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Extraction and Characterization of Sodium Alginate from Egyptian Algae

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Abstract

Sodium alginate was extracted from Egyptian brown algae using two different methods. In one approach, the brown algae were bleached before extracting the sodium alginate, while in the other method, extraction occurred first, followed by bleaching. The choice of extraction method influenced the yield. The method where sodium alginate was extracted before bleaching resulted in products with higher viscosity. Additionally, the properties of the products and the apparent viscosity of the pastes varied depending on the bleaching agent used. For example, at a shear rate of 6.779 sec^{-1}, the apparent viscosity measured 12,520 centipoises with sodium hypochlorite and 8,481.7 centipoise with sodium chlorite. It is advisable to use sodium hypochlorite as the bleaching agent instead of sodium chlorite when extraction precedes bleaching, as it is safer and more cost-effective. All sodium alginate pastes mentioned demonstrate a decrease in apparent viscosity as the shear rate increases. Samples produced with a liquor ratio of 1:10 appeared whiter than those with a ratio of 1:7.5. The resulting products display non-Newtonian pseudoplastic behavior. The method used for extracting sodium alginate from raw algae has a significant impact on the apparent viscosity of the final product.

Keywords: Characterization, Extraction, Egyptian Brown Algae, Sodium alginate.

1. Introduction

Sodium alginate has emerged as a highly researched natural anionic polymer. It is a multifunctional biomaterial, serving roles such as a viscosity enhancer, stabilizer, matrixing agent, encapsulation polymer, bio adhesive, and film former for transdermal and transmucosal drug delivery systems. This natural polysaccharide features a linear structure and is biodegradable, biocompatible, and safe for the body. It contributes strength and flexibility to tissues and is also valued industrially for its gelling, viscous, and stabilizing properties, along with its ability to retain water. Alginate can be synthesized from the cell walls of several brown algae species, including Laminaria hyperborea, Ecklonia maxima, Ascophyllum nodosum, Eisenia bicyclis, and Macrocystis pyrifera, as well as from bacteria such as Azotobacter and Pseudomonas.

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Alginate extracted from brown algae holds commercial significance in the food, pharmaceutical, and cosmetic industries[1–5]. The extraction process from brown algae takes place in an alkaline medium using sodium carbonate, sodium hydroxide, or aluminum hydroxide in several stages after the harvested algae are dried and shredded. The extract is then precipitated with sodium chloride or calcium and filtered. The precipitate (sodium/calcium alginate) undergoes conversion into alginic acid through treatment with diluted hydrochloric acid, and this alginic acid is subsequently transformed into dry sodium alginate powder. To ensure its suitability for use, the alginate is chemically treated to eliminate impurities such as heavy metals, endotoxins, proteins, carbohydrates, and polyphenols before being processed into powder form[2,3,6–9]. Alginate is an anionic polysaccharide present in the outer cell wall of brown algae like kelps. Its primary component is alginic acid, while sodium alginate is the sodium salt of alginic acid. This polymer comprises numerous free hydroxyl and carboxyl groups along its backbone chain. The linear anionic polysaccharide consists of two types of 1,4-linked hexuronic acid residues: β-d-mannuronopyranosyl (M) and α-l-guluronopyranosyl (G). These residues are organized into blocks of repeating M residues (MM blocks), blocks of repeating G residues (GG blocks), and mixed blocks of M and G residues (MG blocks)[10].

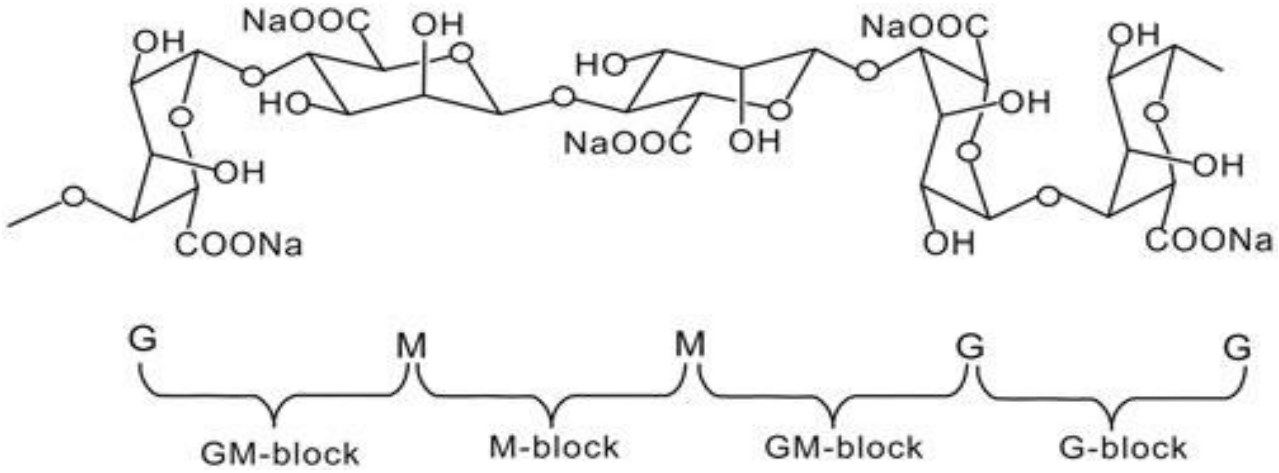


Figure 1: The molecular structure of sodium alginate.

Sodium alginate stands out as a key thickening agent, with over 300 documented commercial uses across diverse industries such as textile printing, paper sizing, welding rod extrusion, ice cream, salad dressing, beer foam stabilization, restructured fruits and vegetables, jellies, and pet foods, among others[11]. Its lack of reactivity with dyes used on cellulose fibers makes it particularly advantageous for applications involving reactive dyes[12]. Along Egypt's coastlines, approximately 4,000-6,000 tons of algae are washed ashore

annually near Alexandria-Tidal. Although harvesting has not yet occurred, red algae are collected for agar extraction. Despite the presence of brown algae in the Red Sea, Egypt continues to import sodium alginate.

The primary goal of this research is to explore an effective method for extracting sodium alginate from Egyptian brown algae and to analyze its physicochemical characteristics, with a focus on its rheological properties.

2 . Materials and Methods

2.1. Materials

2.1.1. Substrate

Various species of Egyptian brown algae from the Red Sea were utilized, including Sargassum dentifolium, Sargassum asperifolium, Sargassum eatifolium, Turbinaria decurreas, Cystoseira trinode, and Padina pavonia.

2.1.2. Chemicals

The chemicals used were of laboratory grade: sodium chlorite, sodium carbonate, hydrochloric acid, sulfuric acid, sodium hydroxide, sodium bisulfite, sodium acetate, acetic acid, and ethyl alcohol.

2.2. Methods

2.2.1. Extraction of sodium alginate

Two distinct methods were employed to extract sodium alginate from Egyptian brown algae: a) Pre-bleaching before extraction: Initially, the algae were ground and bleached twice for five minutes with sodium chlorite solution at concentrations of 1%, 1.5%, and 2%, maintaining a temperature of 98°C. The pH was adjusted to 4 using a buffer solution of sodium acetate and acetic acid in ratios of 1:7.5 and 1:10. The reaction was then halted using sodium bisulfite. The extraction process involved treating the bleached algae with 1% anhydrous calcium chloride for 15 minutes at room temperature using a liquid-to-solid ratio of 1:7.5, followed by filtration. Subsequently, the algae were treated with a 5% hydrochloric acid solution at room temperature for another 15 minutes with a liquid-to-solid ratio of 1:6, then filtered again and rinsed with water. This was followed by treatment with a 4% sodium carbonate solution at a ratio of 1:5 for two hours at 40°C, after which it was centrifuged. Ethyl alcohol was added to the filtrate to precipitate sodium alginate, which was then filtered and dried. b) Extraction followed by bleaching: The raw algae were first soaked in water at a 1:20 ratio for 10 minutes and then filtered. The residual material was treated with 0.2% sulfuric acid at a 1:15 ratio for 10 minutes, followed by another filtration. It was then treated with 3% sodium carbonate at a 1:12 ratio for two hours at 70°C. The bleaching step involved using 2% sodium chlorite at a pH of 3.5, adjusted with a sodium acetate and acetic acid buffer solution, maintained at 98°C for five minutes. Sodium bisulfide was used to stop the reaction after bleaching, and alginic acid was

precipitated. To convert the product into sodium alginate, it was treated with a 1 N sodium hydroxide solution in the presence of ethyl alcohol for 10 minutes at room temperature, followed by filtration. The residue was dissolved in water and centrifuged. Finally, ethyl alcohol was added to the filtrate to precipitate sodium alginate, which was filtered and dried.

2.3. Measurements and Analysis

2.3.1. Chlorine consumption

The consumption of chlorine, measured in mg Cl per gram of raw algae, was determined following a modified version of Whistler's procedure[11]. This involved assessing the reduction in hypochlorite concentration resulting from the oxidation of algae. This reduction is calculated as the difference between the concentration of a blank solution and that of the solution containing algae at the moment when the sample is removed from the hypochlorite solution.

2.3.2.Rheological Measurements

The rheological properties and apparent viscosity of the pure thickener, at a 3% concentration, were evaluated using a Rheomat-15 rotary viscometer from Zurich, Switzerland. The measurements were conducted under specific conditions. Initially, the shearing stress was recorded as the rate of applied shear gradually increased (upward direction). For each shear rate, the shearing stress was noted after 60 seconds. Subsequently, the shearing stress for the same sample was measured again as the rate of shear was reduced back down to the starting level (downward direction). The corresponding apparent viscosity was then computed using the following equation (1):

$$\eta = \tau / D \quad \ldots\ldots\ldots\ldots\ldots\ldots \quad (1)$$

Where: -η = apparent viscosity [poise].

τ= shearing stress [dyne/cm2]. D = rate of shear [S^{-1}].

3. Results and Discussion

Sodium alginate is obtained from brown algae, which also contains other ionic polymers like fucoidan. The crosslinking of these polymers with divalent ions likely helps maintain their presence within the seaweed matrix[11]. As discussed earlier, two techniques were used to extract sodium alginate from Egyptian algae: bleaching the algae prior to extraction or extracting first and then bleaching it, in order to achieve an ivory-colored sodium alginate.

3.1. The process of bleaching algae prior to extracting sodium alginate.

Chlorine dioxide (ClO_2) or related compounds ($HClO_2$ or $NaClO_2$) represent another major class of bleaching agents[14]. In this group, the chlorine is in a higher positive oxidation state than in the

hypochlorite–based bleaches. ClO_2 is usually produced by dissolving sodium chlorite in water to generate chlorous acid, which subsequently decomposes to ClO_2 or, more efficiently, by reacting sodium chlorite with chlorine gas[15].

$$NaClO_2 + H_2O \rightarrow HClO_2 + NaOH$$

$$5\ HClO_2 \rightarrow 4\ ClO_2 + HCl + 2\ H_2O$$

$$2\ NaClO_2 + Cl_2 \rightarrow 2ClO_2 + 2\ NaCl$$

The compounds or species that dominate in ClO_2-based bleaches vary depending on the pH level. In acidic conditions, ClO2 and HClO2 show the highest concentrations, whereas ClO_2– is more prevalent in alkaline environments. In practical applications, chlorite bleaching is usually performed within a pH range of 3.5 to 4.5. This range ensures minimal fiber damage and maintains a beneficial concentration of ClO_2. The chemistry involved in chlorite bleaching solutions is quite intricate, as the formation of different species depends on the sequence and rate at which reagents are added. When the pH falls below 3, excess production of ClO_2 can occur, posing safety and processing issues due to its toxic and corrosive nature[14]. In this study, algae bleaching was performed using varying concentrations of sodium chlorite (1%, 1.5%, and 2%) twice to achieve satisfactory whiteness. The pH was set to 4, and the process was conducted at 98°C for 5 minutes with a liquor ratio of 1:7.5. The extraction of sodium alginate from the bleached algae followed the procedure outlined in the experimental section. A paste of the extracted sodium alginate, at a 3% concentration in distilled water, was prepared to measure its rheological properties. The results are displayed in Figure 2, showing that all the samples exhibit non-Newtonian pseudoplastic behavior, with similar flow curves during both upward and downward measurements. Additionally, Figure 2 illustrates that as the sodium chlorite concentration increases from 1% to 2%, the rheograms shift further from the shear rate axis, indicating an increase in apparent viscosity.

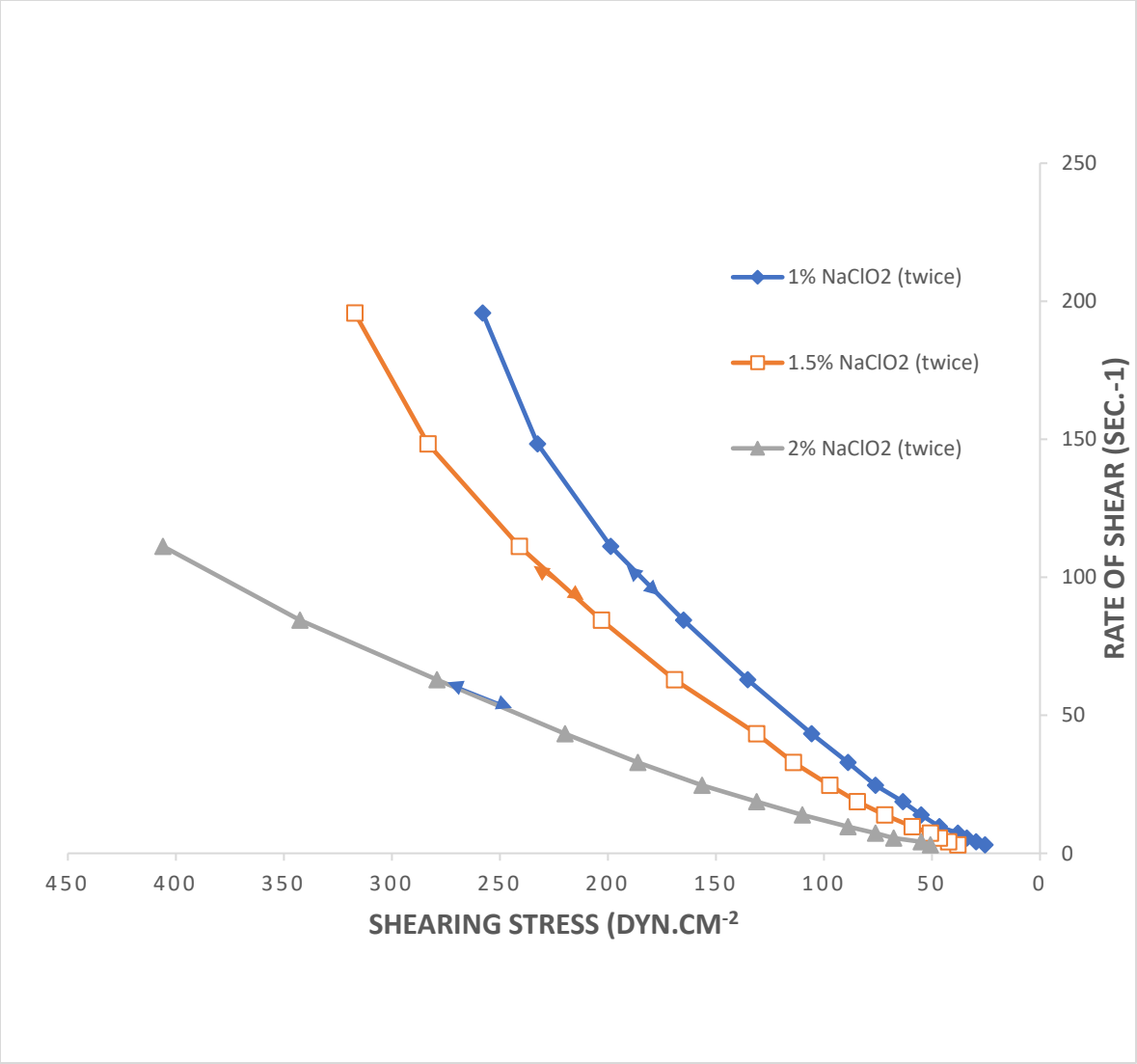


Figure 2: Illustrates how varying concentrations of sodium chlorite influence the rheological properties of sodium alginate samples, measured at a 3% concentration.

The apparent viscosity of all the sodium alginate pastes mentioned decreases as the shear rate increases, as shown in Table 1. However, the increase in apparent viscosity at the same shear rate varies depending on the concentration of sodium chlorite used. For instance, at a shear rate of 7.333 sec^-1, the apparent viscosity measures 519.03, 692.04, and 1038.07 centipoise with sodium chlorite concentrations of 1%, 1.5%, and 2% respectively, applied twice. Additionally, to achieve more bleached samples of sodium alginate, a similar series of experiments was conducted using a liquor ratio of 1:10.

Table 1: Impact of Sodium Chlorite Concentration* on the Apparent Viscosity of Extracted Sodium Alginate Samples at a 3% Concentration

Rate of shear Sec^{-1}	**Apparent viscosity in Centipoise**		
	1% $NaClO_2$ (twice)	**1.5% $NaClO_2$ (twice)**	**2% $NaClO_2$ (twice)**
3.111	815.62	1223.43	1631.24
4.177	708.7	1012.4	1316.18
5.495	615.68	846.5	1231.37
7.333	519.03	692.04	1038.07
9.673	480.91	612.07	918.112
13.94	394.38	515.75	788.766
18.72	338.86	451.8	700.31
24.63	309.06	394.91	635.29
32.86	270.26	347.48	566.27
43.35	243.88	302.42	507.28
62.93	215.04	268.8	443.53
84.5	195.18	240.22	405.38
111.2	178.74	216.77	365.09
148.3	156.84	191.06	-
195.7	131.81	162.07	-

*Concentration of sodium chlorite is 1, 1.5, and 2% twice for 5 min. at 98 ^{0}C at pH4 and L.R. 1:7.5.

The results are presented in Table 2 and illustrated in Figure 3. The data indicate that the rheological characteristics and apparent viscosity of the samples with a liquor ratio of 1:10 are almost identical to those with a liquor ratio of 1:7.5. However, the samples prepared with a 1:10 ratio exhibit a whiter color compared to those using a 1:7.5 ratio. The increase in apparent viscosity with higher concentrations of sodium chlorite can be explained as follows: at lower concentrations, the sodium alginate still contains impurities. As the concentration of the bleaching agent, sodium chlorite, rises, these impurities are eliminated, leading to a higher percentage of active sodium alginate in the product, which results in increased viscosity.

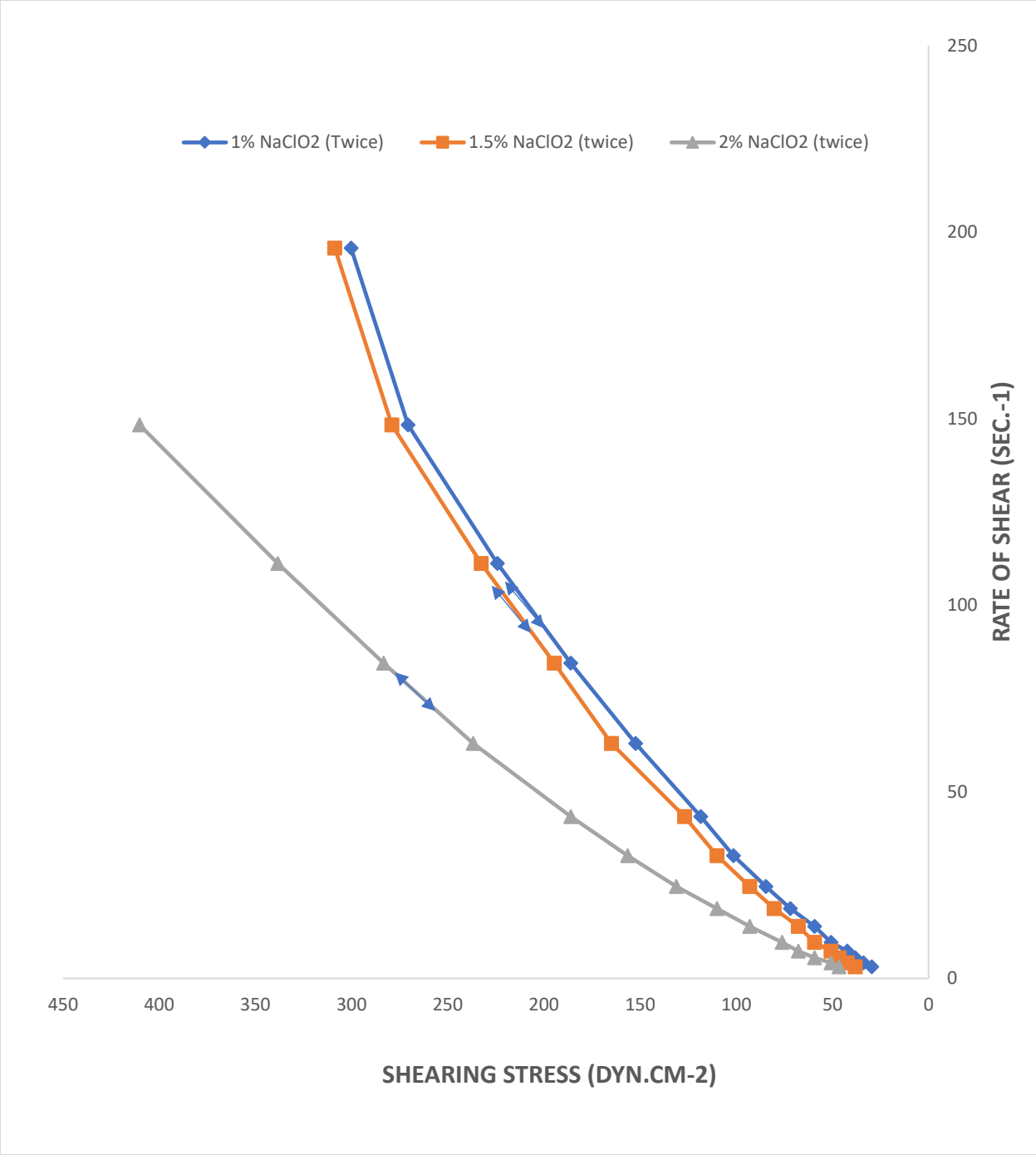


Figure 3: Shows the effect of varying sodium chlorite concentrations on the rheological properties of sodium alginate samples extracted at a 3% concentration.

Table2: Influence of sodium chlorite concentration* on the apparent viscosity of the extracted sodium alginate samples at a 3% concentration.

Rate of shear Sec^{-1}	Apparent viscosity in Centipoise		
	1% $NaClO_2$ (twice)	1.5% $NaClO_2$ (twice)	2% $NaClO_2$ (twice)
3.111	951.55	1223.43	1495.3
4.177	809.95	1012.4	1214.9
5.495	692.64	846.5	1077.4
7.333	576.7	692.04	922.73
9.673	524.63	612.02	786.95
13.94	424.72	485.39	667.4
18.72	384.04	429.22	587.36
24.63	343.4	377.74	532.27
32.86	308.87	334.613	476.17
43.35	273.15	292.66	429.24
62.93	241.92	262.08	376.33
84.5	220.2	230.21	335.31
111.2	201.56	209.168	304.24
148.3	182.5	188.2	276.61
195.7	153.42	157.75	

*Concentration of sodium chlorite is 1, 1.5, and 2% twice for 5 min. at 98 0C at pH4 and L.R. 1:10.

3.2. Extracting sodium alginate and subsequent bleaching

In earlier research, algae underwent bleaching prior to the extraction of sodium alginate. In this study, sodium alginate was extracted from raw algae before bleaching was carried out using either sodium hypochlorite or sodium chlorite. The method for extraction from raw algae is outlined in the experimental section. The extracted sodium alginate samples were bleached using sodium hypochlorite (10 g active chlorine per liter for five minutes at room temperature, L.R., 1:20, at pH 3.5) or sodium chlorite (2% sodium chlorite for five minutes at 98 °C, L.R., 1:10, at pH 3.5). Extracted samples were prepared into pastes at a concentration of 3%, and their rheological properties were assessed. The outcomes are presented in Table 3 and depicted in Figure 4. Figure 4 illustrates that the samples exhibit non-Newtonian pseudoplastic behavior, with coinciding up and down flow curves. Table 3 provides a detailed view of how shear rate

affects the apparent viscosity of the alginate pastes. The data indicate that the apparent viscosity of both sodium alginate pastes diminishes as the shear rate increases. However, the type of bleaching agent also influences the apparent viscosity. For instance, at a shear rate of 6.779 sec^{-1}, the apparent viscosity is 12,520 centipoise with sodium hypochlorite and 8,481.7 centipoise with sodium chlorite.

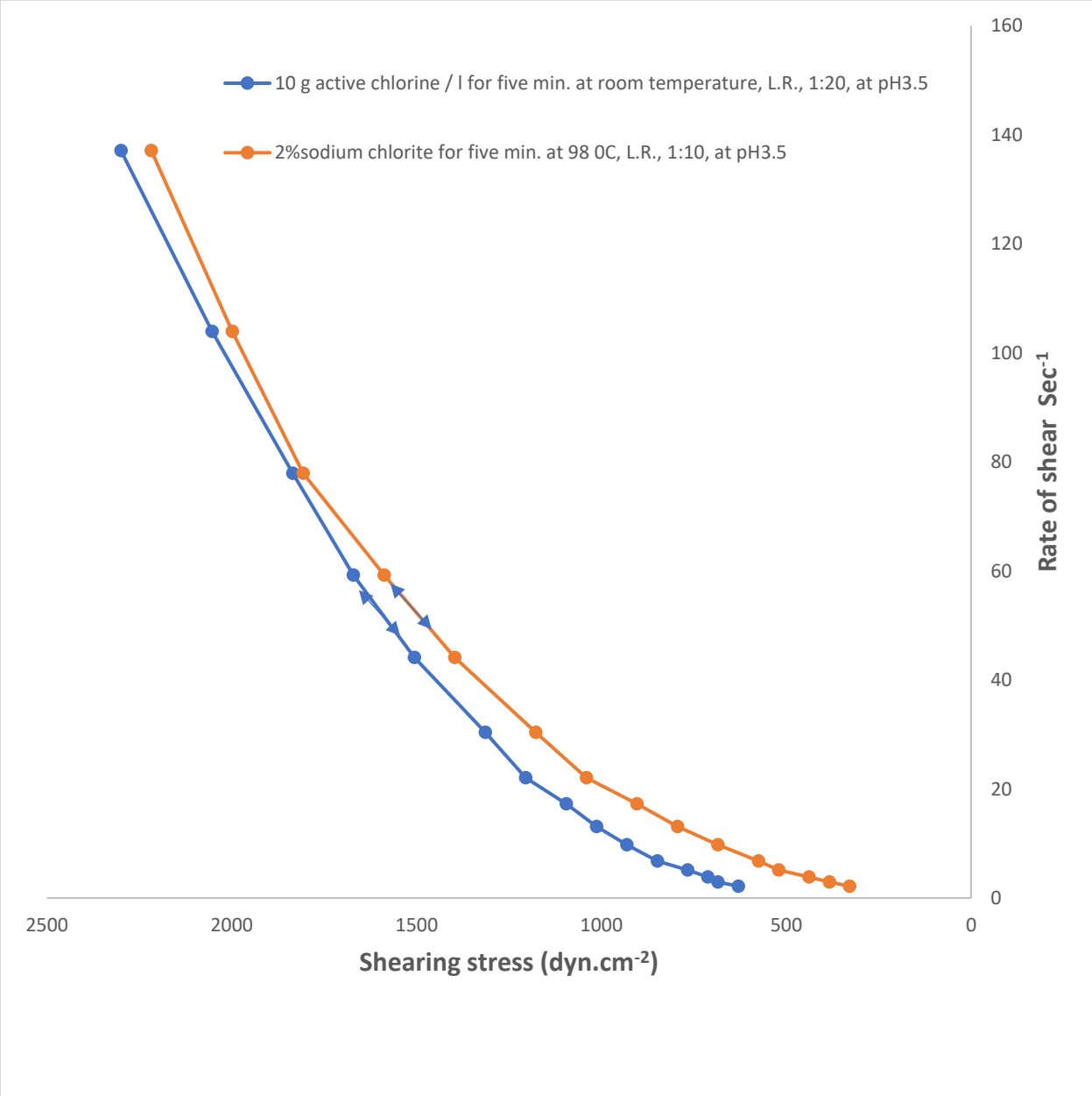


Figure4: Effect of the nature of the bleaching agent on the rheological properties of the extracted sodium alginate samples at a 3% concentration.

Table 3: Influence of the bleaching agent type on the rheological behavior and apparent viscosity of sodium alginate samples at 3% Concentration.

Rate of shear Sec^{-1}	Shearing stress ($dyn.cm^{-2}$)				Apparent viscosity in Centipoise	
	10 g active chlorine/l		2% g $NaClO_2$		10 g active chlorine/l	2% g $NaClO_2$
	upward	downward	upward	downward		
2.18	629.74	629.74	328.56	328.56	28887	15071.5
2.927	684.5	684.5	383.32	383.32	23442	13127.4
3.851	711.88	711.88	438.08	438.08	18485	11375.7
5.139	766.64	766.64	520.22	520.22	14918	10122.98
6.779	848.78	848.78	574.98	574.98	12520	8481.7
9.771	930.92	930.92	684.5	684.5	9527	7005.4
13.12	1013.06	1013.06	794.02	794.02	7721	6051.98
17.26	1095.2	1095.2	903.54	903.54	6345	5234.8
22.03	1204.72	1204.72	1040.44	1040.44	5231	4521.68
30.38	1314.24	1314.24	1177.34	1177.34	4326	3875.3
44.1	1505.9	1505.9	1396.38	1396.38	3415	3166.39
59.22	1670.8	1670.8	1588.04	1588.04	2820	2681.59
77.92	1834.48	1834.48	1807.08	1807.08	2354	2319.1
103.9	2053.5	2053.5	1998.74	1998.74	1976	1923.7
137.1	2299.92	2299.92	2217.78	2217.78	1677	1617.6

Upon comparing the two techniques, namely bleaching the algae before or after extraction, the data reveals that extracting sodium alginate from the algae before bleaching results in products with higher viscosity compared to the samples produced with the alternate method. This observation is consistent regardless of which bleaching agent is employed. The increased viscosity in samples prepared by extraction followed by bleaching can be explained as follows: In the first method, involving bleaching before extraction, the bleaching process needs to be repeated twice to achieve a color similar to commercial sodium alginate samples. In contrast, the second technique requires only a single, shorter bleaching process, subjecting the alginate chains to milder conditions and resulting in a product with higher viscosity. It is worth recommending sodium hypochlorite as a bleaching agent instead of sodium chlorite due to its safety

and cost-effectiveness. For comparative purposes, two commercial sodium alginate samples with varying viscosities—alginate TBVV/N2 (low viscosity) and Cecalginate HV/600 (high viscosity)—were chosen. Their viscosity at various shear rates was compared with laboratory-prepared sodium alginate samples, all at a 3% concentration as detailed in Table 4. The data from Table 4 indicates that the apparent viscosity of the sample prepared using hypochlorite bleaching followed by extraction (sample No. I) is nearly equivalent to that of commercial low-viscosity sodium alginate. Conversely, the sample prepared by extraction followed by sodium chlorite bleaching mirrors the viscosity of commercial high-viscosity sodium alginate. Thus, it can be concluded from these findings that the method of preparing sodium alginate from raw algae significantly impacts the apparent viscosity of the resulting sodium alginate sample.

Table 4: Presents a comparison of the apparent viscosity between commercially available and laboratory-extracted sodium alginate samples, measured at a 3% concentration across various shear rates.

Rate of shear Sec^{-1}	**Apparent viscosity in Centipoise**		**Rate of shear Sec^{-1}**	**Apparent viscosity in Centipoise**	
	commercial low viscosity sodium alginate sample	**Laboratory extracted sample No. I***		**commercial high viscosity sodium alginate sample**	**Laboratory extracted sample No. II***
9.673	349.75	393.47	6.779	6058.41	8481.7
13.94	333.71	333.71	9.771	5324.12	7005.4
18.72	316.27	293.68	13.12	4799.84	6051.98
24.63	309.06	274.72	17.26	4283.08	5234.8
32.86	283.13	244.52	23.03	3804.42	4521.68
43.35	263.39	224.37	30.38	3334.63	3875.3
62.93	248.64	201.6	44.1	2855.96	3166.39
84.5	230.21	185.17	59.22	2450.4	2681.59
111.2	212.97	171.137	77.92	2143.45	2319.1
148.3	196.76	159.69	103.9	1844.65	1923.7
195.7	172.87	136.14	137.1	1577.69	1617.6

***I** Via sodium hypochlorite bleaching, followed by extraction**.**

****II** Via extraction followed by sodium chlorite bleaching

4. Conclusion

Sodium alginate can be extracted from brown algae using two primary methods: the first involves bleaching the algae before extraction, while the second extracts it first, then bleaches it. The second approach results in a product with higher viscosity. Furthermore, the characteristics of the final products and the apparent viscosity of the pastes are affected by the choice of bleaching agent. For example, at a shear rate of 6.779 sec-1, the apparent viscosity measures 12,520 centipoise when using sodium hypochlorite, compared to 8,481.7 centipoise with sodium chlorite. It is advised to use the extraction followed by the bleaching method with sodium hypochlorite, as it is both safer and more cost-effective than using sodium chlorite. The resulting products exhibit non-Newtonian pseudoplastic behavior. These results indicate that the production method for sodium alginate from raw algae significantly influences the apparent viscosity of the final product.

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Declaration section

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Data Availability

The datasets used and/or analyzed during the current study are available from the corresponding author upon reasonable request.

Author contribution statement

Prof. Dr. Mohamed M. El-Molla and Prof. Dr. Amira A. Ragheb proposed the idea, conducted the experiments, recorded the results, and prepared and wrote the research, while Prof. Dr. Madiha A. El-Kashouti reviewed it.

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