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Properties of *Chaetomorpha Antennina* Gel in Presence of Ionic and Non-Ionic Surfactant

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INTRODUCTION

Biopolymers remain to be an interesting class of substances since they are used in industry to provide structure stability (Lewis, 1988). Agar and carrageenans, the phycocolloids obtained from many species of red seaweed (Rhodophyta), are industrially important for their excellent thickening or gelling abilities (Selby, 1993; Therkelsen, 1993). Tanaka *et al.*, (2000) have reported the surfactant mediated thermo-reversible gelation with multiple crosslinking junctions in associating polymer hydrogels (Tanaka, 1998a; Tanaka, 2000; Tanaka, 1998b). Interaction of polymers and surfactants in aqueous solution has been studied by many researchers to explore the potential applications in chemical, petrochemical and pharmaceutical industries (Maltesh, 1990)**.** It has been widely reported that surfactants can influence gelling characteristics of polymers in solution (Prasad *et al.*, 2005). Ermeneko *et al.*, (2001) reported the interaction of κ-carrageenan gel with an ionic surfactant that leads to the formation of an ordered structure. It is also reported that surfactants play an important role in promoting the relation of associating polymers (Kundu, and Kundu, 2001).

In this study, the gelling properties, apparent viscosity, and thermal properties of sulphated polysaccharide of *Chaetomorpha antennina* (CM_{sps}) sol and CM_{sps} gel in presence of various cationic, anionic and non-ionic surfactants were investigated. The surfactants used were sodium lauryl sulphate (SLS), brij35, cetylpyridinium chloride (CPC), cetyltrimethyl ammonium bromide (CTAB), crexlox 4896, crexlox 894, crexlox 895. This investigation was done to evaluate the application potential of this particular hydrogel.

MATERIALS AND METHODS

Collection

Chaetomorpha antennina was collected in February2005 to June 2005 from Diu (020° 042.364’ N, 070° 058.276' E), Veraval (20° 54.932’ N and 070° 20.842’ E) in the west coast of India and Kanyakumari (08° 04 .822’ N, 077° 33.098' E), at southeast coast of India, from inter-tidal zone.

Extraction and purification

Extraction and purification of polysaccharide from depigmented algal powder was done by Siddhanta *et al*., 2001. The 0.5 M eluates containing major amount of polysaccharides were the charged ones (CM_{sps}), which were saved and used for subsequent studies (cf. Siddhanta *et al*., 2001).

Materials

The purified sulphated polysaccharide of *Chaetomorpha antennina* (CM_{sps}) was used for all the experiments. The effects of various surfactants on CM_{sps} sol were studied. Sodium lauryl sulfate (SLS; anionic), cetyl pyridinium chloride (CPC; cationic), brij 35 (non-ionic), and cetyl trimethyl ammonium bromide (CTAB; cationic) were procured as LR Grade surfactants from S.D. Fine Chemicals, Mumbai, India. Ceralox-4896 (non-ionic) ceralox-895 (anionic) and ceralox-894 (non-ionic) were received as gifts from M/s ICI Uniqema, Thane, Mumbai, India. The surfactants were used as received without further purification.

Gel preparation and gel strength measurement

Fifty milliliters of 2% CM_{sps} sol were prepared in a 50ml beaker and the different surfactants were added to the hot sol in varying concentrations (0.1%-0.4%), stirred well, and boiled to ensure complete dissolution. Maximum water loss during boiling was 4% and this loss was compensated by adding the required quantity of hot water into the sol with stirring. The beaker was then covered and allowed to cool to room temperature to form a gel. After the formation of gel at room temperature (Figure 1), the gel was kept overnight at 10°C. Gel strength was measured at 25°C using a Nikkansui-type gel tester (Kiya Seisakusho Ltd., Tokyo, Japan).

Figure II.3.1 Control CM_{sps} (2%) gel

Gelling and melting temperature

Gelling and melting temperatures of CM_{sps} gel in presence of various surfactants were measured in the mixtures as described by Craigie *et al.*, (1978). Measurement of pH was carried out using a Systronics 535 pH meter (Systronics Scientific Instruments, India).

Apparent viscosity

The apparent viscosity of CM_{sps} gel was measured at 80°C using a Brookfield viscometer (Synchrolectric viscometer, Stoughton, MASS02072). Spindle No. 3 with a speed of 60 rpm was used for measuring apparent viscosities.

Differential scanning calorimetry (DSC)

DSC measurements were carried out on a Mettler Toledo DSC Analyzer, DSC/SDTA 851e, Switzerland. For this purpose, approximately 10 mg gel was taken in an aluminum crucible weighing (g) and the measurements were done using a temperature program of 30-200°C at 5°C min^{-1} heating rate in an air atmosphere.

RESULTS AND DISCUSSION

Physical properties

Several cationic, anionic, and non-ionic surfactants were studied for their influence on CM_{sps} sol and gel properties and the results are shown in Table 1. In presence of cationic surfactant, complex formation (Figure 2), presumably due to the interaction of anionic sulfate residue and cationic residue of the surfactant was observed resulting in breakage of gel (Figure 2) with increasing concentration of surfactants. This breakage of gel, however, was not observed in the presence of non-ionic and anionic surfactants. An increase in viscosity was observed in the presence of an increasing concentration of ionic surfactants (Table 2). Similar trends in the variations of viscosity, gel strength, gelling, and melting temperatures were observed with non-ionic surfactants, with different degrees (Table 1). The extent of deviation was, however, not the same with all ionic surfactants. Enhancements were most pronounced with SLS and Ceraox-895. Unlike in the case of other ionic surfactants, the gelling temperature was found to decrease markedly. In contrast to the behavior of ionic and non-ionic surfactants reduced the melting temperature, gelling temperature, and gel strength of the CM_{sps} (Table 1). The gel strength was sensitive to the application level of the surfactants; the gelling temperature was less sensitive to variations in surfactant concentration for most of the surfactants studied.

Figure II.3.2 CM$_{sps}$ (2%) gel with cationic surfactant (CPC)

Table 1 Effect of surfactants on the physical properties of CM$_{sps}$ gel

Surfactant	Nature	Concentration of Surfactant^{a} (%)	Gel strength^{b} (gcm^{-2})	pH (at 80^{o}C)	T$_{gel}$ (^{o}C)^{c}	T$_{m}$ (^{o}C)^{e}
Nil	Control CM$_{sps}$ (2%)	-	330	6.9	78	88
SLS	Anionic	0.1	250	7.6	74	87
		0.2	270	7/7	73	88
		0.3	300	7.7	73	88
		0.4	**350**	7.7	73	**89**
Ceralox-895	Anionic	0.1	330	7.4	74	88
		0.2	330	7.5	77	88
		0.3	350	7.5	77	89
		0.4	**360**	7.6	78	**89**
CPC	Cationic	0.1	180	7.5	76	85
		0.2	170	7.5	77	85
		0.3	160	7.9	77	88
		0.4	**150**	8.1	78	89
CTAB	Cationic	0.1	< 100	8.2	65	86
		0.2	< 100	8.4	66	86
		0.3	< 100	8.6	66	88
		0.4	< 100	8.6	66	89
Brij-35	Non-ionic	0.1	330	7.1	77	86
		0.2	300	7.2	79	86
		0.3	270	7.2	83	85
		0.4	260	7.3	84	84
Ceralox-4896	Non-ionic	0.1	280	7.3	66	86
		0.2	270	7.3	66	86
		0.3	260	7.4	67	85
		0.4	240	7.4	67	84
Ceralox-894	Non-ionic	0.1	280	7.4	76	81
		0.2	280	7.3	76	81

		0.3	270	7.4	77	80
		0.4	250	7.4	77	80

[a] In all cases concentration of surfactant is with respect to the volume of gel of 2% CM_{sps}; SLS: sodium lauryl sulfate; CTAB: cetyl trimethyl ammonium bromide; CPC: cetyl pyridinium chloride; [b] Gel strength was measured in 2% gel at 25°C; [c]Variation in T_{gel} (Craigie's method) was ±1°C; [d] Variation in T_m (Craigie's method) was ±1°C.

Differential scanning colorimetric measurement

To study the thermal stability of CM_{sps} gel in presence of surfactants, DSC measurements was carried out on 2% CM_{sps} gel with (0.4%, w/v) and without different ionic and non-ionic surfactants. Figure 3 shows the DSC heating curves of approximately 10 mg of (a) control CM_{sps} gel. It can be seen from the Figure 3 that the control CM_{sps} gel exhibits one endotherm with peaks at 108°C. A plot with one endotherm like that of control CM_{sps} gel is seen with the anionic, cationic and non-ionic surfactant at 112°C, 108°C and 110°C respectively (Figure 4, Figure 5 and Figure 6). The DSC results for the gels containing surfactants are in reasonable accord with the corresponding values of gel melting temperature (Tm) in Table 1.

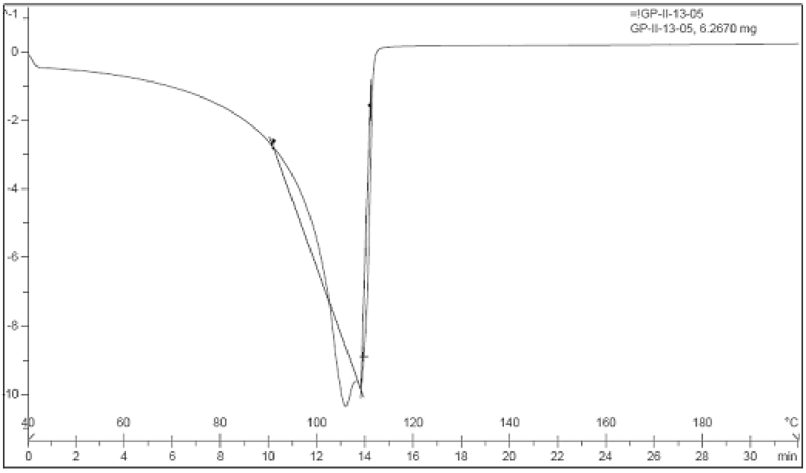

Figure II.3.3 DSC heating curve of native CM_{sps} (2%) gel

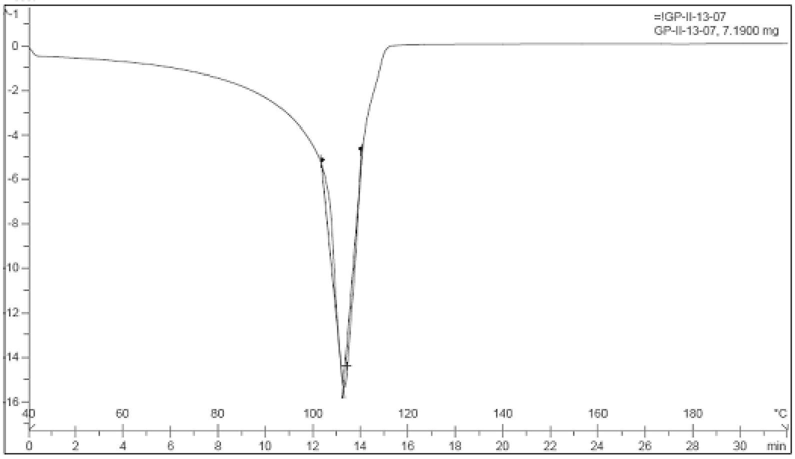

Figure II.3.4 DSC heating curve of CM_{sps}-anionic surfactant (SLS)

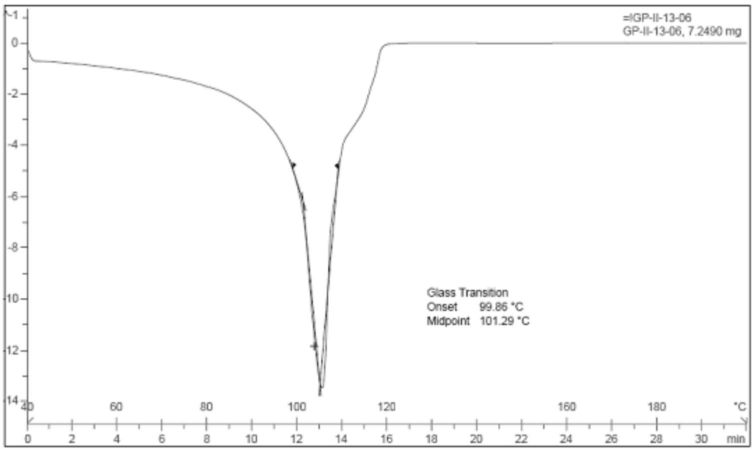

Figure II.3.5 DSC heating curve of CM_{sps}-cationic surfactant cationic (CPC)

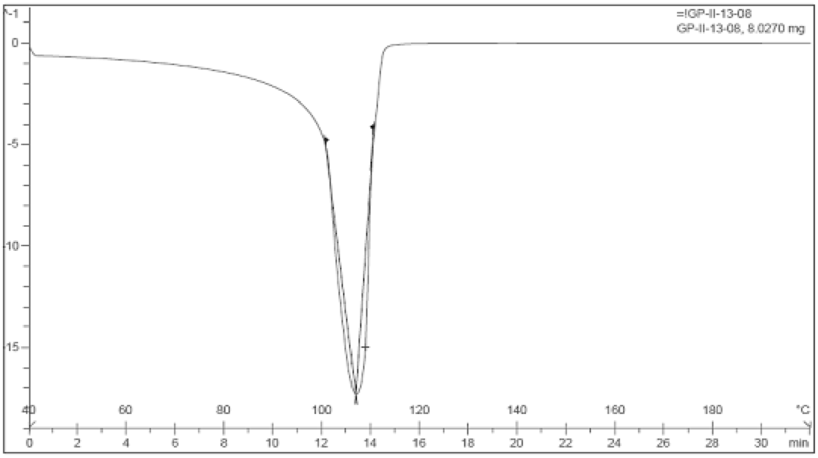

Figure II.3.6 DSC heating curve of CM_{sps}-non-ionic surfactant (Brij-35)

Viscosity measurement

The apparent viscosities of control CM_{sps} as well as CM_{sps} containing different surfactants were measured (Table 2). The viscosity of CM_{sps} sol was sensitive to the concentration of surfactant. The apparent viscosity increased in the presence of anionic surfactant (0.4%w/v) while in the presence of cationic and non-ionic surfactant viscosity decreased (Table 2).

Table 2 Effect of various surfactants (0.4% w/v) on the apparent viscosity of CM_{sps} at 80°C

Surfactant (%w/v)	Nature of surfactant	Viscosity (cPs)
Control CM_{sps} (2%)[a]	-	240
SLS	Anionic	300
Ceralox-895	Anionic	320
CPC	Cationic	200
CTAB	Cationic	200
Brij-35	Non-ionic	230
Ceralox-4896	Non-ionic	230
Ceralox-894	Non-ionic	240

[a] Value for control CM_{sps} gel (in absence of surfactant)

CONCLUSION

It has long been known that certain surfactants cause gel inhibition in the gelling material resulting in a lowering of viscosity. In the present study, it has been demonstrated that anionic surfactants enhance CM_{sps} sol viscosity and also the stiffness of the gel. The gelling and melting temperatures got enhanced upon the incorporation of cationic (CPC and CTAB) and anionic (SLS & Ceralox-895) surfactants. Cationic surfactants, on the other hand, lowered the viscosity of CM_{sps} sol and the stiffness of the gel. Moreover, non-ionic surfactants (Brij-35, Ceralox-4896 & Ceralox-894) also reduced the gelling and melting temperatures. Cationic surfactants showed significant influence on gel strength even concentration range of 0.1-0.4% (w/v). The gelling and melting temperatures are less sensitive to variations in surfactant concentration at 0.1% level. Differential scanning calorimetric studies provided clear evidence of one endotherm in the DSC profile of native CM_{sps} gel. In presence of surfactants, the profiles altered marginally. Cationic surfactants lowered the sol viscosity, which would allow for easier filtration of concentrated extractives, thereby helping to substantially reduce water usage in the process. Further, the lowering of the gelling temperature minimizes the possibility of accidental gelling of hot extractives during work-up operations. When the conventional methods of purification of polysaccharides e.g. freeze-thaw or pressure syneresis, were applied to CM_{sps}, the removal of the surfactant from the polysaccharide was found to be feasible. This would help restore the pristine quality of the starting material. This provides an example of the physical modification of a polysaccharide. The useful effects in presence of surface-active agents described herein may be beneficially used for certain targeted applications of this polysaccharide gel.

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