

The production of sodium alginate from Sargassum seaweed for application as a cosmetic thickener

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Abstract

Cosmetic thickeners are highly used in the cosmetic industry and their primary function is to not only stabilize but also add textures to products which include gels, creams, and lotions. This research focuses on the production of sodium alginate from the Sargassum seaweed with applications as a cosmetic thickening agent. In 2020, the National Environmental Protection Agency (NEPA) of Jamaica informed the public that the influx of the Sargassum seaweed was expected to traverse several beaches across the island. This excess flow of the seaweed tends to form mats and subsequently decomposes on the coastline. This increase in seaweed is linked to the rising temperatures and effects of climate change and sustainable ways of utilizing this will be required as an alternative means of disposal. Seaweed from Jamaica's coastline was oven dried at 35 °C, soaked for 2 hours in Na₂CO₃ and the alginate subsequently extracted using Calcium Chloride. The alginate had an average viscosity of 342 mPa.s and a pH of 10.6 and a yield of 76%. Through the identification of alternative uses for floating seaweed, it will not only improve the appearances of the beaches, but will also reduce the costs associated with their removal.

Keywords seaweed, cosmetics, sustainability, alginate, natural products

I INTRODUCTION

With the projected increase in the Sargassum Seaweed in Caribbean waters, the beaches are greatly affected as this type of seaweed forms extensive mats and attracts insects and deters tourists [1]. Islands such as Jamaica rely on tourism as a major source of foreign exchange and the increase in the floating seaweed on the coastline has a negative impact on tourism and biodiversity. [2] The presence of excess seaweed is related to the heightened ocean temperatures in addition to the availability of nutrients in both the Caribbean Sea and the Gulf of Mexico. Jamaica [2]. Since 2011, a deluge of Sargassum seaweed has been filling coastal waters

and beaches, a direct consequence of this is the reduction in tourism income and a consequential in funding spent on beach cleaning activities. [3]. In 2018, there was the presence of the great Sargassum Belt which spanned 8,850 km from the shores of Africa to the Gulf of Mexico [2]. While the sargassum seaweed is a natural occurrence in Caribbean waters, the increase in sea water temperature in addition to the use of fertilizers have resulted in a perpetual increase in sargassum seaweed. [4]. The processing of the seaweed into useful products has the potential to alleviate the tourism industry while providing useful natural thickener for the growing cosmetics industry and an alternate source of revenue during a declining economy.

A Alginate

Sargassum seaweed is a known source of several plant derived compounds which include alginate, vitamins, carotenoids, fibres, proteins and minerals [5]. Brown seaweed such as the Sargassum contain alginate within their cell walls. Alginate typically exists as magnesium, calcium, magnesium and sodium salts of alginic acid [6]. Alginate is a water soluble carbohydrate that is typically used to thicken aqueous solutions, form gels or water soluble films [6]. It is these useful properties which have facilitated their applications in a vast number of industries such as food, pharmaceutical, textile printing, paper making and as biocatalysts [7].

Alginic acid can account for up to 40% of the dry weight of brown seaweed such as Sargassum spp and is a water soluble hydrocolloid biopolymer.

Alginate is the derivative of alginic acid and is the typical name given to a group of polysaccharides containing 1,4-linked β-D-mannuronic (M) and α-L-guluronic (G) acid residues arranged in a non-regular, block-wise order along a chain and It is the arrangement and ratio of these blocks give the alginate its particular properties [8]. There are over 300 species of Sargassum seaweed available, and as

expected, there are variations in their properties, which are typically linked to their location and would consequently affect their application [9].

Commercially, the forms of alginate used are propylene glycol alginate and alginic acid. Potassium, calcium, triethanolamine and ammonium salts are used on a smaller scale [10]. The colloidal properties of alginate have also been explored through the use of sodium alginate as a suspending agent, flocculant and even as a stabilizer in ice cream.

B Solubility

The solubility of alginates decreases if other compounds such as sugars, proteins, starches and salts of monovalent cations are present [12]. As a result, their presence decreases the rate of hydration and increases mixing time. As a means of mitigation, the aforementioned compounds can be added after hydration and dissolution of the alginate. Alginate inhibition can also be caused by the presence of small quantities of polyvalent ions such as calcium ions. As a way of increasing solubility, these ions have to be complexed with reagents such as sodium hexametaphosphate or ethylenediamine tetraacetic acid (EDTA).

C Viscosity and pH

The degree of polymerization (DP) of an alginate and molecular weight are directly related to the viscosity of alginate solutions [13]. Conversely, reduction of viscosity during storage is a measure of the magnitude of depolymerisation of the alginate.

Alginates, especially sodium alginate, are produced in various grades which refer to the viscosity of their 1% aqueous solution: low, medium and high viscosity alginates. Alginates with a high DP have been found to be more unstable than those with a low DP [14]. Low viscosity sodium alginates (<50 mPa.s) have been stored at 10-20°C with no observable change in three years. Research has shown that higher viscosity alginates are more unstable when stored over a one year period and their instability increases with the temperature. Studies show that solid alginate is best stored in cool conditions of 25°C or less as higher temperatures promote depolymerisation which affects viscosity and gel strength. Also, the moisture of the alginate affects depolymerisation and so they should be stored in a dry area.

Solutions of neutral pH with low to medium viscosity can be stored at 25°C for several years without significant viscosity loss, on the condition that microbial preservative is added as bacterial and mould growth leads to depolymerisation too. Like in the solid form, solutions of highly polymerized alginates are unstable. Similarly, solutions of alginate will depolymerize faster with an elevation in temperature. Stability of solutions is also affected by acidic conditions and the most stable range is pH 5-9. However, propylene glycol alginate solution is most stable at pH 3-4 and will lose viscosity rapidly below pH 2 and above pH 6.

Most uses of alginates are dependent on their ability to increase the viscosity of aqueous systems using low concentrations. The viscosity behaviour of alginates at low concentrations can be described as pseudoplastic which means the viscosity decreases as the rate of shear increases, and this effect is reversible up to very high rates of shear [15]. This effect is more clearly seen with alginates of high molecular weight, in sodium alginate solutions containing calcium ions, and with propylene glycol alginate above 1% concentration. These solutions can also be thixotropic which means that they show a time-dependent thinning at constant shear rate and their recovery to the initial viscosity is also time dependent.

Several factors influence the viscosity of alginate solutions. The viscosity is related to the molecular weight. The higher the molecular weight, the greater its viscosity. The molecular weight may be controlled by altering the extraction conditions. Medium viscosity sodium alginate (200-400 mPa.s) is most commonly used. The concentration also affects viscosity. While there is no simple relationship between concentration and viscosity, [10] developed an equation which may be applied to a wide variety of alginates over a wide range of viscosity changes:

$$\log_{10} \text{ viscosity} = a\sqrt{(\text{concentration})} - b$$

where a is a constant related to the DP of the alginate and b is a constant for a particular type of alginate. Also affecting viscosity is temperature. Viscosity decreases with temperature increase at a rate of about 2.5% per °C. Upon cooling, the viscosity returns to close to its original value though if held above 50°C too long, a permanent loss of viscosity will occur due to depolymerisation. Alginate solutions are not affected by freezing and thawing though McHugh

(1987) notes that if calcium is present, the viscosity will increase and irreversible gelation will occur.

Another factor that influences viscosity is pH. The viscosity of alginate solutions is not affected between pH 5-11. As the pH goes below 5, the free -COO^- ions in the chain become protonated to -COOH , so there is a reduction in the electrostatic repulsion between chains and they are able to come closer and form hydrogen bonds resulting in higher viscosities [11].

If there are low concentrations of calcium ions in an alginate solution, the viscosity will increase and large amounts will result in gelation. Increasing the presence of calcium is thus a way of increasing viscosity without increasing the amount of alginate or the alginate's molecular weight. This increase in viscosity is difficult to predict but it depends on the composition of the alginate and its degree of polymerization as alginates with a higher M/G ratio give greater viscosity changes [11]. However, alginate solutions with calcium are more shear sensitive and show a greater loss of viscosity with stirring than those without. Solutions become more thixotropic as the calcium concentration increases.

D Alternative Natural Cosmetic Thickeners

With the current trend of natural cosmetics, natural thickeners would be suitable to not only stabilize key products but also add additional use properties. Natural thickeners such as xanthum gum, acacia gum and hydroxyethylcellulose. Xanthum gum is an affordable anionic thickener which is compatible with most ingredients. As a result it is widely utilized in the cosmetic industry as a cosmetic thickener.

Hydroxyethylcellulose is an additional natural thickener which is derived from plant cellulose and widely used as a nonionic natural polymer and is also mostly compatible with sodium alginate. Another natural thickener which has applications in the cosmetics industry is acacia gum. It is derived from the sap of the acacia tree and is an anionic polysaccharide thickener.

II MATERIAL AND METHODS

The *Sargassum* seaweed is the habitat for several organisms such as crabs, shrimps, snails and barnacles. Consequently, all collected seaweed was meticulously examined for the presence of these

creatures to ensure that none was harmed during this research.

The alginate production methodology involved collection, moisture tests, extraction, pH and viscosity tests.

A Collection

Fresh *Sargassum* seaweed was sorted and collected from the Hellshire Beach in St. Catherine Jamaica.

B Moisture Analysis

The moisture content of both oven and air dried seaweed were determined. This was done to determine the efficacy of both in seaweed preparation. 30 g samples were allowed to dry in the oven at a temperature of 35 °C for 12 hours. The air dried samples were allowed to dry in the open air for two days.

C Extraction

1. The oven dried seaweed (6g) was measured and placed in a beaker.
2. 150 mL of 0.2M HCL was added to the seaweed. The mixture was subsequently left to soak for two hours.
3. After soaking, 0.2M HCL solution was removed and the seaweed was washed with distilled water.
4. 150 mL of 2% sodium carbonate solution was added to treat the seaweed samples at different conditions: and left to treat the seaweed for:
 - 2 hours at 50°C
 - 3 hours at 40°C
 - 12 hours at room temperature (30°C)
5. At the end of the treatment 3 g of alum was added to the mixture for an hour.
6. After which the top was scraped off and the mixture filtered.
7. After filtration the viscosity of the solution was tested and the precipitation using the Calcium Chloride Method was carried out.

D Calcium Chloride Method

70 mL of 10% calcium chloride was added to 50 ml of filtered seaweed solution. After which a brown precipitate was produced in the solution. It was obtained by centrifuging or straining the mixture or a combination of both depending on the nature of the mixture. The wet weight of this extract was

recorded. Then, 20-50mL of .5 HCL was added to the extract.

E pH Check

pH test was carried out on all samples to ensure that they were within the accepted range of 8.5- 10.5 which is the requirement for cosmetic grade sodium alginate.

F Viscosity

The viscosity of the samples were tested to ensure that the samples were within the desired range for cosmetic grade sodium alginate, (200-400 mPa.s). This was carried out using the Stabinger SVM3000 viscometer.

G Indicator Test

This test was carried out to ensure that the extraction yielded sodium alginate. Less than 1 mg of product was mixed with 1 mL of ferric sulphate. A cherry red-colour was expected if the product was sodium alginate.

IV RESULTS AND DISCUSSION

Moisture Analysis

The following table shows the moisture % of each batch of fresh, air dried and oven dried seaweed used. As expected the fresh seaweed had the highest moisture content. While the literature did not suggest a suitable moisture content for alginate extraction, it was decided to ascertain moisture contents for future research.

Table 1: *Sargassum* moisture content

Seaweed Type	Moisture %
Fresh	83.03
Air Dried (35°C)	10.83
Oven Dried (35°)	6.30

Table 2 and Table 3 highlight the key parameters required for the selection of the optimal conditions for sodium alginate for cosmetics application. Based on the results, the air dried sample at 30 C and 12 hours had the highest yield of 93%, however this

would not be suitable for cosmetics application due to the accepted pH of 8.5 -10.5. The optimum yield was determined to be the second highest value 76% which produced from the oven dried seaweed soaked in Na₂CO₃ for 2 hours at 50°C, it also had a desirable pH of 10.4. The proposed reason for this is because of the length of time and temperature at which the Na₂CO₃ was allowed to soak the seaweed. Na₂CO₃ is the main reagent used in the extraction. Therefore, the longer this reagent is allowed to interact with the seaweed at elevated temperatures the more precipitate will be available for precipitation.

As oven dried and air dried samples both had high and low yields at different temperatures, extraction times and precipitation methods. There was no direct correlation between the yield and the type of seaweed used.

It was expected that either longer temperatures or higher temperatures would have a great effect on yield. The reaction time showed a clear effect as seen with all the results for air dried seaweed. The yield% increased significantly at longer times with the maximum yield % at 93% for the longest time. When oven dried seaweed is analysed, higher temperatures give higher yields though the difference is not as quite as significant

According to the literature review, time of extraction influences the yield % obtained. It further ascertained that temperature is also very important when extracting for *Sargassum* species. However, in our research, it was observed that increasing the length of time rather than increasing the temperature is the key to higher yield.

Table 2: *Sargassum* seaweed Oven Dried Sample Results

Oven Dried Sample Results	1	2	3
Moisture removed (%)	52.90	40.10	52.00
Extraction Time (hrs)	2	12	3
Extraction Temperature (C)	50	30	40
Yield %	76	71	72
pH	10.4	10.4	10.5

Colour of extracted sodium alginate	Off White	Off White	Dark Cream
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Table 3: Sargassum seaweed Air Dried Sample Results

Oven Dried Sample Results	4	5	6
Moisture removed (%)	79.30	58.13	67.25
Extraction Time (hrs)	2	3	12
Extraction Temperature (C)	50	40	30
Yield %	36	67	93
pH	10.8	10.6	10.8
Colour of extracted sodium alginate	White	Dark Cream	Dark Cream

Cosmetic grade Sodium Alginate powder obtained was expected to be between white and Cream/yellowish-brown. All the samples obtained were in this required range. It was noted also that the precipitation method which produced the lightest coloured powders were produced from the ethanol precipitation method. It was also observed that in order to get a lighter colour or the truest colour the sodium alginate paste should be left for four (4) hours at 105°C.

The viscosity of the sodium alginate produced from the best chosen method was measured. It was found to be 342 mPa.s. According to the literature review, the expected viscosity of cosmetic grade alginate should be in the range 300-800 m.Pa.s Hence, the result obtained was indeed within range of that which was expected.

When less than 1 mg of the extracted product was mixed with 1 ml of ferric sulphate a cherry red color was observed and concluded that the product was sodium alginate.

CONCLUSION AND RECOMMENDATION

The objective of the is experiment was not only to successfully extract Sodium Alginate from brown *Sargassum* Seaweed but also to design a method to extract the alginate found in *Sargassum* species in order to use it as a thickening agent in the cosmetic industry in Jamaica. This was achieved by using different conditions for extraction and different methods for precipitation and analyzing which condition and precipitation method causes the highest yield of extract to be produced at cosmetic grade pH and colour. The viscosity was tested to ensure it was also within the expected range for cosmetic grade.

The objective of the experiment was achieved. The sodium alginate was successfully extracted. The properties of the extract were indeed at cosmetic grade as was required with viscosity measuring 342 mPa.s and an average pH 10.6 and the colour of the powder was in the required range of white to yellowish-brown. Lastly, the best extraction method was found to be soaking the oven dried seaweed for 2 hours at 50°C in Na₂CO₃ to produce a yield of 76%.

For industrial adaptation, the above method is best. Drying the seaweed in the oven takes less time and gives more consistent results. The shorter reaction time is also more appealing.

The recommended cosmetic products in which this extracted sodium alginate can be used include body butters, creams, face masks, lotions, conditioners, shampoos and all other cosmetics which are liquid in nature and require the use of a thickening or binding agent.

Recommendations and Future Work

1. Further studies can be carried out to increase yield and reduce pH through the variation of drying time, extraction time and temperature
2. Further study can be carried out using formaldehyde in addition to hydrochloric acid as a reagent in the washing step (step 1). This will help to determine if washing with formaldehyde would cause a higher or lower yield of extract.

3. Further study can be carried out to determine if bleaching the sodium alginate extract will lessen the many advantageous benefits of using Sodium Alginate. Bleaching is done in the commercial production of Sodium Alginate to give a more appealing colour.

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