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Research Article

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The rheological properties of a gel polysaccharide from *Dictyophora indusiata*

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ABSTRACT

In this paper, a gel polysaccharide DIPs-3 was extracted and purified from the fruiting body of Dictyophora indusiata. Further, the rheological properties of DIPs-3 under different conditions were investigated. DIPs-3 solutions were pseudoplastic fluid and their viscosities decreased with the increasing of shear rates. The linear viscoelastic region of DIPs-3 solutions was determined for 20%～100%. Storage modulus G' and loss modulus G" exhibited strong dependence on concentration and temperature. The increasing of concentration and decreasing of temperature were conducive to the formation of gel. The inferred gel point (c_{gel}) of DIPs-3 solution was between 9×10^{-3} to 1.0×10^{-2} mg/mL at 20 °C and the T_{gel} of DIPs-3 solution was between 5 °C to 10 °C at concentration of 7×10^{-3} g/mL. Through the study on exponent n of $G' \sim \omega^{n}$, it showed that increasing concentration and reducing temperature made DIPs-3 gels had bigger gel strength. These results suggested that DIPs-3 could be used as a kind of natural food additives and polysaccharide gel in food industry.

Keywords: *Dictyophora indusiata*; Gel polysaccharide; Rheological properties.

INTRODUCTION

Polysaccharide is an important part of the organism and widely involved in various life activities. It has good biochemical function, such as antitumor [1, 2], antiviral [3, 4], antioxidant [5, 6] and immunomodulatory activities [7]. Polysaccharide gel, which can form gel under a certain condition, is a special part of polysaccharide family. It has a lot of unique properties and been used in many aspects [8, 9]. In recent years, the researchs on xanthan gum [10], gellan gum [11], carrageenan [12], and other polysaccharide gels developed rapidly.

Rheology is a branch of mechanic. It studies the deformation and flow of material. The research on rheological properties of polysaccharide can provide the viscosity, stress, modulus and other parameters of polysaccharide. There are many researchs on the rheological properties of polysaccharides, especially in the polysaccharide gels [13-15]. Rheological analysis of polysaccharides is meaningful for their application in food industry and it provides useful information for the distribution, manufacture, transport and storage of products**.**

Dictyophora indusiata, belonging to *Phalloidin* families, is a precious edible mushroom with delicious taste and high nutritional values [16, 17]. The polysaccharides were the main active components in *Dictyophora indusiata*, which were triggered to its various health properties [18, 19]. In this study, a polysaccharide fraction DIPs-3 was extracted from the fruiting of *D. indusiata* with 5% NaOH/0.05% $NaBH_4$. Our previous study indicated that the structure of DIPs-3 was mainly (1→6)-linked *β*-D-Glc*p* [20]. Interestingly, DIPs-3 could form gel at room temperature without addition anything else in water, indicating it had the potential as gelling agents in food industry. To our knowledge, it was a novel gel polysaccharide from the mushroom. However, up to now, there was no study on the mechanical characteristic of polysaccharide from *D. indusiata*. The rheological study of polysaccharide solutions was a basic way to understand their thickening properties and gelation in aqueous system [21]. Subsequently, a series of rheological experiments of DIPs-3 were performed. This study could provide valuable data for the application of DIPs-3 in food industry.

EXPERIMENTAL SECTION

2.1 Isolation and purification of polysaccharides

The dry fruiting body of *D. indusiata* was purchased from Qing chuan County, Sichuan province, China. Voucher specimen was deposited in the herbarium of the School of Biotechnology and Food Engineering, Hefei University of Technology (No: DI0001). After crushed into powder, fruiting body of *D. indusiata* was extracted with hot water. The residues were collected and extracted twice with 5%NaOH/0.05% $NaBH_4$ at 25°C for 2h. The extracted solution was neutralized with 36% acetic acid and centrifuged to remove precipitation. The supernatant was treated with 30% H_2O_2 to decolorize and removed proteins by Sevag method [22]. After dialyzed, the supernatant was lyophilized to give crude polysaccharide. The crude polysaccharide was dissolved in distilled water. Acetone was slowly added to the polysaccharide solution with ratio 1:1 (volume ratio). The precipitate was collected by centrifuged at 12000rpm for 30min, dissolved in distilled water, dialyzed and lyophilized to obtain polysaccharide fraction DIPs-3.

2.2 Shear viscosity measurement

DIPs-3 was dissolved in double distilled water with the concentration of 6×10^{-3}, 7×10^{-3}, 8×10^{-3}, 9×10^{-3}, 1.0×10^{-2} and 1.2×10^{-2} g/mL, respectively. The solutions were tested after equilibriumed at 20 °C for 24 h. The shear viscosity was determined by a rotational rheometer (DHR-3, TA, USA) with a 40mm plate with 2° angle. Flow curves with increasing shear rate (0.1～$1000s^{-1}$) were measured at 20°C. The testing temperature was controlled at 20 ± 0.01°C by means of a thermostated water jacket.

2.3 Dynamic viscoelastic behavior

Sample concentrations were 6×10^{-3}, 7×10^{-3}, 8×10^{-3}, 9×10^{-3}, 1.0×10^{-2} and 1.2×10^{-2} g/mL. The dependence of storage modulus *G´*and loss modulus *G"* on strain was determined by strain sweep in strain range of 20%～300% under 1Hz at 20°C. For concentration effect, oscillatory measurements were used to determine the storage modulus (*G´*) and loss modulus (*G"*) of samples. For temperature effect, the solutions with concentrations of 7×10^{-3} and 9×10^{-3}g/mL were measured at various temperatures (5°C, 10°C, 20°C and 30°C).

RESULTS AND DISCUSSION

3.1 Shear viscosity test

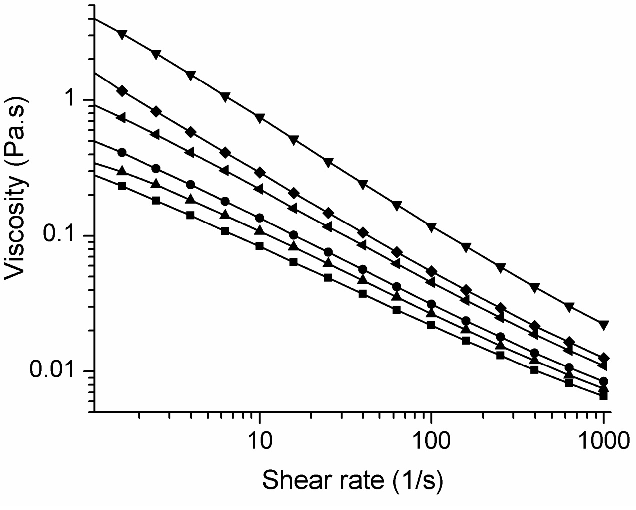

Fig. 1. Dependence of the steady shear viscosity (*η*) on the shear rate for DIPs-3 solutions at 20 °C. The concentrations were 6×10^{-3}, 7×10^{-3}, 8×10^{-3}, 9×10^{-3}, 1.0×10^{-2} and 1.2×10^{-2} g/mL from bottom to top

The dependence of shear viscosity (*η*) on shear rate for DIPs-3 solutions at different concentrations was shown in **Fig. 1**. The viscosity decreased with the increasing of shear rate and the viscosity increased when the concentration increased. It represented a shear-thinning behavior. It had reported that the formula $\eta = k\dot{\gamma}^{n-1}$ could be used to determine the type of fluids [23]. Where *η* represents the shear viscosity, *k* is the front factor, $\dot{\gamma}$ represents the shear rate and *n* is the power law exponent. When the *n* between 0 to 1, it indicated that the fluid was a pseudoplastic fluid. The curves in **Fig. 1** were fitted according to the formula $\eta = k\dot{\gamma}^{n-1}$ and the values of *k* and *n* were given in **Table 1**. All the values of *n* were less than 1 and decreased with the increasing of concentrations. The behavior of DIPs-3 solution was in line with the behavior of pseudoplastic fluids.

It had reported that the polymer in solutions would be arranged in a vertical tubular shape under the action of shear stress before it caused the intermolecular interactions [24]. At this point the shear stress dominated the Brownian motion, so the high polymer solutions had the shear-thinning behavior under low shear rates. The viscosity increased with the increase of concentration may because the spaces between molecules were smaller and the tangles were more likely to happen. Most shear rates were between 0.1~1000 s^{-1} in the process of food processing, the shear-thinning behavior of DIPs-3 solution maybe conducive to its application in food. Similar phenomenon was also found in other polysaccharides [25, 26].

Table 1. Power law model-fitted parameters

Concentration (g/mL)	K	n-1	n	R^2
6×10^{-3}	0.301	-0.562	0.438	0.999
7×10^{-3}	0.396	-0.580	0.420	0.999
8×10^{-3}	0.542	-0.611	0.339	0.999
9×10^{-3}	0.997	-0.662	0.338	0.999
1.0×10^{-2}	1.559	-0.716	0.284	0.999
1.2×10^{-2}	4.375	-0.755	0.245	0.999

3.2 Dependence of modulus on strain

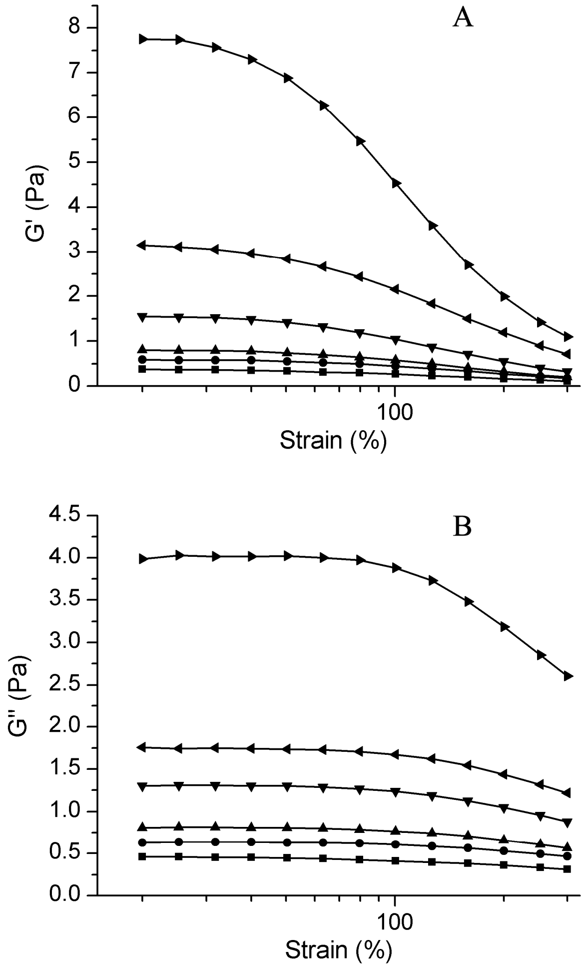

Fig. 2. Storage modulus G′(A) and loss modulus G"(B) as a function of strain for DIPs-3 in water with concentrations of 6×10^{-3}, 7×10^{-3}, 8×10^{-3}, 9×10^{-3}, 1.0×10^{-2} and 1.2×10^{-2} g/mL from bottom to top

The dependence of storage modulus *G′*and loss modulus *G"* on strain was shown in **Fig. 2**. The strain range was from 20% to 300%. It could see that the storage modulus *G′*and loss modulus *G"* were constant in the strain range of

20%～100% under the condition of each concentration. The linear viscoelastic region could be determined for 20%～100%. G' was larger than G'' when the concentration was more than 9×10^{-3} g/mL and G' was smaller than G'' when the concentration was less than 9×10^{-3} g/mL in the linear viscoelastic region.

3.3 Influence of concentration on dynamic viscoelastic behavior

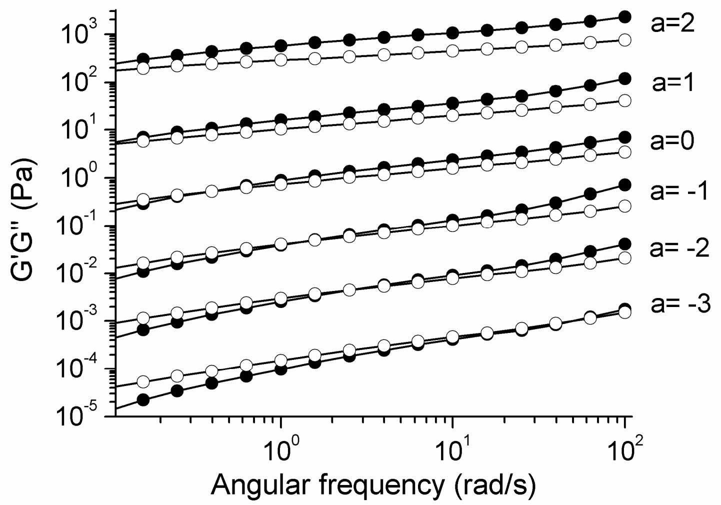

Fig. 3. Storage modulus G' (solid symbols) and loss modulus G'' (open symbols) as a function of angular frequency ω for DIPs-3 solutions at 20°C. The concentrations were 6×10^{-3}, 7×10^{-3}, 8×10^{-3}, 9×10^{-3}, 1.0×10^{-2} and 1.2×10^{-2} g/mL from bottom to top. The data were multiplied by 10^a to avoid overlapping

The dependence of storage modulus G' and loss modulus G'' on angular frequency ω for DIPs-3 solutions at 20°C was shown in **Fig. 3**. Loss modulus G'' was in a major position when the concentration was under 9×10^{-3}mg/mL. It was obvious characteristic of polymer solution. The curves of G' and G'' crossed in intermediate frequency area and they almost overlapped in the whole frequency range when the concentration reached 9×10^{-3}mg/mL. When the concentration was 1.0×10^{-2} mg/mL, G' was larger than G'' and the curves almost parallel to each other. It indicated the formation of weak gels [13, 15]. It could be concluded that the DIPs-3/water system transformed from solution to gel in the concentration range from 9×10^{-3} to 1.0×10^{-2} mg/mL. The gel point c_{gel} could be inferred that between 9×10^{-3} to 1.0×10^{-2} mg/mL at 20 °C. In **Fig. 3**, the curves of G' and G'' crossed at low concentration and the curve intersections tended to appear in low frequency region with the increasing of concentration. When the concentration was greater than 1.0×10^{-2} mg/mL, G' was always bigger than G''. These results suggested that the increasing of concentration was conducive to the formation of gel.

3.4 Influence of temperature on dynamic viscoelastic behavior

The dependence of storage modulus G' and loss modulus G'' on angular frequency ω for DIPs-3 solutions at concentrations of 7×10^{-3} and 9×10^{-3} g/mL under various temperatures was shown in **Fig. 4**. It could see that G' and G'' increased with the increasing of ω in **Fig. 4A**. Two curves intersected in intermediate frequency region at higher temperatures and G' was bigger than G'' in high frequency region. With the decrease of temperature, the frequency at the curve intersection was reduced. When the temperature was 5 °C, G' was always greater than G'' and the two curves were almost parallel to each other, which indicated the formation of gel [13, 15]. It could be concluded that the gel point T_{gel} of DIPs-3 was between 5°C to 10°C at concentration of 7×10^{-3} g/mL. The dependence of modulus on angular frequency ω for 9×10^{-3} g/mL DIPs-3 solutions was shown in **Fig. 4B**. The overall trend was close to **Fig. 4A**. However, there were some differences between them. 9×10^{-3} g/mL DIPs-3 solutions had formed gel at 10°C with G'always greater than G''。It confirmed the conclusion in *3.3* that the increasing of concentration was conducive to the formation of gel at the same time. From **Fig. 4**, it could be concluded that the DIPs-3 solutions were sensitive to temperature. Reducing temperature helps to gel formation.

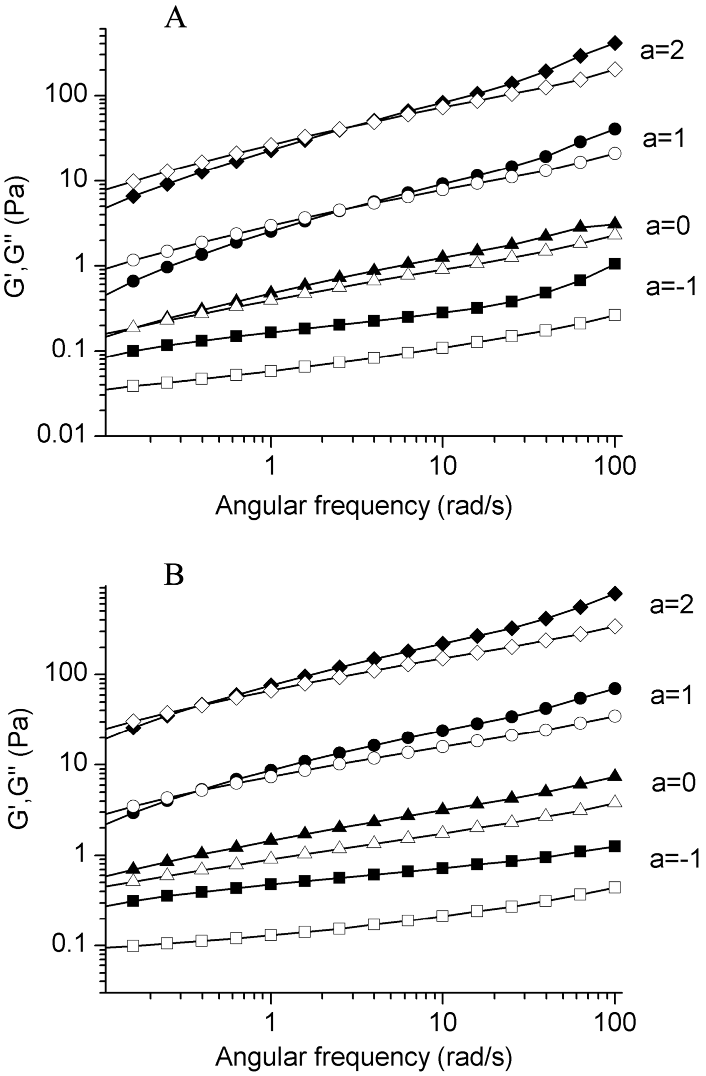

Fig. 4. Storage modulus *G´* (solid symbols) and loss modulus *G"* (open symbols) as a function of angular frequency *ω* for DIPs-3 solutions at concentrations of 7×10^{-3} (A) and 9×10^{-3} (B) g/mL under various temperatures. The temperatures are 30, 20, 10, 5°C from top to bottom. The data were multiplied by 10^{a} to avoid overlapping

3.5 Exponent n along with the change of concentration and temperature

It had reported that the change trend of G' along with the angular frequency could be fitted with formula $G' \sim \omega^n$ [27]. The exponent n of $G' \sim \omega^n$ could be used to determine the behavior of the solution. When exponent n was around 0, system presented the behavior of elastic gel and when exponent n was around 1, system presented the behavior of weak gel [21]. As shown in **Fig. 5A**, the value of n decreased with the increasing of concentration of DIPs-3. It suggested the increasing of gel strength of DIPs-3 solutions. The DIPs-3 solutions tended to form elastic gels. In **Fig. 5B**, the value of n increased with the increasing of temperature. It suggested the decreased of gel strength of DIPs-3 solutions, which confirmed the previous conclusion that low temperature was conducive to gel formation. At the sane time, it illustrated that the gel of DIPs-3 had bigger gel strength at lower temperature.

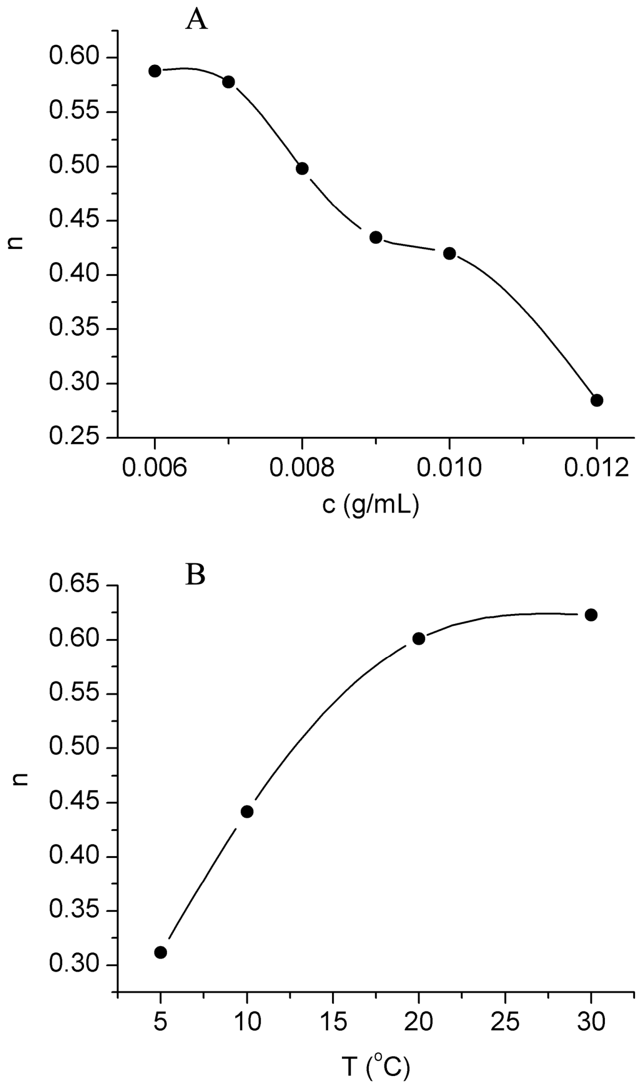

Fig. 5. (A) Exponent n of $G' \sim \omega^n$ as a function of concentration for DIPs-3 solutions at 20 °C; (B) Exponent n of $G' \sim \omega^n$ as a function of temperature for DIPs-3 solutions at concentration of 7×10^{-3} g/mL

CONCLUSION

In this study, a polysaccharide, which could form gel, DIPs-3 was extracted and purified from the fruiting body of *Dictyophora indusiata*, a precious edible mushroom. The rheological properties of DIPs-3 in aqueous solution were investigated. DIPs-3 solutions were pseudoplastic fluids. The linear viscoelastic region of DIPs-3 solutions was determined for 20%～100%. Storage modulus G' and loss modulus G'' exhibited strong dependence on concentration and temperature. The increasing of concentration and decreasing of temperature helps to the formation of gel. The inferred gel point (c_{gel}) of DIPs-3 solution was between 9×10^{-3} to 1.0×10^{-2} mg/mL at 20 °C and the T_{gel} of DIPs-3 solution was between 5°C to 10°C at concentration of 7×10^{-3} g/mL. The study on exponent n of $G' \sim \omega^n$ showed that increasing concentration and reducing temperature made DIPs-3 gels had bigger gel strength. These results suggested that DIPs-3 could be used as a polysaccharide gel in food industry. Also, it provided a better understanding of *Dictyophora indusiata* and useful data for its application in food industry.

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