



Extraction and characterization of *Cystoseira compressa*-derived alginate, collected from reef coral region of Red sea, Egypt

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Abstract: Alginate was extracted as sodium alginate from the brown algae *Cystoseira compressa* which collected from reef coral of tidal region along Red sea coast of Egypt. Fourier transformed infrared (FTIR) spectroscopy revealed the presence of O-H, COO-, C=O chemical functions groups indicating the presence of mannuronic and uronic acids. Thermogravimetric-differential thermal analysis (TGA) and differential thermal analysis (DSC) exhibited the thermostability of alginate. DSC analysis revealed its semi crystalline nature characterized by endothermic behavior with onset temperature 651.0° C. Rheological study revealed that viscosity of alginate solutions (0.125, 0.250 and 0.500 mg/ml) as a function of shear rate reached maximum values of 8.03, 12.1, 22.1 cP viscosity respectively at shear rate of 40 S-1.

keywords: *Cystoseira compressa*, DSC, FTIR, Rheology, Sodium alginate, TGA

1. Introduction

Seaweeds represent a significant sustainable resource in aquatic ecosystem. Red Sea tidal coastal region extend about 1200 Km at the east board of Egypt, and enriched with macroalgae. Macroalgae introduce a potential source of bioactive components where phaeophytes contain large anionic polysaccharides in their cell walls predominated by alginates which contain β -D-mannuronic acid (M) with and α -L-guluronic acid (G). Currently the biotechnological application of alginate are used in textile, food industry, pharmaceutical industry and biomedical industries, according to alginate bio-compatibility, reduce toxicity in addition to low cost [1, 2]. Alginates are anionic polymer, can interact with many divalent ions to form "egg-box" junctions [3]. Alginate extraction is a process of two phases. Firstly, transforming the insoluble alginate to soluble sodium alginate then glycuronan is diffused solution [4, 5].

2. Materials and methods

Collection, identification of *Cystoseira compressa* samples

Cystoseira compressa was gathered from the intertidal zone of Red Sea at the coastal

area of Safaga, Egypt. Algal samples were washed carefully, then dried at 60°C and crushed to small particles.

Extraction of alginate

Extraction was performed after the protocol of [6] with some modifications. 10g dry algal biomass was boiled in 150 ml distilled water for 30 min. After filtration, algal biomass was mixed with 150 ml 0.5% CaCl₂ solution and boiled for 30 min. Then, the aqueous phase was decanted and the biomass residue was boiled with 150 ml of 0.5% NaCl solution for 1 h. After removing supernatant, 50 ml of 3% Na₂CO₃ solution was added to the algal residue with stirring for 30 min at 100°C then sodium alginate was precipitated by ethyl alcohol.

Characterization of Alginate

Fourier Transform Infrared (FTIR) spectroscopy analysis

The FTIR profile of alginate sample was determined using Shimadzu FTIR-8400 S in the range of 4000-500 cm⁻¹ at 1 cm⁻¹ resolution.

Thermogravimetric analysis (TGA)

TGA was calculated by a thermo analyzer of the type 50-H in the range of 25°C-1000°C.

Differential scanning calorimetric (DSC) analysis

The DSC was measured using NETZSCH STA 409° C/CD thermo gravimetric analyzers. Alginate sample 10.0 mg was heated under helium atmosphere at a rate of 10° C/min within a temperature of 15° C to 1000° C. The graph was plotted with heat flow versus temperatures.

Rheology measurement

The cone-plate geometry on a stress-controlled rheometer [7] was used for the dynamic rheological measurements of the extracted alginate solutions at 25°C.

3. Results and Discussion

Fourier transformed infrared (FTIR) spectroscopy analysis.

FT-IR absorption bands (cm^{-1}) of alginate, chemical functional groups were identified according to [8]. Data demonstrated pattern of chemical function groups dominated with absorption beaks at 3464, 2925 and 2654 cm^{-1} of both O-H and N-H stretching vibrations designating alcohols, phenols, carboxylic group and primary amines, respectively. Spectral peaks ranged from 3400 cm^{-1} to 3600 cm^{-1} could be assigned to alcohol and acids [9].

Thermal properties:

The thermograph (Fig.2a) demonstrates the outline of degradation of alginate that followed in three overlap steps. Firstly, step included 10.65% mass loss of alginate by raising temperature to 587.89°C. The secondary step was recognized by progressive mass loss reached 48.19% at temperature 748.89°C. The final step was characterized by weight loss of 3.50% at 988.89°C.

The DSC profile (fig. 2b) exhibited two notable endothermic peaks illustrated the crystallization temperature T_c of alginate; first peak T_{c1} has one tip at 112° C with onset point 107.9° C that may be contributed to water loss. Second sharp peak T_{c2} exhibits one tip at 727.1° C at a fixed period at 651.0° C exhibiting the fractional crystalline nature of alginate which could be attributed to the

melting temperature of alginate [10]. DSC profile recorded enthalpy (latent heat) values - 0.2848, -344.0 $\mu\text{V s/mg}$, respectively.

Rheological characteristic

At shear rate 40 S^{-1} , the viscosity of alginate solutions (0.125, 0.250 and 0.500 mg/ml) recorded maximum shear rate of 8.03, 12.1, 22.1 centipoises (cP), respectively (fig. 3).

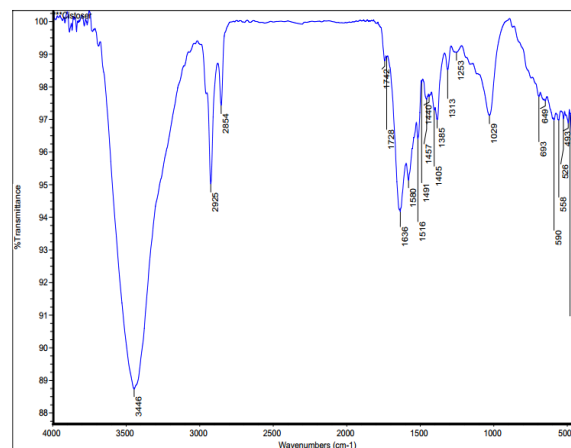


Fig. 1 Fourier transformed infrared (FTIR) spectroscopy analysis

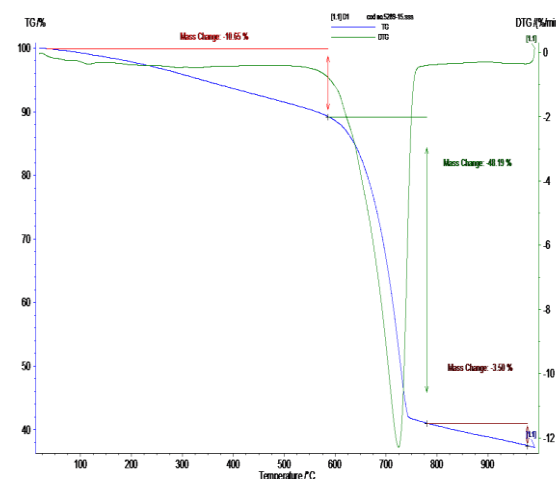


Fig. 2a Thermal gravimetric analysis of alginate (TGA)

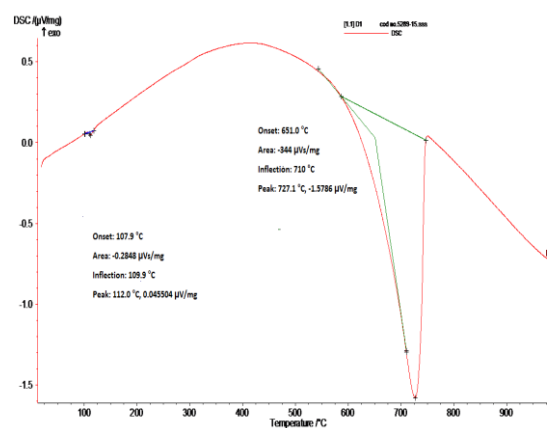


Fig. 2b Differential scanning calorimetric of alginate (DSC)

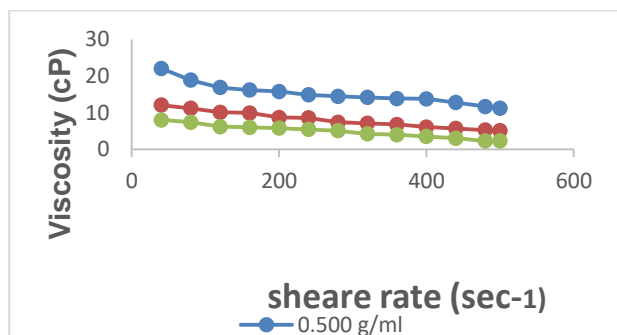


Fig. 3 Viscosity as a function of shear rate of *Cystoseira* alginate solutions(0.125, 0.250 and 0.500 g/ml)

4. Discussion

FTIR profile illustrated spectral band of C-H stretching vibrations assigned to alkanes, C=O indicated carbonyl group (amide I band), COO⁻ stretching vibrations assigned carboxylate in addition to C-O-C stretching vibrations [11, 12]. Moreover, the two spectral bands allocated at 884 cm⁻¹ and 939 cm⁻¹ referring to mannuronic and uronic acids functional groups respectively.

Thermogravimetry is a technique adopted to study the thermal properties by increasing temperature at a fixed interval, and the mass loss is documented for investigating the thermal stability. Alginate structure mainly composed of carboxylate groups in the polymer matrix and it is subjected easily to decarboxylation [13]. This showed the direct interaction between COO⁻ groups and the attraction of alginate for binding water molecules [14]. By the end of the third step, alginate depolymerization was detected which might be ascribed to the breakdown of C-O and C-C bonds in the ring units with evolution of CO, CO₂ and H₂O as suggested by [15]. Steady stages were recognized when temperature raised above 790° C before of the final mass loss that could be according to decomposition of the remains of the organic component of alginate and /or the constitution of polynuclear aromatic and graphitic carbon structure [16].

Alginate mass loss detected in the first step could be contributed to water dehydration [16-18]. Whereas, mass loss of the second step could be attributed to the allocation of COO⁻ groups which bind water molecules for stimulating this thermal behavior as designated previously by [14] So, the alginate

thermograph illustrated total mass loss about 62.34% which confirmed its relative stability nature [14]. Alginate thermogram led to the conclusion that alginate exhibited strong level of thermal stability revealing 62.34 % total mass loss and 37.66 % residual mass. [19]. The pronounced thermal stability detected for alginate in addition to high residual contents could be interpreted on basis of its complicated and heterogeneous nature containing high sulphate and uronic acid.

Differential scanning calorimetry (DSC) is an efficient thermo analytic technique for investigating the heat quality needed for characterizing the thermal behavior of natural polymer. Studies of [20] and [21] indicated that degradation and burning of carbon residues from alginate polymer was detected at temperature above 500° C. Prolonged heating via increasing temperature above 650° C resulted in ash formation due to carbonization of alginate backbone [22, 23]. The decomposition of Alginate could be expected with following appearance of this endothermic peak. Exposing amorphous polymer to increasing temperature resulted in decreasing its viscosity and at a certain temperature the polymer molecules attain adequate freedom for changing to rebuilding their molecules spontaneously into a crystalline pattern whereas this temperature is identified as crystallization temperature as explained by [24] and [25].

While with increasing shear rate to 500 S⁻¹, viscosity values decrease dramatically to 2.3, 5.1, 11.2 cP respectively [26].

Conclusion

The Egyptian *Cystoseira compressa* derived alginate could be introduced as good candidate for a thermostable phycocolloid could be used in many biotechnological applications.

4. References

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