 mL) of 4% (w/v) was prepared by mixing at room temperature for about an hour using a magnetic stirrer from Hangzhou QIWEI Instrument Co. LTD (Model: M-CL). The films were made by pouring 15 g of the mixture into a petri dish with a diameter of 8.5 cm and a depth of 2 mm. Films that were not treated were left to air dry for 24 hours while films treated with 10% (w/v) CaCl$_2$ solution were left for 15 minutes at room temperature in the solution and then rinsed with distilled water before being left to air dry at room temperature for 24 hours.

To speed up the drying process of the pure solutions without CaCl$_2$ a dehydrating machine from frunuts (Model TS-9688-3) was used where the solution in a petri dish was left for 2 hours at 70 °C instead of overnight to explore the ratio between the ingredients for the alginate blend.
2.2. Characterization of Samples

2.2.1. Morphology

The morphology of samples was placed under a microscope with a magnification of 1600x.

2.2.2. Transparency

To measure the transparency of the mask, a flashlight was used with a light intensity of around 8-12 lumens. This property was tested due to aesthetic reasons: considering users’ convenience.

2.2.3. Absorbance of different solvents

When immersed in water, membranes were first measured for mass and recorded as \( m_i \). After being heated for 2 hours on the electric heater at 100 degrees, the membrane is weighed again as \( m_f \), as shown in formula (1). In alcohol, the films’ mass was first measured and recorded as \( m_0 \). After being immersed in 7 g of 100% industrial alcohol for 43 hours, it was weighed again as \( m' \) (formula (2)). To test the absorbance of glycerol, a film was placed in a petri dish with 3g of glycerol and left overnight. After 43 hours the mass of the film was taken using an electronic balance (formula (3)).

\[
\text{Water content\%} = \frac{m_i - m_f}{m_i} \times 100% \\
\text{Alcohol\%} = \frac{|m_0 - m'|}{m_0} \times 100% \\
\text{Glycerol\%} = \frac{|m_0 - m'|}{m_0} \times 100%
\]

2.2.4. Thermal stability

Similarly, the film sample was weighed before as \( m_1 \), it was then heated up for 15 minutes at 40 °C and its mass was recorded every 5 minutes until it reached its final mass, \( m_2 \) (formula (4)).

\[
\text{Thermal stability\%} = \frac{m_1 - m_2}{m_1} \times 100%
\]

2.2.5. Mechanical properties

The strain (\( \epsilon \)) of an object is calculated by formula (5), where \( \epsilon \) equals the stress of the membrane, \( \delta L \) is the change in length of the film, and \( L \) is its original length. Since the shape of the film resembles a circle, the original length and the length after elastic elongation were both measured by taking two points farthest away from each other, which approximates the diameter of a circle. To measure the strain, one end of the film was held stationary by hand on the table and was aligned to a ruler, the other end was slowly pulled, with about the same amount of force, while the observer stared at the other end of the ruler. The moment just before the membrane broke from the center was taken as the maximum elongation, and the final length at its most elastic moment was used to subtract from the original length, to obtain the change in length of the biofilm.

\[
\epsilon = \frac{\delta L}{L}
\]

The stress is calculated by formula (6), where \( \sigma \) is the stress of the membrane, \( F \) is the force applied to this object and \( A \) is the area of the object that receives the force. The force exerted on the membrane is characterized by different masses. The applied area is the total area of the bottom of masses that directly touch the film surface, which can be derived from \( A = \pi r^2 \), where \( r \) is the radius of the bottom circle. The film was held by hand at the edge, and masses were placed in the center of the film so every part of the membrane would receive an equal amount of force with little uncertainty. If the mass was placed at the corners, the other parts would receive less force and the difference between the force exerted on each part increases, causing the corner of the film to break more easily, which lowers the accuracy of the measurement. When the membrane broke from the center, the weight of masses on the membrane before the elongation turned inelastic was measured as the applied force.
3. **Result and Discussion**

3.1. **Morphology**

Through Fig. 2(a) it is observed the film without Ca$^{2+}$ ions have some visible molecules. However, in Fig. 2(b), the connection between the Ca$^{2+}$ ions and COO$^-$ is very visible. This phenomenon is due to the formation of cross-linking, which is a chemical bond between the divalent ions, Ca$^{2+}$, and the two adjacent carboxyl groups of guluronic acids. As the Na$^+$ ions are replaced with Ca$^{2+}$ ions, a 3D network structure forms, known as the “Egg-box” model [11]. This improves the mechanical and barrier properties of the polymer. In addition, calcium alginate provides antiviral, antibacterial and antioxidant properties, which are important qualities regarding face masks. The addition of CaCl$_2$ to the sodium alginate mixture is crucial when modifying and controlling the properties as it acts as a control system for excessive moisture [12, 13]. Since there are more molecules connected, agreeing with the egg-box model and proving cross-linking happens. 

![Fig. 2 (a) Membrane without CaCl$_2$ and (b) Membrane with CaCl$_2$. (Picture credit: Original)](image)

3.2. **Transparency**

When the films cover the flashlight, both lights appear dimmer. This is because light must travel through a medium, therefore some of the light is absorbed and since the medium (sodium alginate film) is not smooth light also scatters. Noticeably, the light passing through the biofilm with the presence of Ca$^{2+}$ ions scatter a bit more than the non-crosslinked sample, showing a larger halo on the optical screen, which indicates lower transparency of the crosslinked membrane. This is because the cross-linking effect due to the addition of calcium chloride causes the structure to be coordinated and more tightly connected, leaving little space between polymer chains. Hence, it is more difficult for light to travel through the crosslinked film and can only be absorbed by the membrane, making it less transparent. Thus, the film without CaCl$_2$ is preferred to clearly show the facial features, increasing the convenience for clients.

3.3. **Absorbance of Different Solvents**

3.3.1 In water

The membrane samples formed with the addition of CaCl$_2$ and those without showcased a well-preserved characteristic of water storage capacity, given both values of water content are over 75% of their previous weight. This can depict how much serum can be absorbed in the mask, as a large percentage is only water. However, when comparing the films there is a considerable 13.8% difference between the water content of the 78.9% film formed with CaCl$_2$ and 92.7% film without
CaCl₂ (Table 1), the film with cross-linking has more bonds between the alginate strands and the Ca²⁺ ions, consequently, alginate chains become less exposed and therefore, the water content decreases. Due to the morphology and inner structure of the material, the content of water in the film with Ca²⁺ ions is higher, with a leading percentage of 92.7%.

### Table 1. Water absorbance

<table>
<thead>
<tr>
<th>Sodium alginate film</th>
<th>Mass before heating (g)</th>
<th>Mass after heating (g)</th>
<th>Water content (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>With CaCl₂</td>
<td>5.70</td>
<td>1.20</td>
<td>78.90</td>
</tr>
<tr>
<td>Without CaCl₂</td>
<td>15.00</td>
<td>1.10</td>
<td>92.70</td>
</tr>
</tbody>
</table>

### 3.3.2 In alcohol

This experiment tests the alcohol absorbency of the film samples, aiming to simulate a similar environment to the serum content within masks where a variety of alcohol compounds that function as sterilization is used. As shown in Table 2, the percentage change of both films has shown a decrease in mass.

The alginate film with CaCl₂ has a 15% decrease in mass before and after 43 hours, which is relatively small compared to the sample without CaCl₂. This is because of the slight polarity of calcium alginate used to form the film with CaCl₂. As alcohol is a polar solvent and calcium alginate is a polar solute, calcium alginate can dissolve in alcohol, thus causing a slight reduction of mass. However, for the film without CaCl₂, agar alginate, is highly polar therefore it can dissolve in the polar solvent, alcohol in this experiment. Thus, explaining the 33.3% decrease in mass.

In addition, the difference of 18.3% in mass of the films made with and without CaCl₂, can be explained by the polarity of the final product, where agar alginate is more polar compared to calcium alginate. Hence, it is deduced that the addition of CaCl₂ makes the masks less soluble and, therefore, a better material for containing the serum of masks. However, since the proportion of alcohol in mask serum is minimal, and the percentage change of both materials is below 35%, both films are suitable to be used.

### Table 2. Alcohol absorbance

<table>
<thead>
<tr>
<th>Sodium alginate film</th>
<th>Original mass (g)</th>
<th>Mass after 43 hours (g)</th>
<th>Percentage change (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>With CaCl₂</td>
<td>0.4</td>
<td>0.34</td>
<td>15.0</td>
</tr>
<tr>
<td>Without CaCl₂</td>
<td>0.9</td>
<td>0.6</td>
<td>33.3</td>
</tr>
</tbody>
</table>

### 3.3.3 In glycerol

From the statistics in Table 3, the mass of the film with CaCl₂ had a 480% increase in mass, while sodium alginate without CaCl₂ had an increase of 410% in mass. Even though both films absorbed at least 4 times higher than their original mass, the film without CaCl₂ has a lower absorbance rate (410%) and, therefore favored since the serum in the face mask must last for at least half a year. Hence, the facial mask made without CaCl₂ can provide clients with necessary moisture for the skin.

### Table 3. Glycerol absorbance

<table>
<thead>
<tr>
<th>Material: Sodium Alginate film</th>
<th>Original mass (g)</th>
<th>Mass after 43 hours (g)</th>
<th>Percentage change (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>With CaCl₂</td>
<td>0.3</td>
<td>1.74</td>
<td>480</td>
</tr>
<tr>
<td>Without CaCl₂</td>
<td>1.0</td>
<td>5.1</td>
<td>410</td>
</tr>
</tbody>
</table>

### 3.4. Thermal Stability

The thermal stability of the mask was tested in case customers would heat and use them as stress relievers, or when the mask is mistakenly left in a hot environment. Through the course of 15 minutes, the change of weight for the sample formed with CaCl₂ was within 0.2 g, where its final mass was 0.5 g, forming a 0.1 g increase compared to the previous mass of 0.4 g before the heating, as shown in Fig. 3 and Table 4. This abnormal scenario can only be explained through the following two reasons: (i) One possibility is that a reaction took place in the material when heated up, therefore it would not
be qualified to be used as a warm mask. (ii) After heating, the surrounding temperature of the air increased, therefore density decreased, causing the thrust applied from the atmosphere to the heater and sample to decrease. Hence, it is not necessarily that the mass increases, but rather a physics phenomenon.

As for the film without CaCl\textsubscript{2} it had a continuous decrease in mass, to 28.6% of its previous weight. This is due to the decrease in water content as it is being dried on the heater for 15 minutes. Considering the usage of a petri dish to perform the experiments upon both samples, the exposure to oxygen determines the evaporation of water within films, demonstrating both the ability of water uptake and stability of the membrane surface itself.

Thus, given the uncertainty behind the percentage change for the sample with CaCl\textsubscript{2}, where it was unable to preserve the qualities for it to be safely applied to the face. On the other hand, the contrasting membrane without CaCl\textsubscript{2} has proved to be capable to withstand the heat.

![Fig. 3 Thermal stability trend (Picture credit: original)](image)

<table>
<thead>
<tr>
<th>Sodium alginate film</th>
<th>Mass before (g)</th>
<th>Mass after heated for 15 minutes (g)</th>
<th>Percentage change in mass (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>With CaCl\textsubscript{2}</td>
<td>0.4</td>
<td>0.5</td>
<td>25.0</td>
</tr>
<tr>
<td>Without CaCl\textsubscript{2}</td>
<td>1.4</td>
<td>1</td>
<td>-28.6</td>
</tr>
</tbody>
</table>

**3.5. Mechanical Properties**

The strain and stress of the membrane was experimented to explore whether the mask can fit all types of face shapes.

**3.5.1 Strain**

The strain of the membrane with calcium chloride is 0.029, which is almost 30 times smaller than 0.85—the one without calcium chloride (Table 5). This shows the elongation of the biofilm without the addition of calcium chloride is much more elastic than the other. This can be explained through the process of ion exchange and the cross-linking effect: since the polymers now crosslink with each other, they are connected more tightly and become more coordinated, leading to a more kinetically stable structure. Hence, rather than the loosely connected membrane without calcium chloride, it is more difficult to stretch the crosslinked biofilm. This argument is further supported by the observation of the stress of the membrane.
Table 5. Data for calculating strain

<table>
<thead>
<tr>
<th>Material: sodium alginate film</th>
<th>Original length L (cm)</th>
<th>Maximum length L_{max} (cm)</th>
<th>Change in length δL (cm)</th>
<th>Strain ε</th>
</tr>
</thead>
<tbody>
<tr>
<td>With CaCl\textsubscript{2}</td>
<td>6.8</td>
<td>7.0</td>
<td>0.2</td>
<td>0.029</td>
</tr>
<tr>
<td>Without CaCl\textsubscript{2}</td>
<td>6.5</td>
<td>12</td>
<td>5.5</td>
<td>0.85</td>
</tr>
</tbody>
</table>

3.5.2 Stress

The stress of the biofilm with CaCl\textsubscript{2} turns out to be 2.54, which is almost 13 times smaller than the one without calcium chloride (Table 6). It indicates that the non-crosslinked membrane can endure larger forces, showing its capability to carry heavier objects. The cross-linking effect of adding calcium chloride can also explain this phenomenon: divalent cations exchanging with Na\textsuperscript{+} ions link COO\textsuperscript{-} groups of different polymer chains together, causing the chains to cross-link and form a more coordinated three-dimensional structure [14]. Thus, when the membrane with the presence of calcium chloride experiences an external force, it cannot be elastically enlarged, causing each unit area of the film to receive more force. On the contrary, the non-crosslinked membrane has the freedom to elongate so the total surface area applying the force increases and each unit area can hold less force. Therefore, the membrane without calcium chloride has a higher stress value.

Table 6. Data to calculate stress

<table>
<thead>
<tr>
<th>Sodium alginate film</th>
<th>mass ( m ) (g)</th>
<th>Maximum force ( F ) (N)</th>
<th>total area ( A ) (m\textsuperscript{2})</th>
<th>Stress ( \sigma )</th>
</tr>
</thead>
<tbody>
<tr>
<td>With CaCl\textsubscript{2}</td>
<td>300</td>
<td>2.94</td>
<td>1.16</td>
<td>2.54</td>
</tr>
<tr>
<td>Without CaCl\textsubscript{2}</td>
<td>600</td>
<td>5.89</td>
<td>0.184</td>
<td>32.0</td>
</tr>
</tbody>
</table>

To observe the mechanical properties of the membrane more clearly, the strain and stress values are integrated together in a coordinate system to create a strain-stress graph, as shown in Fig. 4. The point on the lower-left corner represents the yield strength of the membrane with calcium chloride, the upper-right point represents the yield strength of the membrane without calcium chloride. It is concluded that the non-crosslinked film has both higher strain and can withstand higher stress showing better mechanical properties than the crosslinked one. Moreover, from the mechanical perspective, the film without calcium chloride is more appropriate for producing beauty masks because its high strain value makes it easier to deform in order to adapt different facial features of customers.

![Strain-stress graph of agar alginate biofilm. (Picture credit: original)](image-url)
3.6. Comparison of Samples

The properties discussed are summarized in Table 7, which shows that the agar alginate film without the addition of calcium chloride generally has better characterizations, thus is more appropriate for making a mask.

<table>
<thead>
<tr>
<th>Characterization</th>
<th>Film with CaCl₂</th>
<th>Film without CaCl₂</th>
</tr>
</thead>
<tbody>
<tr>
<td>Transparency</td>
<td>Low</td>
<td>High</td>
</tr>
<tr>
<td>Thermal stability</td>
<td>Unstable</td>
<td>Stable</td>
</tr>
<tr>
<td>Mechanical strength</td>
<td>Less elastic</td>
<td>More elastic</td>
</tr>
<tr>
<td>Water uptake</td>
<td>Lower content</td>
<td>Higher content</td>
</tr>
<tr>
<td>Alcohol absorbance</td>
<td>Less absorbent</td>
<td>More absorbent</td>
</tr>
<tr>
<td>Glycerol absorbance</td>
<td>More absorbent</td>
<td>Less absorbent</td>
</tr>
</tbody>
</table>

4. Conclusion

This research aims to find out the most suitable material for beauty masks by synthesizing agar, sodium alginate, and glycerin in various compositions to adjust the membrane properties. The sample without calcium chloride has a higher water content of 92.7% compared to the crosslinked sample, this indicates better serum absorbance and capability to preserve moisture in masks. Regarding thermal stability, the non-crosslinked sample is preferred as its mass steadily decreases due to dehydration during heating, while the calcium chloride-containing membrane poses a potential risk of skin reaction due to an internal reaction within the sample. In terms of mechanical properties, the membrane without calcium chloride exhibits superior yield strength, due to its elasticity. The crosslinked biofilm isn't suitable for cosmetics as it doesn't readily conform to different facial shapes and features. In organic solvents, both films absorb up to 35% of their mass and dissolve in alcohol due to their polarity, with the crosslinked film dissolving slower. However, for typical facial masks with low alcohol content (below 10%), this factor is less critical. In glycerol, the non-calcium chloride film absorbs slowly, while the crosslinked one absorbs quickly; even dissolving. To extend shelf life, lower absorbance is favored, making the non-crosslinked membrane more reliable. Unfortunately, due to time constraints, deformation patterns couldn't be tested. Further investigation can be conducted to analyze elastic and inelastic deformations, and potentially whether it follows Hooke's Law. Moreover, film sample degradation also wasn't tested due to time limitations. However, another study found that Agarivorans, a marine bacterium, exhibited strong alginate degradation capabilities with a large degradation circle and high enzyme activity. Highlighting the eco-friendly and efficient nature of using bacteria for bio-based degradation, with no greenhouse gas emissions.

Authors Contributions

All the authors contributed equally, and their names were listed in alphabetical order.

References


