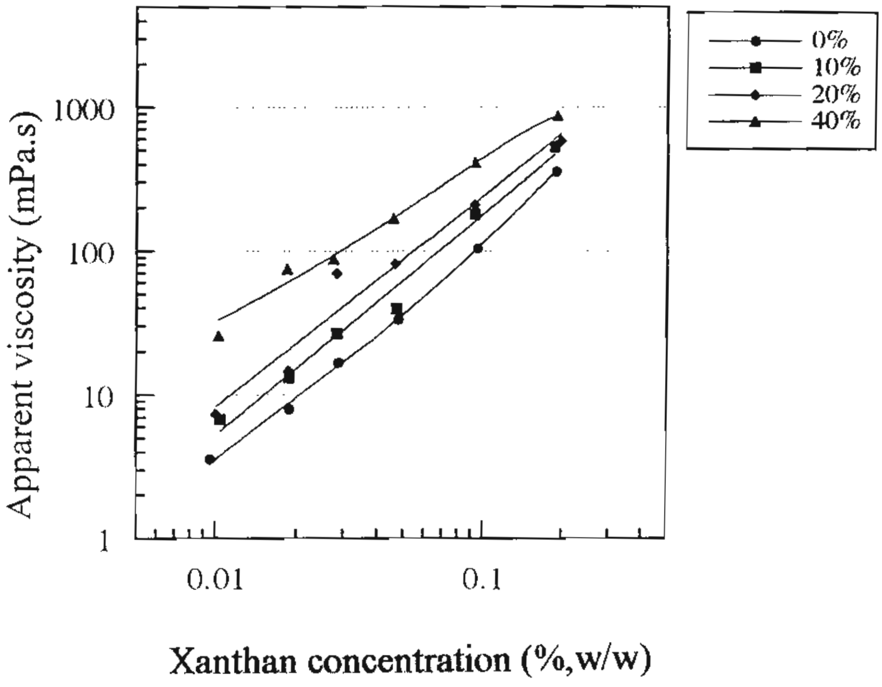


Fig. 3.33 Effect of sucrose concentration on apparent viscosity of xanthan solutions as a function of concentration at 25°C.

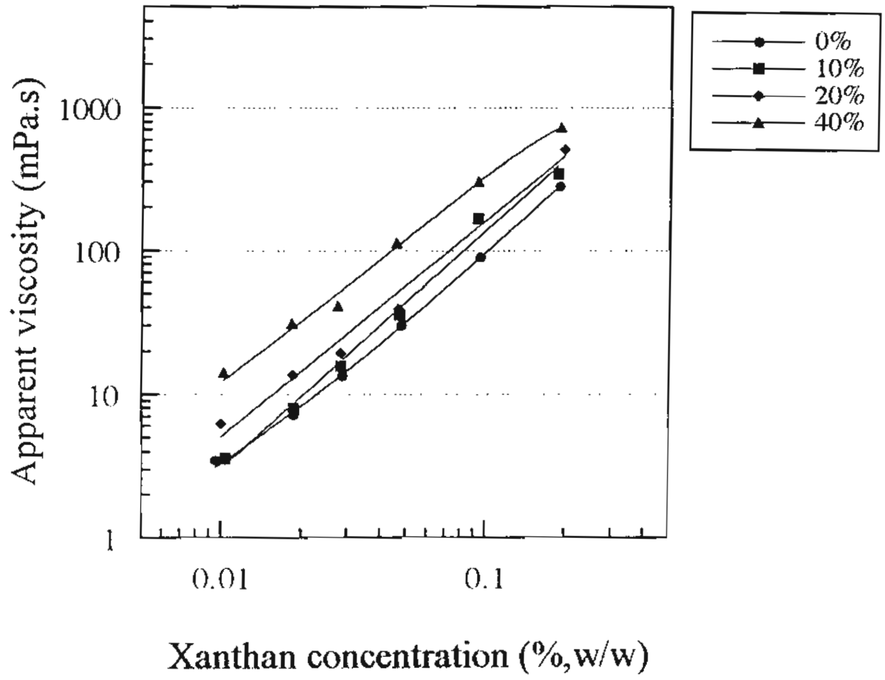


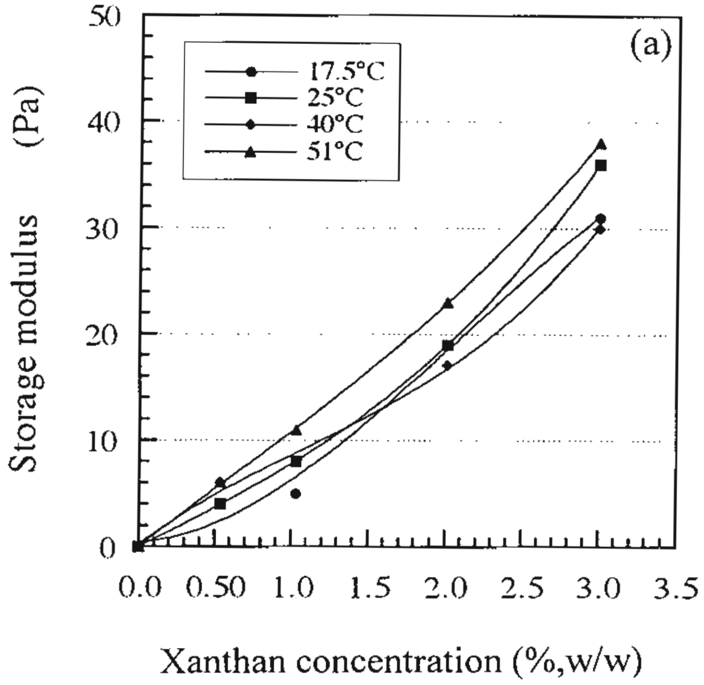
Fig. 3.34 Effect of sucrose concentration on apparent viscosity of xanthan solutions as a function of concentration at 40°C.

ซึ่งวัดส่วนที่เป็น elastic ของระบบแซนแทน/ซูโครส/น้ำ หรือแสดงระดับของการเป็นของแข็งของระบบโดยบันทึกว่าเป็นปริมาณพลังงานที่เก็บไว้ในระบบต่อรอบ และค่า loss modulus (G″) ซึ่งวัดส่วนที่เป็น viscous ของสารละลายและบันทึกเป็นพลังงานที่สูญเสียไปต่อรอบ (Rao, 1992; Ross-Murphy, 1994)

ในสารละลายแซนแทน จะมีค่า G′ และ G″ มากขึ้น เมื่อความเข้มข้นของแซนแทนเพิ่มขึ้น โดย G′ มีค่าสูงกว่า G″ เมื่อเปรียบเทียบที่ความเข้มข้นและอุณหภูมิเดียวกัน อย่างไรก็ตาม ผลของอุณหภูมิต่อค่า G′ และ G″ ในแต่ละระบบไม่แตกต่างกันมากนัก เมื่ออุณหภูมิที่ทำการวัดแตกต่างกัน ดังแสดงในภาพที่ 3.35 ค่า G′ ที่ไม่เป็นศูนย์แสดงให้เห็นว่า มีอันตรกิริยาระหว่างแซนแทน-แซนแทน ในขณะที่ G″ ของสารละลายแซนแทนมีค่าใกล้เคียงกันมาก เมื่ออุณหภูมิที่ทำการวัดระหว่าง 10, 25, 40 และ 55°C เมื่อทำการวิเคราะห์หาค่า G"/G' (ที่เรียกว่า tan δ) แล้วเขียนกราฟความสัมพันธ์ระหว่าง G"/G' กับอุณหภูมิของระบบ แซนแทน/น้ำ พบว่า ค่า G"/G' มีค่าเกือบคงที่เมื่ออุณหภูมิเพิ่มขึ้น (ภาพที่ 3.36) ในระบบที่แซนแทนมีความเข้มข้น 0.5% (w/w) เมื่อระบบมีความเข้มข้นของแซนแทนมากกว่า 0.5% (w/w) แม้ว่าค่า G"/G' มีแนวโน้มขึ้นกับความเข้มข้นของแซนแทนโดยมีค่าลดต่ำลง ผลที่ได้ชี้ให้เห็นว่า แซนแทนกระจายตัวอยู่ในน้ำโดยมีคุณสมบัติทาง viscoelastic ที่เสถียรในช่วงกว้างของอุณหภูมิ

การเติมซูโครสทำให้ค่า G′ และ G″ ที่วัด ณ อุณหภูมิ 25°C มีค่าสูงขึ้น (ภาพที่ 3.37) เมื่อเติมซูโครสสูงขึ้นถึง 40% พบว่า ค่า G′ และ G″ เพิ่มขึ้นเมื่อความเข้มข้นของแซนแทนมากขึ้น ซึ่งแสดงให้เห็นว่าสารละลายมีความคงตัวสูงขึ้น โดยความเข้มข้นของแซนแทนและซูโครสมีผลต่อค่า dynamic moduli ทั้งสองของระบบที่มีแซนแทน/ซูโครส/น้ำ

ส่วนในระบบ แซนแทน/ซูโครส/น้ำ พบว่า G"/G' มีค่าเกือบคงที่ในระบบที่ไม่มีการเติมซูโครส ยกเว้นในระบบที่แซนแทนมีความเข้มข้น 3% (w/w) (ภาพที่ 3.38) ในทางตรงกันข้ามระบบที่มีซูโครสอยู่ 10 ถึง 40% (w/w) พบว่า ค่า G"/G' ลดลงอย่างรวดเร็วเมื่อความเข้มข้นของแซนแทนเพิ่มขึ้นมากกว่า 1.5% (w/w) ซึ่งชี้ให้เห็นว่า ค่า G' จะเพิ่มขึ้นมากกว่า G" เมื่อความเข้มข้นของซูโครสเพิ่มขึ้น จากผลการทดลองที่ได้ชี้ให้เห็นว่า ซูโครสมีผลต่อคุณสมบัติทางด้าน elastic มากกว่าคุณสมบัติทางด้าน viscous ของระบบที่ความเข้มข้นของแซนแทนสูงขึ้น อย่างไรก็ตาม จากภาพที่ 3.38 ที่ความเข้มข้นประมาณ 3% (w/w) ค่า G"/G' ของระบบที่มีแซนแทน 3% (w/w) ไม่ว่าจะมีปริมาณซูโครสเท่าใด จะมีค่า G"/G'ใกล้เคียงกันมาก ซึ่งแสดงให้เห็นว่า ที่ความเข้มข้นแซนแทน 3% (w/w) การเติมซูโครสมีผลต่อระบบนี้ โดยทำให้ระบบมีอัตราส่วนของระดับขององค์ประกอบที่เป็นส่วนของ viscous และ elastic ใกล้เคียงกัน ซึ่งระบบที่มีค่า G"/G' ต่ำอาจเรียกว่า เจลอ่อน (weak gel) (Ross-Murphy, 1994)



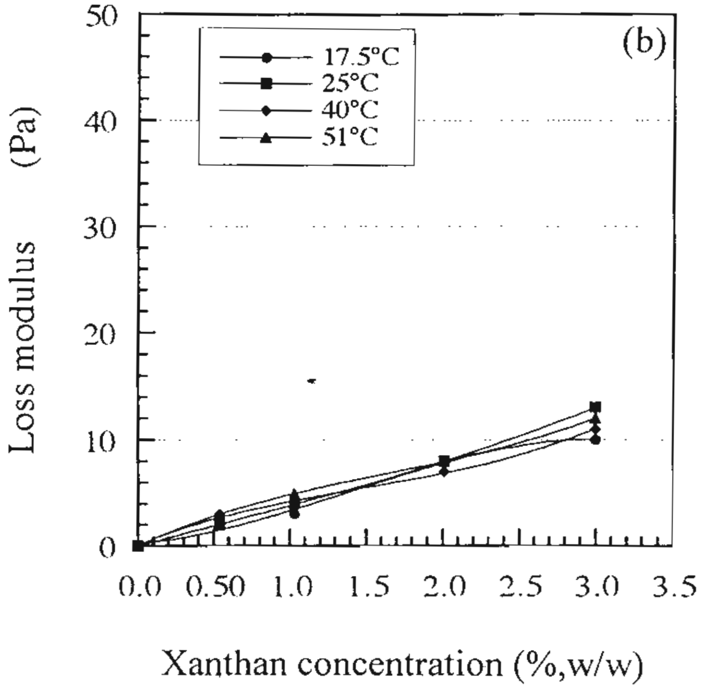


Fig. 3.35 Storage (a) and loss (b) moduli of xanthan solutions for different temperatures.

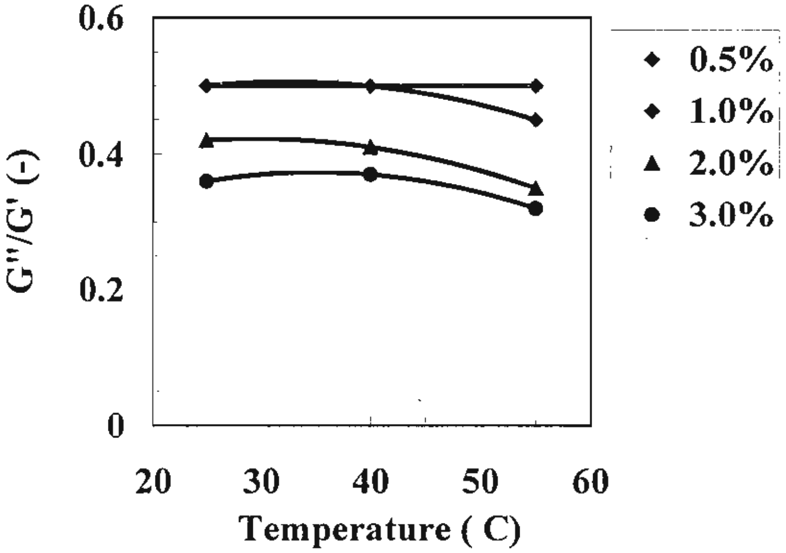


Fig.3.36 Effect of xanthan concentration on G"/G' values of xanthan/water systems as a function of temperatures.

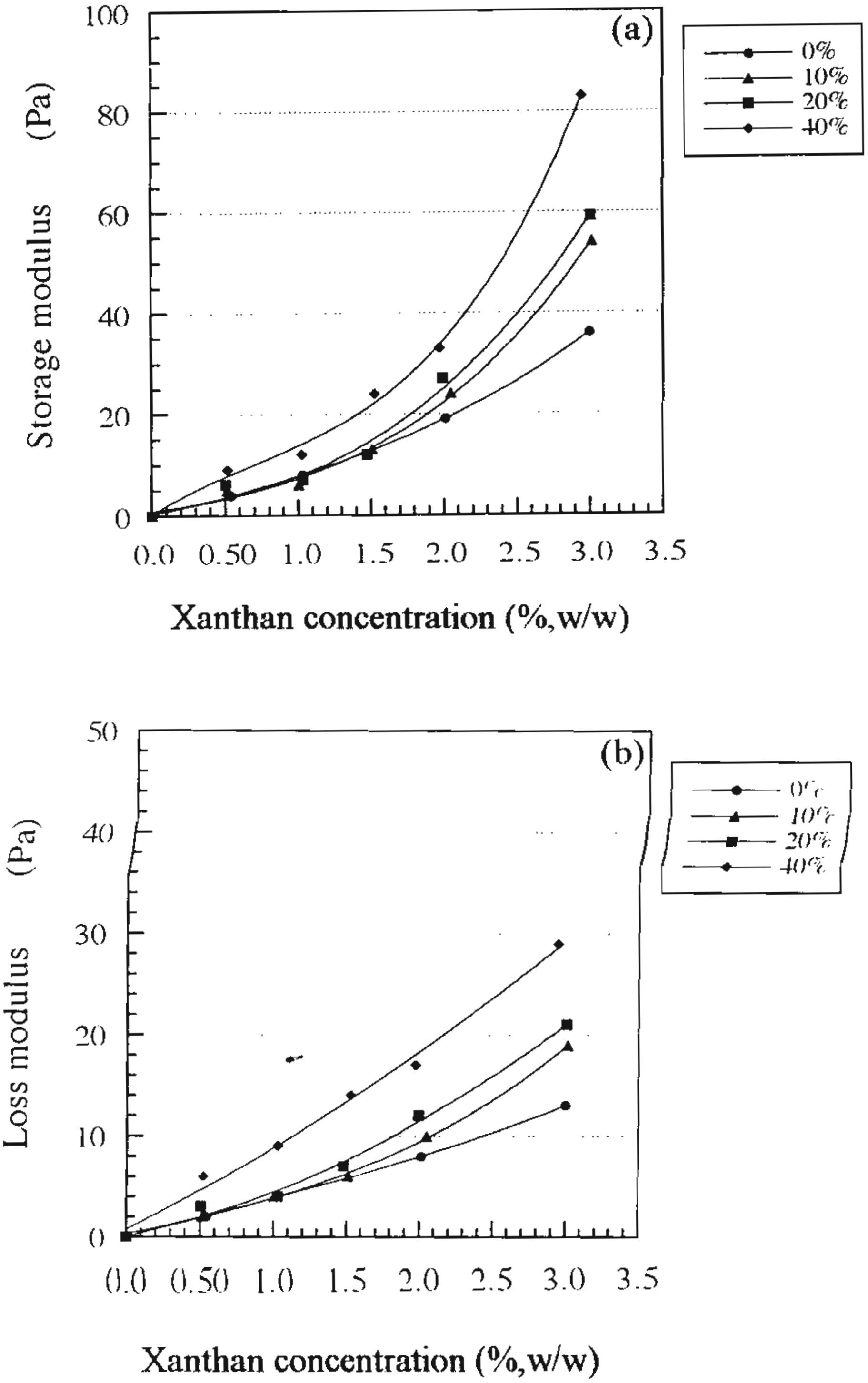


Fig. 3.37 Storage (a) and loss (b) moduli of xanthan solutions containing different sucrose content at 25°C.

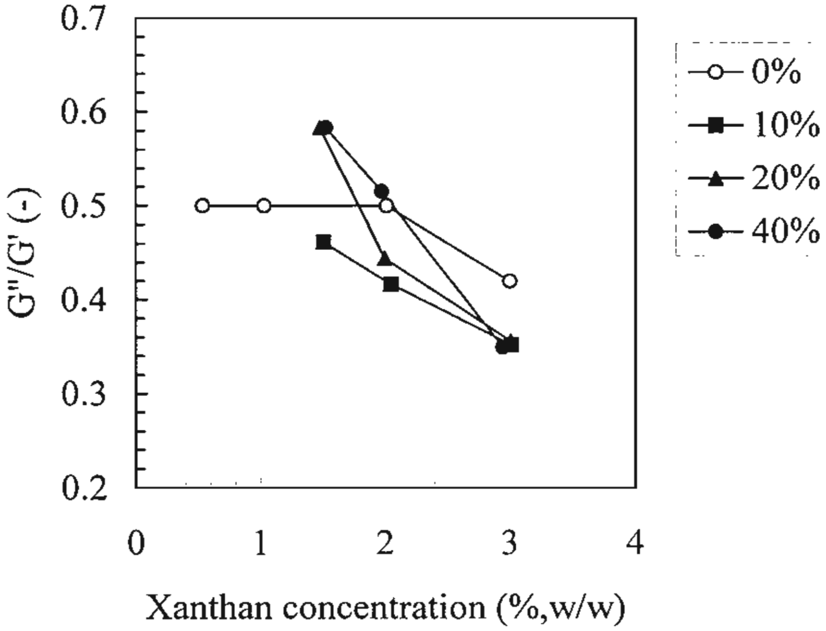


Fig. 3.38 Effect of sucrose on G″/G′ values of xanthan solutions.

3.4.4. สมการที่อธิบายผลของอุณหภูมิและซูโครสต่อคุณสมบัติทางด้านรีโอโลจี

(1) ผลของอุณหภูมิ

เนื่องจากแซนแทนมักใช้ในผลิตภัณฑ์อาหาร ซึ่งจำเป็นต้องผ่านกระบวนการต่างๆ ในระหว่างการแปรรูปอาหาร การเก็บ การขนส่ง การตลาดหรือการบริโภค อาหารเหลวจึงต้องผ่านอุณหภูมิที่แตกต่างกัน ด้วยเหตุนี้ จึงจำเป็นต้องมีการคุณสมบัติทางรีโอโลจีซึ่งเป็นฟังก์ชันกับอุณหภูมิเช่นกัน

ผลของอุณหภูมิต่อค่าความหนืดในสารละลายสามารถอธิบายได้ด้วยสมการ Arrhenius ดังสมการต่อไปนี้

$$\ln \eta_a = \ln \eta_o - E_a/RT$$

เมื่อ η_a = apparent viscosity ที่อุณหภูมิ T (K)

η_o = ค่าคงที่

E_a = พลังงานกระตุ้น สำหรับความหนืด

R = ค่าคงที่ของแก๊ส (= 8.314 J/mole.K)

T = อุณหภูมิสัมบูรณ์ (K)

โดยความสัมพันธ์ของ Arrhenius ได้จากการสร้างกราฟของ ln (viscosity) กับส่วนกลับกับอุณหภูมิสัมบูรณ์ ดังแสดงในภาพที่ 3.39 ขนาดของพลังงานกระตุ้นของสารละลายพอลิเมอร์ที่ทำการศึกษาได้จากความชันของกราฟแต่ละความเข้มข้นของแซนแทนจะอยู่ในช่วง 2.7-10.6 kJ/mole ซึ่งจะมีค่าต่ำกว่าของน้ำที่ 16.3 KJ/mole (ตารางที่ 3.3) จากผลการทดลองที่ได้ชี้ให้เห็นว่า การเปลี่ยนแปลงของความหนืดของสารละลายพอลิเมอร์ขึ้นกับอุณหภูมิน้อยกว่าของน้ำ หรืออาจกล่าวได้ว่า การเปลี่ยนแปลงของความหนืดของสารละลายพอลิเมอร์แทบจะไม่ขึ้นกับอุณหภูมิ

Table 3.3 Activation energies of the aqueous xanthan dispersions and water

Xanthan concentration (%, w/w)	Activation energy (kJ/mole)
0 (water)	16.3
0.01	4.1
0.02	5.1
0.03	2.7
0.05	6.1
0.10	10.6
0.19	9.9
0.29	6.1

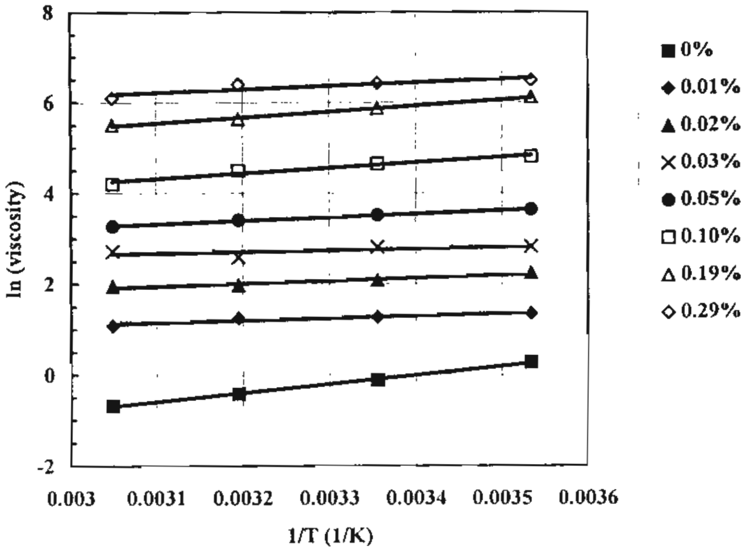


Fig. 3.39 **Arrhenius plot between ln of viscosity and 1/T for different xanthan concentrations.**

เพื่อให้เกิดความเข้าใจในเรื่องคุณสมบัติทางด้านโมเลกุลในสารละลายเจือจางของพอลิเมอร์ (Rao, 1995) สำหรับสารละลายแซนแทน จะใช้ค่า specific viscosity (η_{sp}) ซึ่งได้จากนิยามดังสมการ (3.3)

$$\eta_{sp} = (\eta - \eta_s)/\eta_s$$

เมื่อ η = ความหนืดของสารละลาย (solution)

η_s = ความหนืดของตัวทำละลาย (solvent)

η_{sp} = specific viscosity

โดยค่า specific viscosity เป็นการ contribution ของพอลิเมอร์ต่อความหนืดที่เกิดขึ้นในระบบ ซึ่งแสดงให้เห็นถึงปริมาตร (effective volume) ที่ครอบครองโดยพอลิเมอร์

อุณหภูมิมีผลต่อค่า specific viscosity น้อยมาก และผลที่ได้สามารถแทนด้วยกราฟหลักเพียงกราฟเดียว (ภาพที่ 3.40) ซึ่งชี้ให้เห็นว่าการเปลี่ยนแปลงอุณหภูมิจาก 10°C ถึง 55°C ไม่ได้มีผลต่อการเปลี่ยนแปลงรูปร่างและขนาดของแต่ละ random coils ของแซนแทนในสารละลาย

(2) ผลของซูโครส

ในระบบของแซนแทน/ซูโครส/น้ำ การมีส่วนร่วม (contribution) ของแซนแทนต่อความหนืดของสารละลายสามารถประเมินได้จากสมการ (3.3) โดยการใช้ความหนืดของสารละลายซูโครสเป็นค่า η_s (ความหนืดของ solvent) ดังแสดงในภาพที่ 3.41 ซึ่งแสดงค่าของ specific viscosity ที่ได้จากการคำนวณเป็นฟังก์ชันกับความเข้มข้นของแซนแทนที่อุณหภูมิ 25°C ผลของซูโครสต่อ specific viscosity มีค่าน้อยมากในความเข้มข้นของแซนแทน < 0.3% และผลที่ได้จากทุกระบบสามารถสามารถแทนด้วยกราฟเส้นเดียว ดังนั้น ผลที่ได้จากภาพที่ 3.41 ชี้ให้เห็นว่า การเติมซูโครสไม่ได้เปลี่ยนแปลงรูปร่างและขนาดของแต่ละ random coil ของแซนแทนในสารละลาย เมื่อพิจารณาผลของการเติมซูโครสที่อุณหภูมิสูงขึ้น (40°C) กราฟของ specific viscosity ที่เป็นฟังก์ชันกับความเข้มข้น (ภาพที่ 3.42) ที่ได้จะคล้ายกับภาพที่ 3.41 ที่ทุกข้อมูลแสดงด้วยกราฟเส้นเดียว เมื่อเปรียบเทียบกับผลของวอเตอร์แอคทิวิตี้หรือค่าสัดส่วนของน้ำที่แข็งตัวได้ คุณสมบัติทางรีโอโลจีของสารละลายแซนแทนที่มีการเติมซูโครสจะขึ้นกับปริมาณของแซนแทนแม้ที่ระบบซึ่งมีความเข้มข้นของแซนแทนต่ำกว่า 0.3%

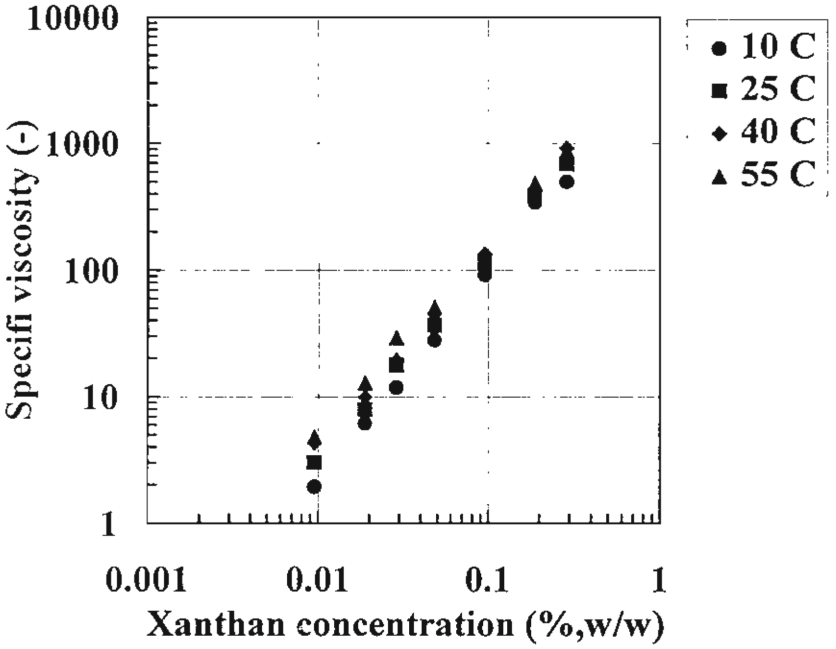


Fig. 3.40 Specific viscosity of xanthan solution at different temperatures.

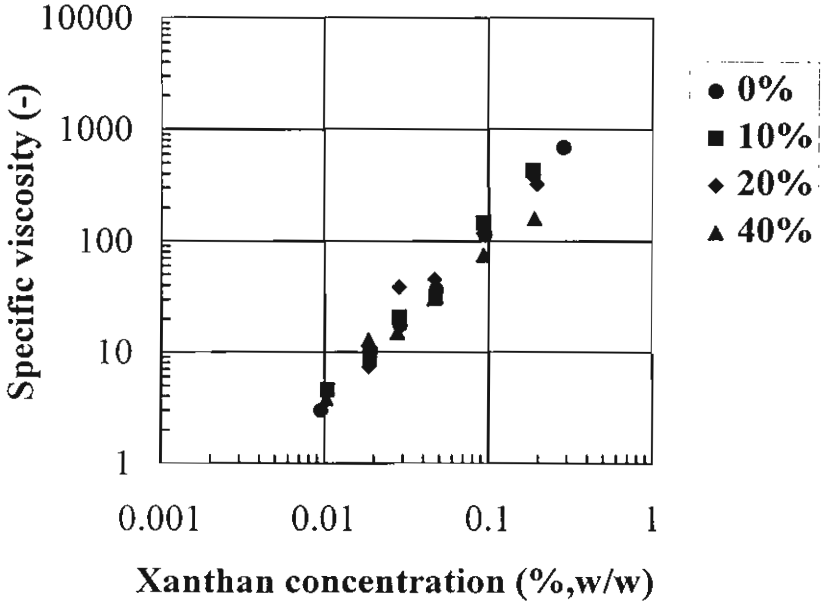


Fig. 3.41 Effect of sucrose on specific viscosity of xanthan solutions at 25°C

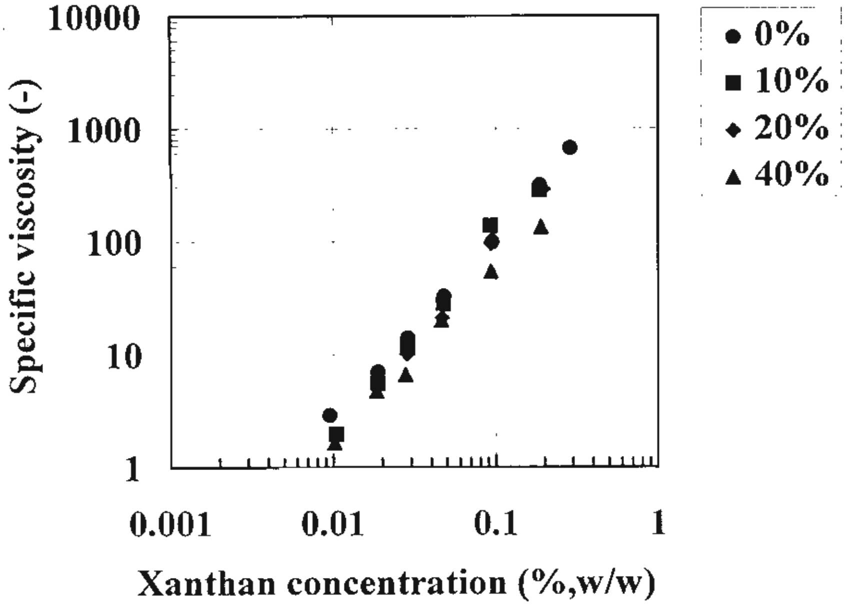


Fig. 3.42 Effect of sucrose on specific viscosity of xanthan solutions at 40°C.

IV เอกสารอ้างอิง

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Effect of sucrose on physical properties of alginate dispersed aqueous system

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Abstract

The effect of sucrose on water activity, the fraction of freezable water, and viscoelastic properties of alginate dispersed aqueous system was investigated. Water activity and the fraction of freezable water were almost independent on examined alginate concentration but decreased with increasing sucrose concentration, indicating that colligative properties of those systems were mainly determined by the sucrose content. The specific viscosity values were represented by a single master curve for all the systems containing lower concentration of alginate than 0.5% (w/w). For the systems containing higher concentration of alginate than 0.5% (w/w), the ratio of the loss modulus, G", to the storage modulus, G'; G"/G' (= tan δ) was almost independent on alginate concentration in the systems without sucrose but the G"/G' values decreased with increasing alginate concentration in the systems containing sucrose. These results suggest that interactions between sucrose and alginate were negligible at a low alginate concentration but the elastic nature of the systems was enhanced due to sucrose-alginate interactions at a high alginate concentration.

Key words: alginate, water activity, fraction of freezable water, viscosity, storage modulus, loss modulus.

Introduction

Food macromolecules play a critical role in the formulation of a wide range of food products such as beverages, bread, cheese, desserts, dressings, ice-cream and so on (Dickinson & Lorient, 1995) by improving product mouthfeel properties, handling and stability characteristics with consistent properties, shelf stability and good consumer appeal and acceptance (Imeson, 1992). Since these qualities of foods are strongly related to rheological properties, knowledge of these properties of food macromolecule systems is essential for predictability of product properties, process design and evaluation, quality control, and consumer acceptability of food products (Rao, 1977; Roos & Karel, 1991).

An important functional property of food polysaccharides is controlling rheological properties of an aqueous phase in foods through entanglement and association of polymer chains or gelation behaviour (Moe *et al.*, 1995). Many of food polysaccharides have linear molecular chains with a large molecular weight consisting of unit saccharides while most food proteins have spherical shape with a lower molecular weight. Therefore, even at a very low concentration, i.e., a few percentage of total weight, polysaccharides can show very high viscosity or sometimes gelation. Alginates, linear random copolymers of D-mannuronic acids (M-blocks) and L-glucuronic acids (G-blocks) commercially extracted from brown seaweeds, are one of the most commonly used food polysaccharides as additives to improve, modify, and stabilize the texture without changing nutritional values (Pomeranz, 1991).

In product development, food macromolecules are usually used in the presence of other food ingredients and addition of those food ingredients itself also changes physical properties of foods. Therefore, the quality of final food products depends on the contributions from individual ingredients and interactions among them. Sugar is one of the most important food ingredients and used for making dessert foods or confectioneries such as dessert jellies. Adding sugar into foods is known to change the solvent (water) properties in foods. As a result of reducing water activity or freezable water fraction with increasing sugar concentration in an aqueous phase, sugar has been shown to change important physical properties for controlling food processing such as thermal conductivity (Pongsawatmanit & Miyawaki, 1993).

Despite the importance of the interactions among ingredients and water in food systems, only a limited understanding of their effects on physical properties of foods is available. Alginate/sucrose/water systems would represent a typical example of foods since sucrose is the most common dietary disaccharide and the chief sweetener in sweet food preparation (Medved, 1986). In this paper, effects of sucrose on physical properties of alginate dispersed aqueous systems were investigated. Viscosity measurements at a low alginate concentration and dynamic viscoelasticity measurements at a high alginate concentration were performed. Associated changes in the state of water, namely the water activity and the fraction of freezable water, were also investigated.

Materials and Methods

Materials

Sodium alginate extracted from *Lessonia nigrescens* (Kanto Chemical, Tokyo, Japan, Lot. no. 802S1707; approximate molecular weight and M/G ratio are 176,000 and 1, respectively) was used without further purification. The moisture content was determined by drying 1.00 g samples of sodium alginate at 107°C for 4-6 h. The content of sodium, potassium, and calcium ions was determined by an atomic absorption spectroscopy and the content of Cl^- and SO_4^{2-} was determined by an ion chromatography method. The results of sample characterization are summarized in Table 1. Water used in preparation and experiment was purified (Millipore, resistivity 18 MΩcm) after distillation. All other chemicals were of reagent grade quality.

Table

Preparation of alginate solutions

A precisely weighed amount (±0.1 mg in error) of the alginate powder and sucrose were suspended in water and then dissolved at room temperature by stirring with a magnetic stirrer for at least 6 h. The sample was kept overnight at 25°C before measurement. Zero to 2.5% (w/w) of alginate solutions containing 0, 10, 20 and 40% (w/w) of sucrose were prepared.

Water activity measurement

Water activity of sample solutions containing 0-2.5% (w/w) alginate and 0-40% (w/w) sucrose was measured using Thermoconstanter (Novasina, Zurich, Switzerland) controlled at 25°C. The apparatus was calibrated before sample measurements by using the saturated salt solutions; SAL-90, SAL-75, SAL-53, SAL-33 (Novasina, Zurich). Water activity values were recorded after the equilibrium was reached for 1 h. Measurements were done in two replications.

Freezable water measurement

Differential Scanning Calorimetry (DSC) was performed to evaluate the fraction of freezable water in the sample solutions. The enthalpy change during heating process of frozen samples was determined using a DSC apparatus (Low temperature Unit, Rigaku Corporation, Tokyo, Japan) and then converted into the amount of melted ice. The instrument constant of the apparatus was measured using standard materials (distilled water, mercury, benzene, and chloroform) with a known latent heat of phase transition at a certain temperature. Fifteen to 25 mg of each sample solution was transferred into an aluminum pan and then sealed. The samples were cooled down to -70°C or below to ensure complete freezing and then heated up at a constant heating rate, 1.0°C/min. The endothermic enthalpy of the samples during the heating process was measured using aluminum oxide (Al_2O_3) as a reference sample. The samples were weighed before and after the measurement to confirm that no weight was lost. Measurements were done at least in duplicate. The values of enthalpy change due to the melting of ice in the samples were determined as the area under the endothermic curves measured using a planimeter and then converted into the fraction of freezable water according to the conventional procedure (Pongsawatmanit and Miyawaki, 1993).

Rheological measurements

The apparent viscosity of dilute alginate solutions (0.1-0.5%, w/w) was measured with a cone and plate type viscometer (Visconic, ED type, 1°34' in the cone angle and 2.4 cm in diameter, Tokimec, Tokyo, Japan). One mL of the sample solution was poured into the sample cup and the viscosity values were measured at 25 and 40°C in the shear rate range of 1.92-383 s^{-1}. The viscosity values at the lowest shear rate where the value was in the reliable range of the apparatus were taken as apparent viscosity values.

At a high concentration of alginate solutions (0.5-2.5%, w/w) dynamic viscoelasticity measurements were performed using a Rheolograph sol (TCU-6, Toyo seiki, Tokyo, Japan). The alginate solutions were poured into the sample holder (1.5 mL) using glass pipettes and conditioned at least 30 min to equilibrate the samples at a desired temperature (25°C). The surface of the samples was covered with a thin layer of silicone oil to prevent water evaporation during measurements. The sample solutions were subjected to 2 Hz sinusoidal shear oscillations of a stainless steel blade (sus 304, 10 mm W x 25 mm L x 0.6 mm T) with an amplitude of 125 μm only when the storage modulus (G') and the loss modulus (G") were measured.

Results and Discussion

Relatively low concentration of polysaccharides in solution finds significant use in the food industry to control the rheological properties of many modern convenience food products (Krumel & Sarkar, 1975; Clegg, 1995). In this study, the concentration range of alginate and that of sucrose were varied from 0 to 2.5 and 0 to 40% (w/w), respectively, by considering practical usage in the food industry. As shown later, a system containing 2.5% (w/w) alginate and 40% (w/w) sucrose was high enough in concentrations to show gel-like behavior.

Effect of sucrose on water activity and fraction of freezable water

Since water is a major component in almost all foods and frequently used as a diluent of food solids, physical properties of foods, behaviour in processing, microbial growth during food storage, and so forth, are strongly related to the physicochemical properties of water in the system (Roos, 1995). Properties of water in foods exhibit differences that depend on the effect of dissolved compounds on the phase behavior of water (Roos, 1995) such as the water activity and the fraction of freezable water.

Water activity is a measure of the availability of water in an equilibrium system, which correlates with the stability of foods in terms of chemical reactions and microbial growth (Herrington & Vernier, 1995). Figure 1 shows water activities of alginate aqueous solutions containing sucrose as an additive. The water activity values slightly decreased as the sucrose concentration increased up to 40% (w/w). In contrast, no significant change in the water activity was observed when alginate concentrations ranged from 0% (w/w) to 2% (w/w) at a fixed sucrose concentration. The water activity value of the most thick solutions which contained 40% (w/w) sucrose and 2% (w/w) alginate was about 0.95; that of pure water being unity. The results in Fig. 1 suggest that water activity values of alginate aqueous solution containing sucrose are largely determined by the sucrose content. In the practical concentration range used in the food industry, the molar amount of components having low molecular weight is considered dominant to that of polymers. Therefore, a colligative property of water such as water activity would be dependent mostly on the content of low molecular weight substances.

F1

Figure 2 shows the fraction of freezable water as a function of added sucrose concentration. As a comparison, the fraction of freezable water in sucrose/water systems without alginate was taken from a previous study (Pongsawatmanit & Miyawaki, 1993). Adding sucrose significantly decreased the fraction of freezable water with increasing sucrose concentrations. At the same sucrose concentration, the fraction of freezable water did not depend on the alginate concentrations. Although the fraction of freezable water was more sensitive to the sucrose content than the water activity, the both results shown in Fig. 1 and Fig. 2 clearly demonstrate that

Fi

physical properties of water in alginate/sucrose/water systems are mainly determined by the high concentration of sucrose.

Effect of sucrose on viscosity

Alginates function as a stabilizer in foods through its abilities to hydrate and increase viscosity of the system (Onsøyen, 1992; Marshall & Arbuckle, 1996). The effect of sucrose addition on viscosity of alginate solutions was investigated at a low concentration of alginate. As shown in Fig. 3, apparent viscosity at 25°C increased with increasing alginate concentration. At a fixed alginate concentration, the systems containing a higher amount of sucrose had a higher viscosity value. Fig

For polymer solutions, the specific viscosity (η_{sp}) can be defined by the following equation:

$$\eta_{sp} = (\eta - \eta_s)/\eta_s \qquad (1)$$

where η and η_s are the viscosities of the solution and the solvent, respectively. Values of the specific viscosity are considered to be the contribution of polymer to viscosity, reflecting the effective volume occupied by polymers in the system. In alginate/sucrose/water systems, the contribution of alginate to solution viscosity can be evaluated by using viscosity values of sucrose solution as η_s. Figure 4 shows the calculated values of the specific viscosity as a function of alginate concentrations. The effect of sucrose on specific viscosity values were small and all the results can be represented by a single curve. At a sufficiently low concentration, alginate is considered to exist as disordered random coils in solutions and the individual coils are well separated from one another being free to move independently (Morris, 1995). Therefore, the results in Fig. 4 indicate that adding sucrose did not change the shape Fig and the size of the individual alginate random coils in solutions. Considering the effect of sucrose addition at the higher temperature (40°C), specific viscosity values were plotted as a function of concentration (Fig. 5). Similar to the results in Fig. 4, all the data can be represented by a single master curve except for low concentration of alginate at 40% (w/w) sucrose. In contrast to the results of the water activity or the freezable water fraction, rheological properties of alginate solutions with added sucrose were strongly dependent on the amount of alginate even at a low polymer concentration <0.5% (w/w).

Fig

Effect of sucrose on the viscoelastic properties

With increasing polymer concentration, the elastic nature of the system due to overlapping or entanglement of polymers becomes pronounced. As a result, the system shows a viscoelastic behavior at a high polymer concentration. In this regime, two separate components of viscoelasticity are obtainable by measuring the stress response of materials to a sinusoidal shear oscillation: the storage modulus, G', (in phase) and the loss modulus, G", (out of phase) which are a measure of the elastic and the viscous component, respectively. On a molecular basis, the magnitude of storage modulus, G', is dependent upon degree of rearrangements taking place within the period of oscillation and the loss modulus, G", is a measure of the energy dissipating as heat per cycle of sinusoidal deformation (Rao, 1992; Ross-Murphy, 1994).

For the systems with a higher alginate concentration than 0.5% (w/w), G' and G" increased with increasing alginate content (Fig. 6). Non zero values of the storage modulus, G', represent the existence of elastic nature while relatively larger values of G" than G' at each condition indicate a typical liquid-like behavior of these systems. Addition of sucrose increased both G' and G" values. The systems containing 40%

Fig

(w/w) sucrose showed significant large values of both G' and G" indicating that the solutions are highly stabilized by sucrose addition.

Fig

In Fig. 7, G"/G' values (so called tan δ) are plotted against alginate concentrations. The values of G"/G' were almost constant for the systems without added sucrose. In contrast, the systems containing 10 to 40% (w/w) sucrose showed rapid decrease in G"/G' with increasing alginate concentrations indicating G' values increased more sharply than G" with increasing sucrose concentration. The G"/G' value for the system containing 2.5% (w/w) alginate and 40% (w/w) sucrose was unity indicating the same degree of viscous and elastic components exist. Such systems with low ~~high~~ G"/G' values would be inferred as a weak gel (Ross-Murphy, 1994). Consequently, sucrose was found to affect the elastic character rather than the viscous behaviour of systems at a high alginate concentration. Based on the results of specific viscosity at a lower alginate concentration shown in Fig. 4, it was suggested that the effect of interactions between alginate and sucrose on rheological properties was negligible at a lower alginate concentration than 0.5% (w/w). When the concentration of alginate were increased (higher than 0.5%, w/w), where overlapping or entanglement of polymer chains are expected, effect of sucrose on elastic nature of the system was pronounced by increasing both concentrations of sucrose and alginate.

To summarize, adding sucrose to alginate dispersed systems decreased the water activity and the freezable water fraction, the values of which are mainly determined by sucrose concentration, while rheological properties were positively affected. With increasing alginate concentration, elastic nature of the alginate/sucrose/water system became pronounced due to sucrose-alginate interaction especially in the alginate concentration range higher than 0.5% (w/w).

Acknowledgments

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Tables

Table 1 Composition of cation and anion in the alginate powder.

Biopolymer	Moisture content (%)	Amount of cation (%, w/w)			Amount of anion (%, w/w)	
		Na^+	K^+	Ca^{2+}	Cl^-	SO_4^{2-}
Sodium alginate	9.48	8.10	0.07	<0.01	0.08	0.27

Figure Legends

Fig. 1 Effect of sucrose on water activity in alginate solutions containing 0% (♦), 0.5%(w/w) (■), 1.0%(w/w) (▲), and 2.0%(w/w) (●) alginates.

Fig. 2 Effect of sucrose on fraction of freezable water in alginate solutions containing 0% (♦), 0.5%(w/w) (■), 1.0%(w/w) (▲), and 2.0%(w/w) (●) alginates.

Fig. 3 Apparent viscosity of sucrose/alginate/water systems containing 0% (●), 10%(w/w) (■), 20%(w/w) (♦), and 40%(w/w) (▲) sucrose.

Fig. 4 Specific viscosity at 25°C of sucrose/alginate/water systems containing 0% (●), 10%(w/w) (▲), 20%(w/w) (■), and 40%(w/w) (♦) sucrose.

Fig. 5 Specific viscosity at 40°C of sucrose/alginate/water systems containing 0% (●), 10%(w/w) (▲), 20%(w/w) (■), and 40%(w/w) (♦) sucrose.

Fig. 6 Storage (a) and loss moduli (b) of sucrose/alginate/water systems containing 0% (●), 10%(w/w) (▲), 20%(w/w) (■), and 40%(w/w) (♦) sucrose.

Fig. 7 Effect of sucrose on G"/G' value of sucrose/alginate/water systems containing 0% (○),10%(w/w) (▲), 20%(w/w) (■), and 40%(w/w) (♦) sucrose.

Fig. 1

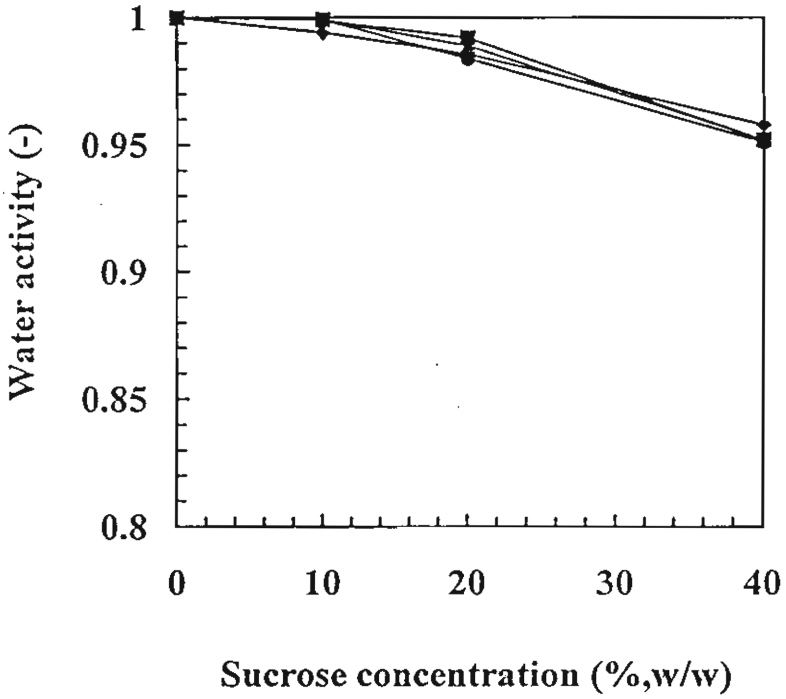
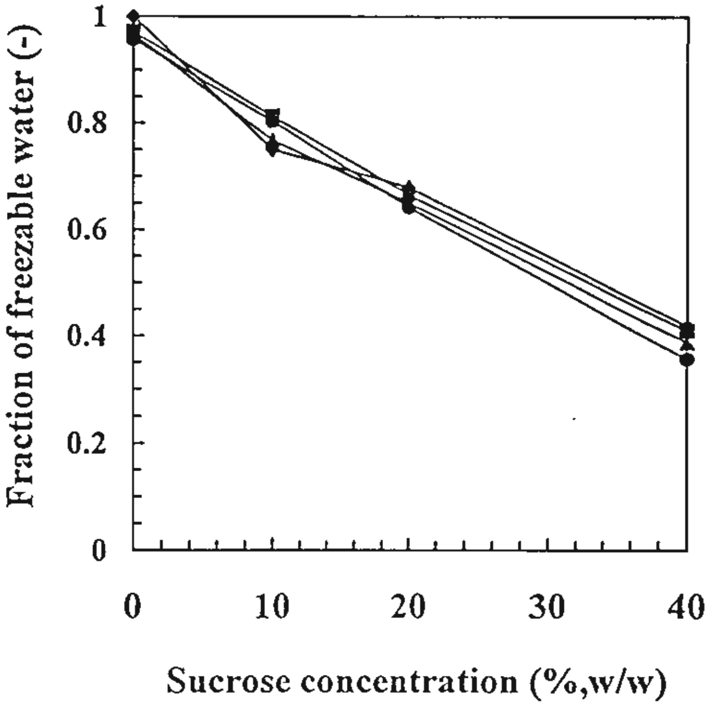

Water activity (-)
1
0.95
0.9
0.85
0.8
0
10
20
30
40
Sucrose concentration (%,w/w)

Fig. 2



Fraction of freezable water (-)
1
0.8
0.6
0.4
0.2
0
0
10
20
30
40
Sucrose concentration (%,w/w)

Fig. 3

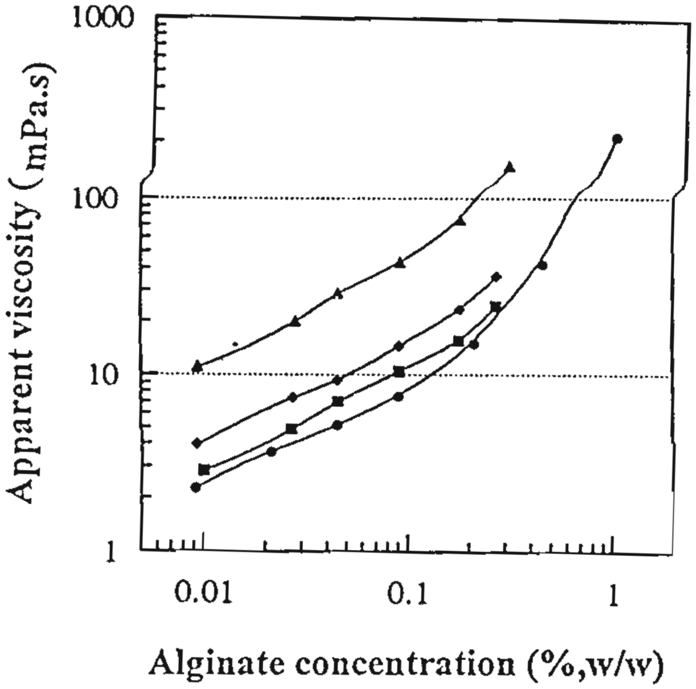

1000
100
10
1
Apparent viscosity (mPa.s)
0.01
0.1
1
Alginate concentration (%,w/w)

Fig. 4

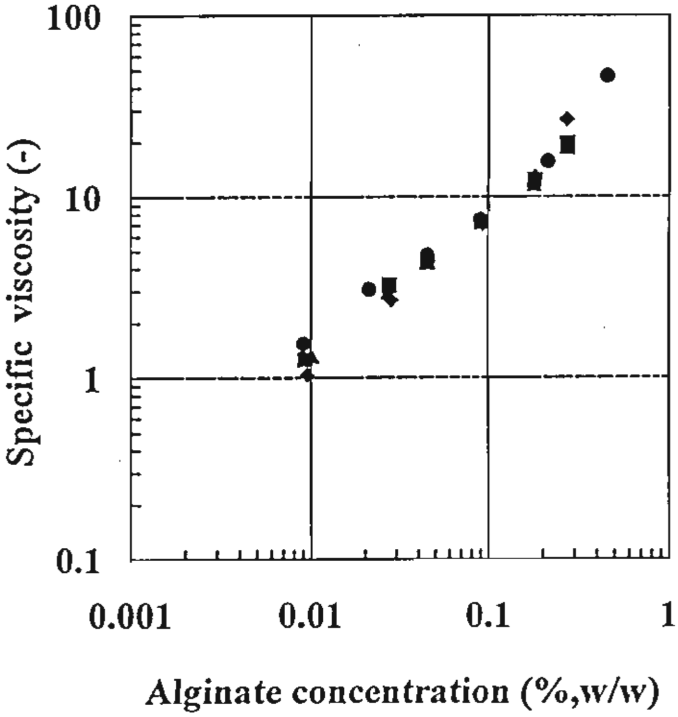

100
10
1
0.1
0.001
0.01
0.1
1
Specific viscosity (-)
Alginate concentration (%,w/w)

Fig. 5.

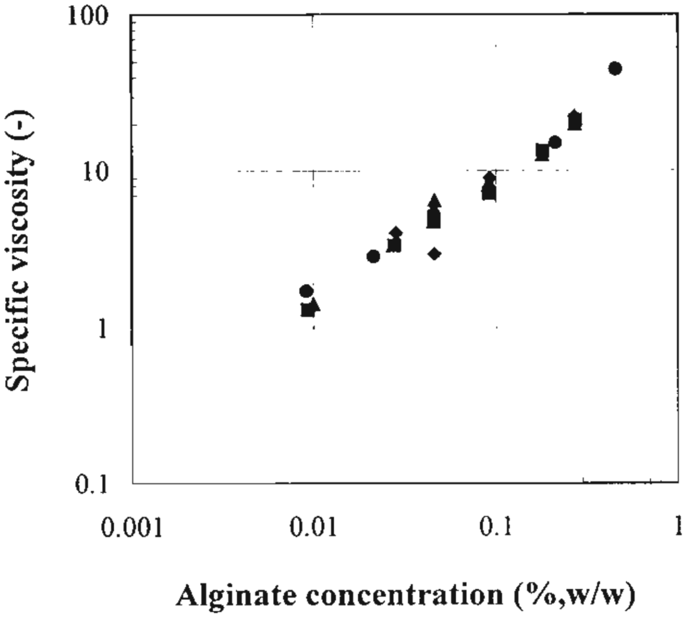
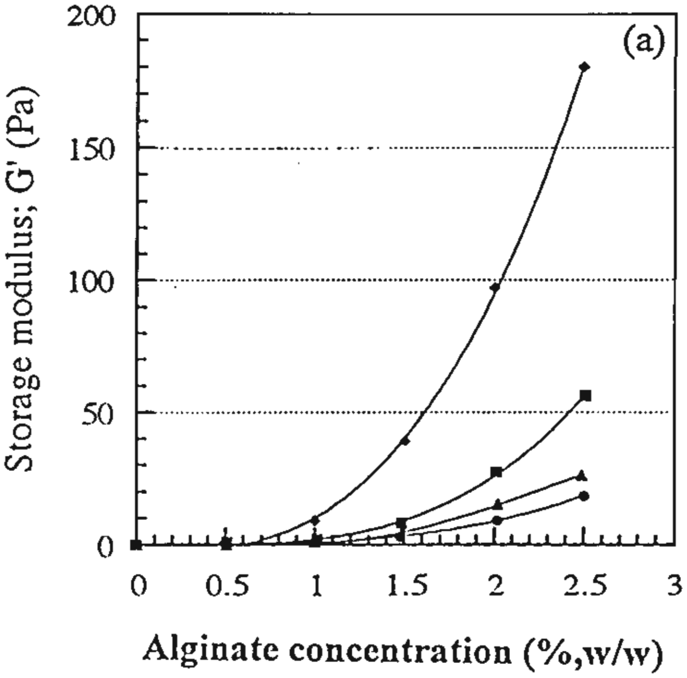

100
10
1
0.1
0.001
0.01
0.1
1
Specific viscosity (-)
Alginate concentration (%,w/w)

Fig. 6


(a)
Storage modulus; G' (Pa)
0
50
100
150
200
0
0.5
1
1.5
2
2.5
3
Alginate concentration (%,w/w)

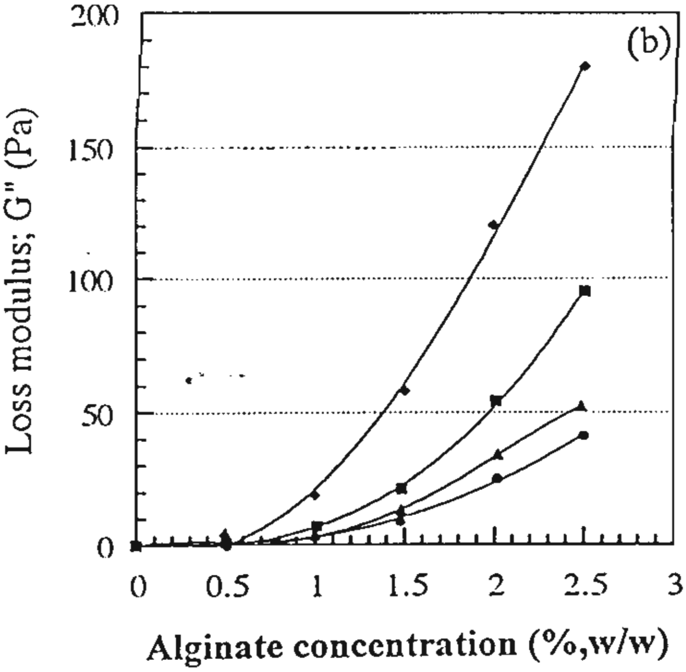
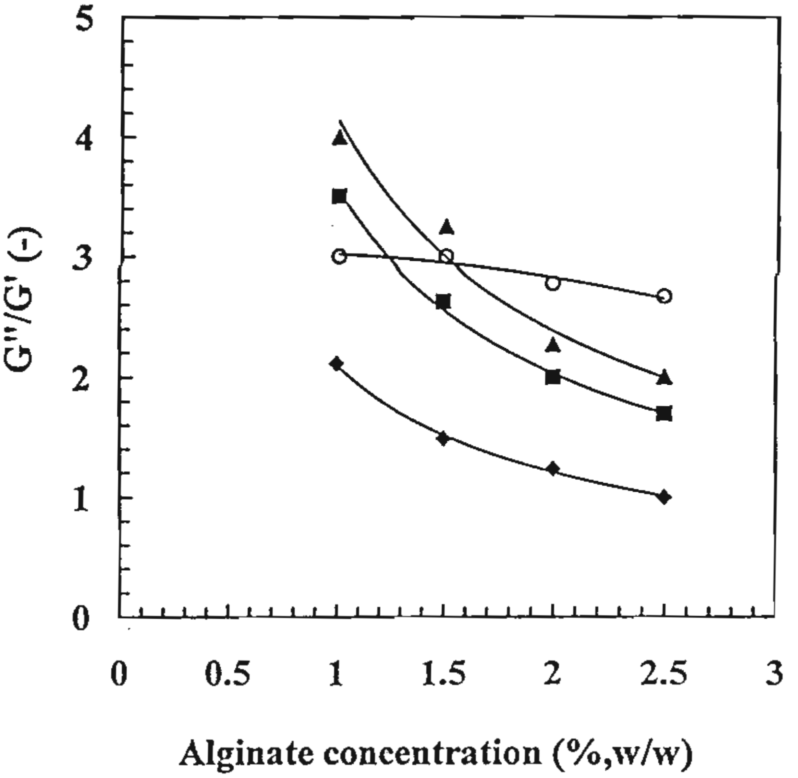

(b)
Loss modulus; G" (Pa)
0
50
100
150
200
0
0.5
1
1.5
2
2.5
3
Alginate concentration (%,w/w)

Fig. 7



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EFFECT OF TEMPERATURE ON VISCOELASTIC PROPERTIES OF AQUEOUS ALGINATE SOLUTIONS

By

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ABSTRACT

The effect of temperature on viscosity and viscoelastic properties of alginate dispersed aqueous system was investigated. Apparent viscosity decreased with increasing temperature. The specific viscosity values were represented by a single master curve for the different temperature of alginate solutions containing lower concentration of alginate than 0.5% (w/w). The activation energy of the Arrhenius relationship between ln of viscosity for polymer solutions and reciprocal of absolute temperature was in the same order as that of water. For the systems containing higher concentration of alginate than 0.5% (w/w), the storage and loss moduli increased with alginate concentration but decreased with increasing temperature. When considering the ratio of the loss modulus, G", to the storage modulus, G'; G"/G' (= tan δ) values increased with increasing temperature but for the higher polymer concentration (2.0%, w/w), the G"/G' values increased less than that of system with 1.5% (w/w) alginate.

These results suggest that effect of temperature on the viscoelastic properties of aqueous alginate solutions was lower with increasing alginate concentration.

Key words: alginate, viscosity, storage modulus, loss modulus, activation energy.

INTRODUCTION

Alginates, linear copolymers of D-mannuronic acids (M-blocks) and L-glucuronic acids (G-blocks) extracted from brown seaweeds, are one of the most commonly used food polysaccharides (Pomeranz 1991). An important functional property of food polysaccharides is controlling rheological properties of an aqueous phase in foods through entanglement and association of polymer chains or gelation behaviour (Moe *et al.* 1995). Rheological properties are important in the design of flow processes, in quality control, in storage and processing stability measurements, and in predicting texture (Davis 1973). In addition, understanding of rheological property improves product mouthfeel properties, handling and stability characteristics with consistent properties, shelf stability, and good consumer appeal and acceptance (Imeson 1992) in the formulation of a wide range of food products such as beverages, bread, cheese, desserts, dressings, ice-cream and so on (Dickinson and Lorient 1995).

The physical state and molecular mobility of polymers are affected by temperature and also by composition of food solids. Viscosity can be related to the molecular mobility (Roos 1995). Usually, viscosities of dilute solutions of materials, particularly polymers, are investigated to improve understanding of the molecular properties (Rao 1995). Viscoelastic properties of biopolymers such as carbohydrates and proteins can also be used to characterize their three-dimensional configuration in solution. This configuration affects their functionality in many food products. Through an understanding of how the molecular structure of polymers affects their rheological properties, it is possible to better predict and improve the flow behaviour of these polymers through various modifications (Kokini, 1992).

The characterization of the relationship between the rheological properties of alginate/water system and temperature is expected as an approach for a more understanding of the thickening properties and stabilizing capacity during processing and storage which are important for the product and process development in food industry. In this paper, effects of temperature on rheological properties of aqueous alginate solutions were investigated. Viscosity measurements at a low alginate concentration and dynamic viscoelasticity measurements at a high alginate concentration were performed.

MATERIALS AND METHODS

Materials

Sodium alginate (Kanto Chemical, Tokyo, Japan, Lot. no. 802S1707; approximate molecular weight is 176,000 and M/G ratio is 1) extracted from *Lessonia nigrescens* was used without further purification. The moisture content of the sample is about 9.48 % determined by drying 1.00 g samples of sodium alginate at 107°C for 4-6 h. The content of sodium, potassium, and calcium ions in alginate powder was determined by an atomic absorption spectroscopy and the content of Cl^- and SO_4^{2-} was determined by an ion chromatography method. The results of sample characterization are shown in Table 1. Water used in preparation and experiment was purified (Millipore, resistivity 18 MΩcm) after distillation. All other chemicals were of reagent grade quality.

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Preparation of alginate solutions

A precisely weighed amount (±0.1 mg) of the alginate powder was suspended in water and then dissolved at room temperature by stirring with a magnetic stirrer for at least 6 h. The sample was kept overnight at 25°C before measurement. Zero to 2.0% (w/w) of alginate solutions were prepared.

Rheological measurements

The apparent viscosity of dilute alginate solutions (0.1-0.9%, w/w) was measured with a cone and plate type viscometer (Visconic, ED type, 1°34' in the cone angle and 2.4 cm in diameter, Tokimec, Tokyo, Japan). One mL of the sample solution was poured into the sample cup and the viscosity values were measured at 10, 25, 40 and 55°C in the shear rate range of 1.92-383 s^{-1}. The viscosity values at the lowest shear rate where the value was in the reliable range of the apparatus were taken as apparent viscosity values.

At a high concentration of alginate solutions (0.5-2.0%, w/w) dynamic viscoelasticity measurements were performed using a Rheolograph sol (TCU-6, Toyo seiki, Tokyo, Japan). The alginate solutions were poured into the sample holder (1.5 mL) using glass pipettes and conditioned at least 30 min to equilibrate the samples at a desired temperature. The surface of the samples was covered with a thin layer of silicone oil to prevent water evaporation during measurements. The sample solutions were subjected to 2 Hz sinusoidal shear oscillations of a stainless steel blade (sus 304, 10 mm W x 25 mm L x 0.6 mm T) with an amplitude of 125 µm only when the storage modulus (G') and the loss modulus (G") were measured.

RESULTS AND DISCUSSION

Effect of temperature on viscosity

Alginates function as a stabilizer in foods through its abilities to hydrate and increase viscosity of the system (Onsøyen 1992; Marshall and Arbuckle 1996). The effect of temperature on viscosity of alginate solutions was investigated at a low concentration of alginate. As shown in Figure 1, apparent viscosity increased with increasing alginate concentration. At a fixed alginate concentration, the systems at the higher temperature had a lower viscosity value.

To improve understanding of the molecular properties in dilute solutions of polymers (Rao 1995), the viscosity of the solution and the solvent are used to calculate the specific viscosity (η_{sp}) defined as the following equation.

$$\eta_{sp} = (\eta - \eta_s)/\eta_s \qquad (1)$$

where η and η_s are the viscosities of the solution and the solvent respectively. Values of the specific viscosity (η_{sp}) are considered to be the contribution of polymer to viscosity. reflecting the effective volume occupied by the polymers in the system.

The effect of temperature on specific viscosity values were small and all the results can be represented by a single curve (Figure 2). At a sufficiently low concentration, alginate is considered to exist as disordered random coils in solutions and the individual coils are well separated from one another being free to move independently (Morris 1995). Therefore, the results in Figure 2 indicate that changing temperature from 10°C to 55°C did not change the shape and the size of the individual alginate random coils in solutions.

The influence of temperature on viscosity for a solution was characterized by an Arrhenius relationship

$$\ln \eta_a = \ln \eta_o - E_a/RT \qquad (2)$$

Where η_a is the apparent viscosity at temperature T (K), E_a is activation energy for viscosity, R is the gas constant (= 8.314 J/mole.K) and η_o is a constant. The Arrhenius relationship was studied by plotting ln of viscosity as a function of 1/T (Figure 3). The magnitude of activation energy varied over the range approximately 15.0 -21.8 kJ/mole for the studied polymer solutions but was close to that for water (Table 2). This suggests that the change in the viscosity of polymer solutions with temperature is mainly due to the change in the viscosity of the solvent (water) at low concentration of the polymer.

Fig.

Tab

Effect of temperature on viscoelastic properties

With increasing polymer concentration, the elastic nature of the system due to overlapping or entanglement of polymers becomes pronounced. As a result, the system shows a viscoelastic behavior at a high polymer concentration. In this regime, two separate components of viscoelasticity are obtainable by measuring the stress response of materials to a sinusoidal shear oscillation: the storage modulus, G', (in phase) and the loss modulus, G", (out of phase) which are a measure of the elastic and the viscous component, respectively. On a molecular basis, the magnitude of storage modulus, G', is dependent upon degree of rearrangements taking place within the period of oscillation and the loss modulus, G", is a measure of the energy dissipating as heat per cycle of sinusoidal deformation (Rao 1992; Ross-Murphy 1994).

For the solutions with high alginate concentration (>0.5%), G′ and G″ increased with the increase of alginate (Figure 4). Nonzero value of G′ found in Fig Figure 4 shows alginate-alginate interaction. In addition, G′ and G″ decreased with increasing the studied temperature from the 17.5°C to 51°C. The higher the temperature the lower the modulus values obtained.

Another parameter which is useful in indicating the rheological properties of a system, G"/G' value (so called tan δ; ratio of the energy lost to the energy stored for each cycle of the deformation) was plotted against temperature. The values of G"/G' increased with increasing the temperature of the aqueous alginate solutions but for the higher polymer concentration (2.0%, w/w), the G"/G' values increased less than that of the system with 1.5% (w/w) alginate (Figure 5). This indicated that a higher alginate Fig concentration was found to affect the elastic character rather than the viscous behaviour of systems as the temperature increased because of the overlapping or entanglement of polymer chains.

In conclusion, increasing the temperature of alginate dispersed systems decreased the apparent viscosity for the solution mainly due to the change of viscosity of water because activation energy of alginate aqueous systems evaluated from temperature dependence of viscosity is almost the same as that of water. With increasing alginate concentration, the elastic nature of the alginate/water system became pronounced due to alginate-alginate interaction even at a higher temperature of 55°C.

ACKNOWLEDGEMENTS

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Tables

Table 1 Composition of cation and anion in the alginate powder.

Biopolymer	Moisture content (%)	Amount of cation (%, w/w)			Amount of anion (%, w/w)	
		Na^+	K^+	Ca^{2+}	Cl^-	SO_4^{2-}
Sodium alginate	9.48	8.10	0.07	<0.01	0.08	0.27

Table 2 Activation energies of the aqueous alginate solutions and water

Alginate concentration (%, w/w)	Activation energy (kJ/mole)
0 (water)	16.3
0.01	14.9
0.02	20.0
0.05	16.1
0.09	19.2
0.21	15.9
0.46	18.6
0.91	21.8

Figure Legends

Figure 1 Apparent viscosity of alginate/water systems for different temperatures.

Figure 2 Specific viscosity of alginate/water systems for different temperatures.

Figure 3 Arrhenius plot between ln of viscosity and reciprocal of temperature for different alginate concentrations.

Figure 4 Storage (a) and loss moduli (b) of alginate/water systems for different temperatures.

Figure 5 Effect of alginate concentration on G″/G′ value of alginate/water systems for different temperatures.

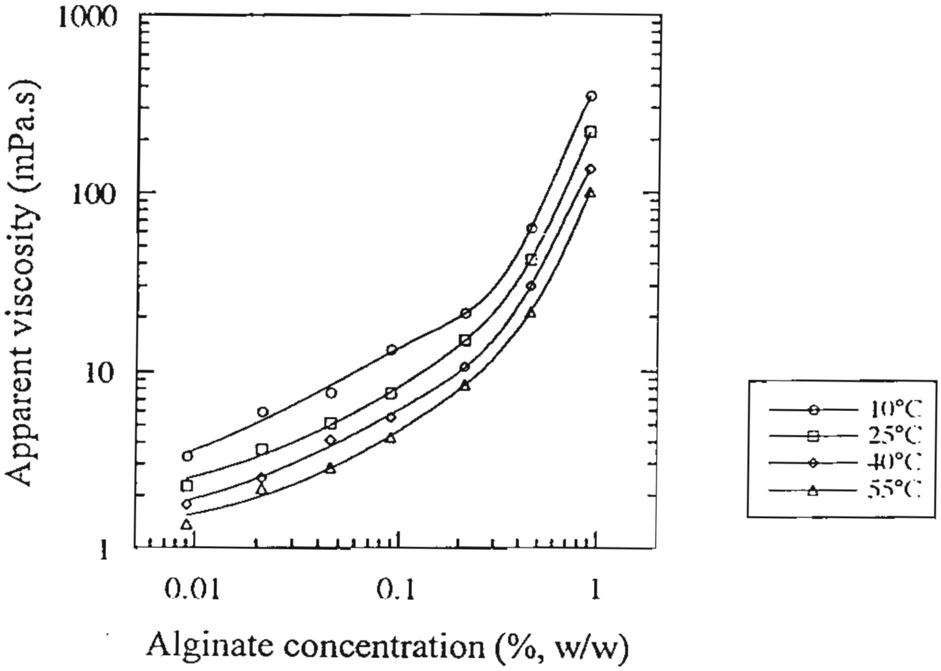

Figure 1 Apparent viscosity of alginate/water systems for different temperatures.

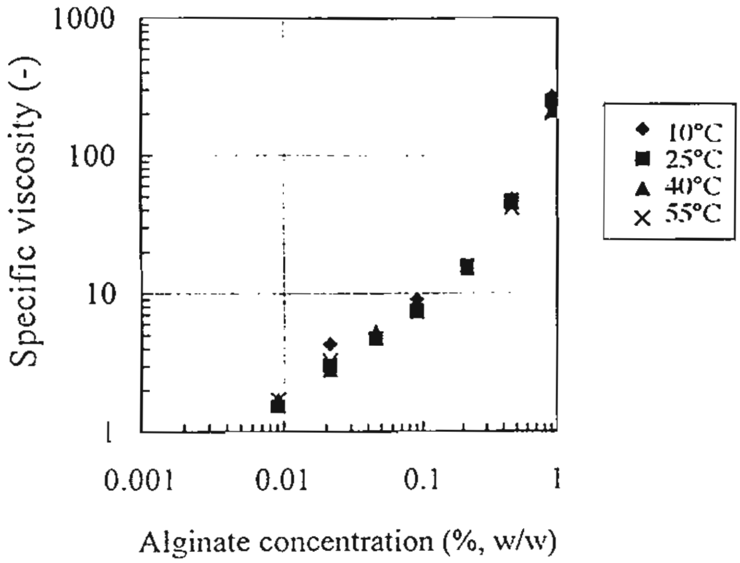

Figure 2 Specific viscosity of alginate/water systems for different temperatures.

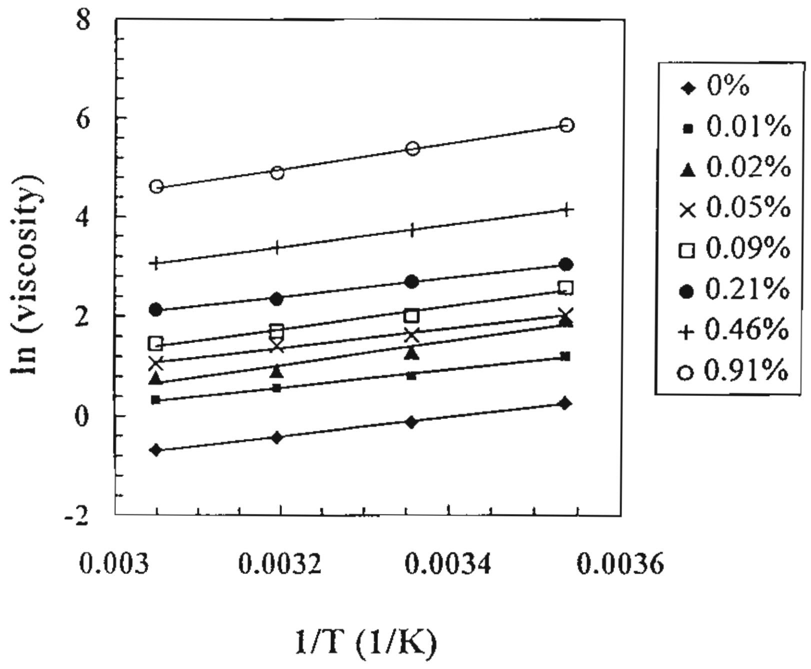

Figure 3 Arrhenius plot between ln of viscosity and reciprocal of temperature for different alginate concentrations.

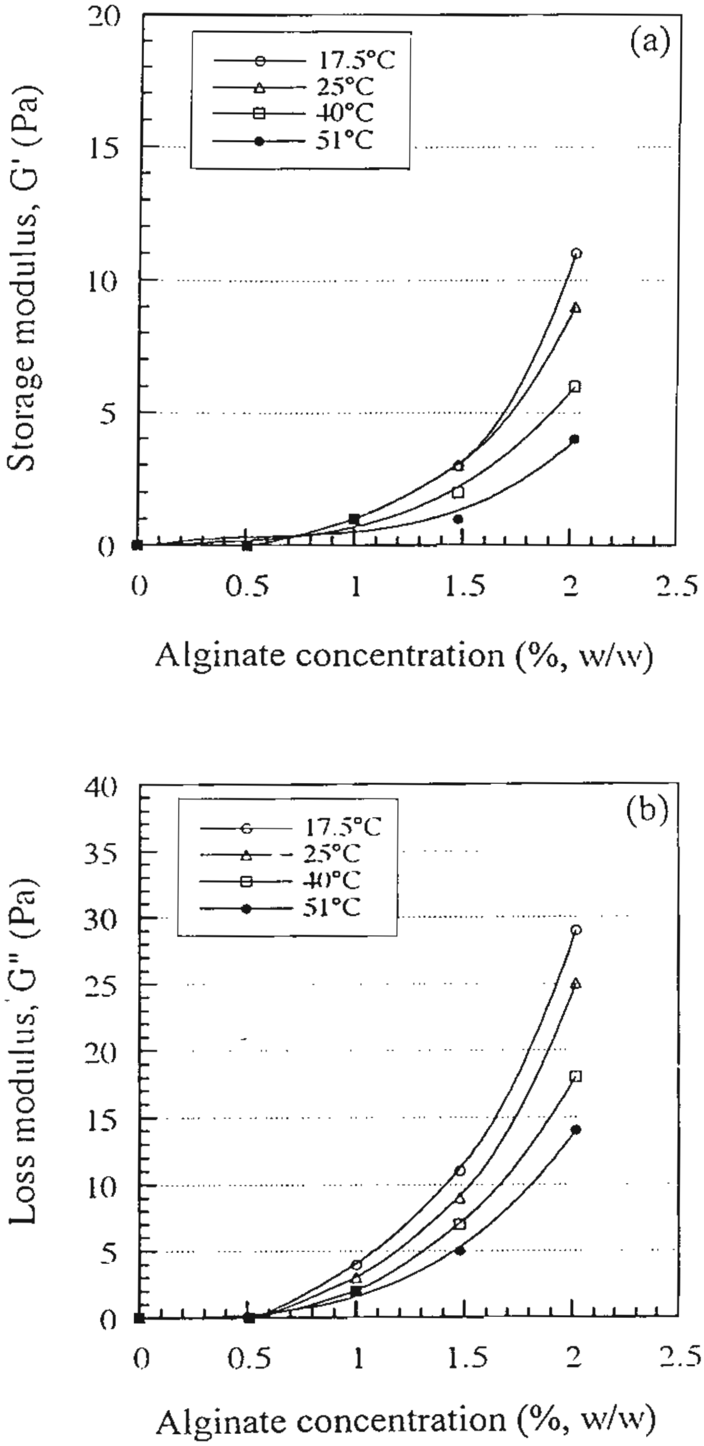

Figure 4 Storage (a) and loss moduli (b) of alginate/water systems for different temperatures.

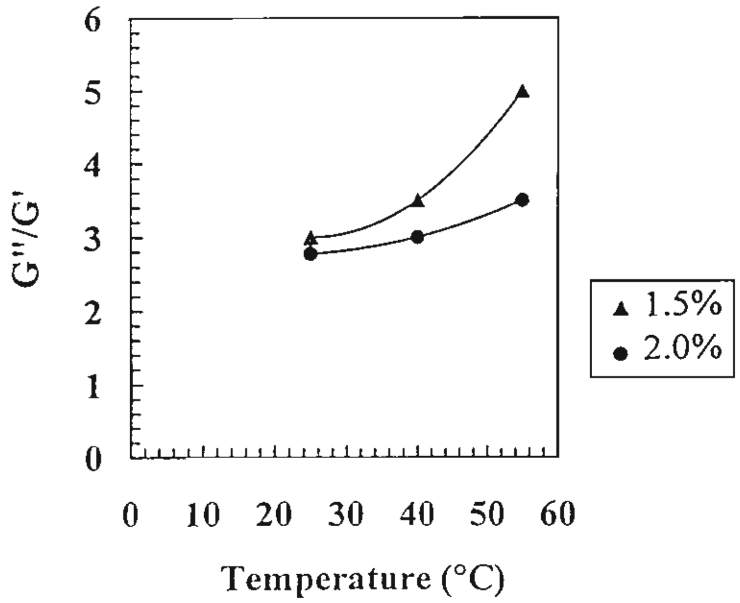

Figure 5 Effect of alginate concentration on G″/G′ value of alginate /water systems for different temperatures.

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EFFECT OF CONCENTRATION AND TEMPERATURE ON VISCOELASTIC PROPERTIES OF AQUEOUS XANTHAN DISPERSIONS

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ABSTRACT

Effect of concentration and temperature on viscoelastic properties of xanthan dispersed aqueous systems was investigated. For low xanthan concentration solutions <0.5%w/w, apparent viscosity increased with increasing concentration and was almost independent on temperature. Specific viscosity values, reflecting the effective volume occupied by the polymer in the system, were represented by a single master curve for the different temperatures of xanthan solutions. The activation energy of xanthan dispersion estimated by the Arrhenius plot between viscosity and the absolute temperature was lower than that of water,. indicating that the viscosity of aqueous xanthan dispersion is less sensitive to temperature than that of water. When a small amplitude dynamic rheological test method was applied to the systems containing higher xanthan concentrations >0.5%w/w, the storage (G') and loss (G") moduli increased with increasing xanthan concentration and G' was much higher than G", exhibiting weak gel-like behavior. The G"/G' (= tan δ) values were almost constant when temperature is increased while their concentration dependence showed a tendency that higher polymer concentration gave lower G"/G' values. These results suggest that xanthan dispersed aqueous systems have stable viscoelastic properties in a wide range of temperature.