

Effect of Salt Concentration and Temperature on the Rheological Properties of Guar Gum-Dead Sea Salt Gel

Oyunchimeg Zandraa, Nabanita Saha*, Tomas Saha, Takeshi Kitano, Petr Sába

*Centre of Polymer Systems, University Institute, Tomas Bata University in Zlin,
Tr. T. Bati 5678, Zlin, 76001, Czech Republic*

*Contact e-mail ID: nabanita@ft.utb.cz

Abstract: Dead Sea Salt (DSS) contains 21 minerals including magnesium, calcium, sulfur, bromide, iodine, sodium, zinc and potassium etc. On the other hand, Guar Gum (GG) is a natural polysaccharide, water soluble, having highest molecular weight and good gelling property. Due to such advantageous properties of DSS and GG, it has been taken interest to prepare a user friendly medicated gel (designated as GG-DSS gel) adding additional ingredients: e.g. Glycerol, Thymol, Ethanol, Seabuckthorn oil, and essential oils for the improvement of the gel's medicated values and healing properties for the curing of general skin diseases/treatment. In this study, the viscoelastic behavior of GG-DSS gels were characterized by dynamic storage (G') and loss (G'') moduli and complex viscosity as a function of angular frequency. The effect of DSS concentration and temperature on these properties of GG-DSS gels was investigated. All GG-DSS gels having different amount DSS shows G' higher than G'' over a wide range of angular frequency. Both moduli have the trends to increase with the increase of temperature (temperature varies from 28°C to 50°C), and increase with the increase of DSS concentration higher than about 10%. In conclusion, it can be mentioned that the knowledge about the rheological properties of DSS-GG gels may be useful during the characterization of similar kinds of medicated gel.

Keywords: Dead Sea Salt, Guar Gum, Polysaccharide, Gel, Rheological Property.

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INTRODUCTION

Dead Sea Salt (DSS) is a value based mineral salt, available from natural source, i.e. Dead Sea water. About, 21 minerals such as calcium, sodium, magnesium, bromide, potassium, zinc, sulfur, phosphorus, manganese etc. are present in DSS. These minerals are usually used to detoxify our body (inside) or for nourishing our body from surface which is essential to maintain the proper function and health of skin [1]. Moreover it is reported that twelve vitamins and nine minerals (calcium, copper, iron, magnesium, phosphorus, selenium, zinc, potassium and sodium.) were recognized as essential for skin [2]. Guar gum (GG) is a natural polysaccharide, consists of a linear long chain molecule of β (1 \rightarrow 4)-linked D-mannose residues with single linked α (1 \rightarrow 6)-D-galactose [3]. Moreover, GG as one of the significant naturally occurring non-ionic polysaccharides has broad range of applications in pharmaceutical industry and other commercial sectors which are potential aspirant of natural biopolymer due to its diverse structure, properties, non-toxicity, biodegradability, stability over wide pH range, solubility in hot and cold water and economical processing [4,5]. GG has its unique rheology modifying properties widely used across different industries such as oil well drilling, textile, paper, paint, cement cosmetic, food, pharmaceutical and so on [4]. Due to several advantageous properties of DSS and GG, interest has been taken by the biomaterial research team of Tomas Bata University in Zlin, Czech Republic to prepare a user friendly medicated gel (designated as „GG-DSS gel”). The general properties of GG-DSS gel has already been reported [6,7,8]. This paper focuses about the effect of DSS concentration and temperature on the rheological properties of GG-DSS gel as the knowledge of the rheological properties of GG-DSS gel is important as the microstructural environment of gel or mobility is responsible for drug diffusion (DSS) and compatibility with skin, can indirectly probed using this measurement. Thus, the main objective of this study is to understand the viscoelastic properties of the semisolid product (GG-DSS medicated gel) from practical applications or commercial production points of view.

EXPERIMENTAL

The GG-DSS gel was prepared at room temperature (20-22°C) in two steps, applying simple slow stirring method to achieve the optimum/effective gel composition. To evaluate the effect of salt concentration within the gel, the amount of DSS was varied from 4 to 20 wt.% keeping the content of GG fixed at 1.5 wt. %. The compositions of each series of gel are shown in Tables I.

TABLE 1. Composition of GG-DSS gel (wt. %)

Sample Index	→ 1	2	3	4	5	6	7
Components							
Guar Gum	1.5	1.5	1.5	1.5	1.5	1.5	1.5
Dead Sea Salt	4.0	10.0	11.0	13.0	15.0	17.0	20.0
Glycerol	10.0	10.0	10.0	10.0	10.0	10.0	10.0
Thymol	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Ethanol	2.5	2.5	2.5	2.5	2.5	2.5	2.5
SB oil	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Thym (ess. oil)	0.05	0.05	0.05	0.05	0.05	0.05	0.05
Citrus (ess.oil)	0.05	0.05	0.05	0.05	0.05	0.05	0.05
Water				up to 100 %			

The morphology of GG-DSS gel was investigated using scanning electron microscopy (SEM) (Phenom World Pro) which is operated in the high-vacuum/secondary electron imaging mode at an accelerating voltage of 5–10 kV. The freeze-dried samples were prepared using Lyofilizator CoolSafe 110-4 PRO. The images were taken at the magnification of 100x–10kx.

The physical-chemical behaviour of GG-DSS gel was examined by ATR-FTIR spectroscopic using gels which have the different amounts of DSS content. The spectra were obtained at the wavenumber of 2000-600 cm^{-1} at room temperature with uniform resolution of 2 cm^{-1} . Attenuated total reflectance ATR-FTIR was used with NICOLET 320 FTIR Spectrophotometer with “Omnice” software package.

The rheological properties of GG-DSS gel were performed using a parallel plate type rheometer (ARES, Rheometrics Scientific, USA) with an “RSI Orchestrator” software package. A 25 mm diameter (measuring geometry) parallel plate with a gap of about 1-1.5 mm, was maintained during the whole measurement. An accurate amount of gel put between the two plates, and then upper plate was descended slowly to maintain the gap of measurement. Dynamic viscoelastic properties were measured under different oscillatory flow, employing small strain (1%) amplitude to maintain the measurements within the linear viscoelastic region (LVER). Beside salt concentration, to evaluate the effect of temperature in rheological behaviour (i.e. changes in storage modulus (G'), loss modulus (G'') or complex viscosity (η^*) of GG-DSS gel), the dynamic frequency sweep tests as a function of a wide range of angular frequencies (0.1-100 rad/s) were carried out at 25°C – 50°C under 1% strain. Relation among G' , G'' and η^* is given by the following equation.

$$\eta^* = (G'/\omega)^{1/2} + (G''/\omega)^{1/2} \quad (1)$$

RESULTS AND DISCUSSION

Morphology of GG-DSS gel

The apparent feature of the GG-DSS gel (with and without seabuckthron oil) is presented in Fig.1.1. The GG-DSS gel without seabuckthron oil is white and transparent. The internal structure of each GG-DSS gel (investigated in freeze-dried condition) is depicted in Fig. 1.2 (except sample 2) as the sample containing 10% and 11 % DSS showed more or less similar morphology. It can also be seen from the images in Fig. 1.2 (a) to (f) that there is significant divergence in internal gel structure due to the presence of different amount of DSS (varies between 4-20 wt %). It seems that the amount of DSS plays some influence on internal morphology of the GG-DSS medicated gel.

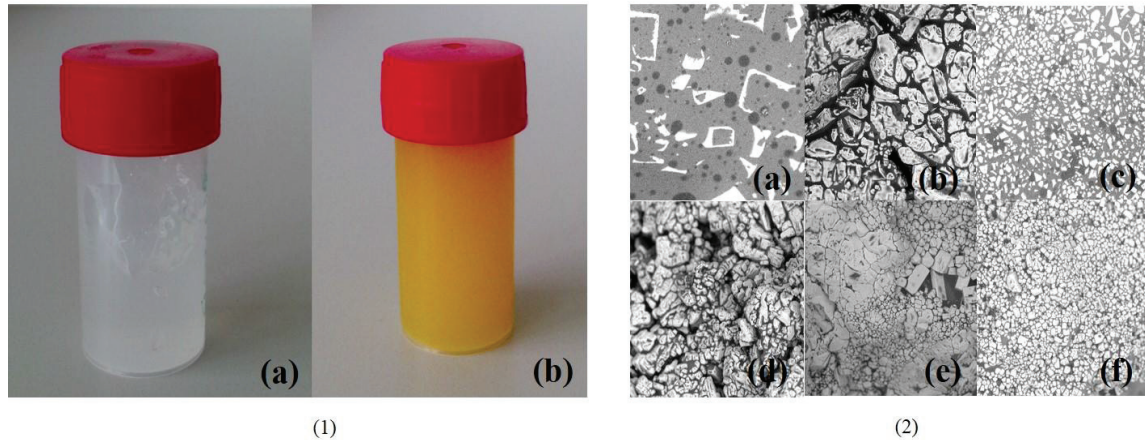


FIGURE.1.1. Visual image of GG-DSS gels: (a) GG-DSS gel without SB oil and (b) GG-DSS gel with SB oil and **FIGURE.1.2.** SEM image of freeze dried GG-DSS gels: (a) DSS: 4%, (b) DSS: 11%, (c) DSS: 13%, (d) DSS: 15%, (e) DSS: 17% and (f) DSS: 20% where, amount of GG: 1.5% is constant in all samples.

The physical-chemical nature of GG-DSS gel

The FTIR spectra of GG-DSS gel at room temperature is shown in Fig. 2. From these figures, it is observed that GG exhibits the characteristic absorption band at 3390 cm^{-1} and 2900 cm^{-1} due to O-H stretching vibrations of the polymer associated with C-H stretching vibrations. Additional information from the characteristic absorption bands of GG appears at 1042 cm^{-1} due to C-H bending and O-H bending vibrations. On the other hand the presence of DSS in GG-DSS gel confirmed due the presence of amide band i.e. 1641 cm^{-1} which is the characteristics of DSS.

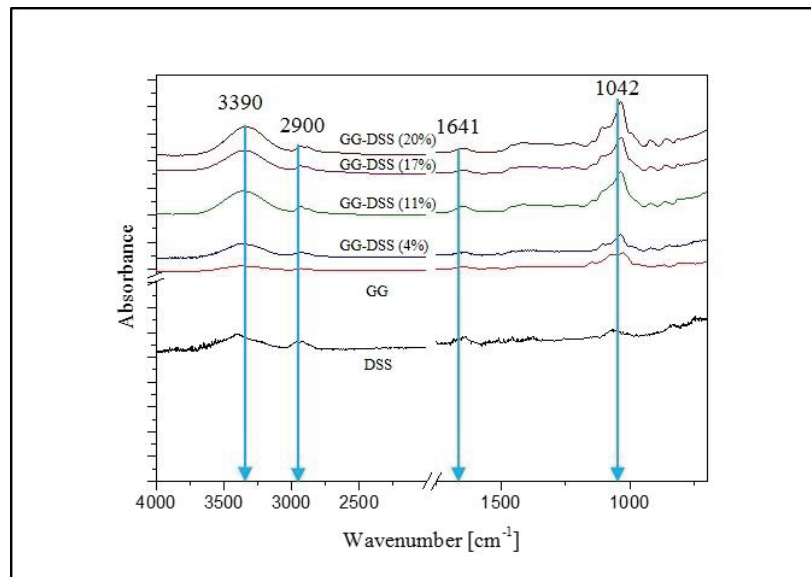


FIGURE.2. FTIR spectra of pure DSS (powder), pure GG (powder), and GG-DSS gels

The Rheological properties of GG-DSS gel

The rheological properties of GG-DSS gels are presented in Figs. 3-6. Figure 3 shows the storage modulus G' , loss modulus G'' and complex viscosity η^* of GG-DSS gels as a function of angular frequency ω at 1% strain and 28°C . From Fig.3, it is found that the storage modulus G' (Fig.3a) increases with increasing DSS content. Loss modulus G'' (3b) increases for gels containing up to 11% of DSS. But, the gels containing more than 13% of DSS, the values of G'' started to decrease gradually. From Fig.3(c), it is found that η^* of all gels decreases linearly with the increase of ω and this behavior means that all the gels show typical pseudo-plasticity.

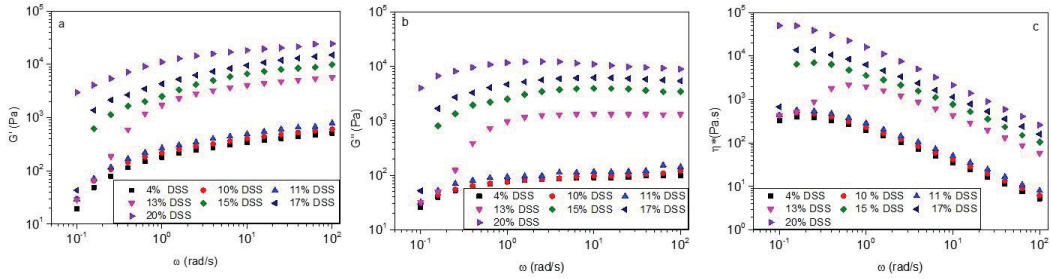


FIGURE 3. Frequency sweep for guar gum gel with different amount of DSS: a) storage modulus, b) loss modulus, and c) complex viscosity.

The influence of concentration of DSS on G' and G'' at different angular frequencies is shown in Fig. 4. Both G' and G'' are almost independent on the concentration of DSS in the region of lower concentration than around 10%, however, these values increase sharply at higher than 10% concentration, and both moduli increase with the increase of angular frequency over whole range of DSS concentration.

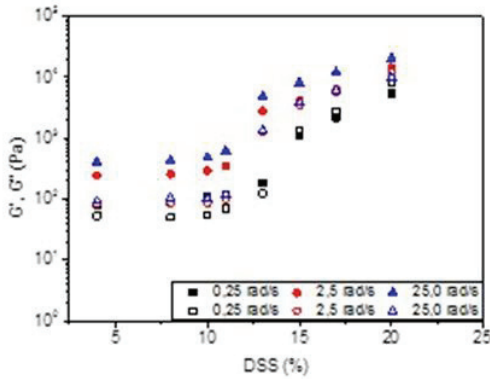


FIGURE 4. Effect of the concentration of GG and DSS on G' and G'' as a parameter of angular frequency (0.25, 2.5, 25.0 rad/s) under 1% strain.

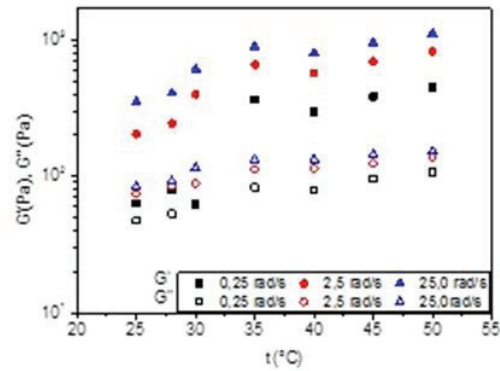


FIGURE 6. Effect of temperature on G' and G'' of GG-DSS gel with 4% of DSS as a parameter of angular frequency (0.25, 2.5, 25.0 rad/s) under 1% strain.

Figs. 5 and 6 show the effect of temperature on the viscoelastic properties of GG-DSS gel. From both of figures, it can be seen that both G' and G'' increase with the increase of temperature that is not typical behaviour of gels. GG is a high molecular weight polysaccharide that can form extensive intermolecular H-bonding resulting in high solvation and thereby increased viscosity [8]. On the other hand, it is important to mention that the test sample of GG-DSS gel is containing the lowest amount (4%) of salts compare to other GG-DSS gels.

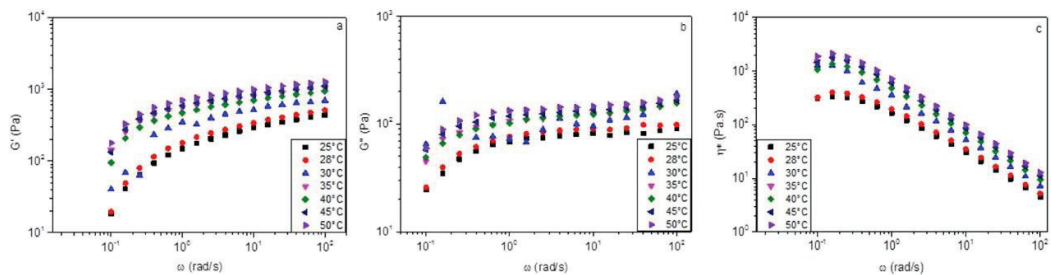


FIGURE 5. Effect of the temperature on G' , G'' and η^* of GG-DSS gel with 4% of DSS.

CONCLUSIONS

- ✓ The knowledge of the rheological properties of GG-DSS gel is important as the microstructural environment of gel or mobility is responsible for drug diffusion (e.g.DSS) and compatibility with skin, can indirectly be probed using this measurement.
- ✓ Hence, the main objective of this study is to measure the viscoelastic properties of GG-DSS gel, especially the influence of *DSS (quantity)* and *temperature (dependent)* on the viscoelastic properties of GG-DSS gel as a function of angular frequency was investigated.
- ✓ The quantity/amount of DSS varies from (4-20) % exhibits a remarkable influence on the formation of internal gel structures as well as physical-chemical properties (see Figs. 1.2 and 2).
- ✓ The viscoelastic properties of GG-DSS gel shows that the storage modulus, G' is higher than the loss modulus G'' over whole range of angular frequency (ω) region, and both moduli increase monotonously with the increase of ω . Both G' and G'' are almost independent on the concentration of DSS in the region of lower concentration than around 10%, however, these values increase sharply at higher than 10% concentration. The complex viscosity, η^* decreases exponentially with the increase of ω .
- ✓ The influences of temperature on GG-DSS gel (having diverse amount of DSS) demonstrated more interesting information i.e. increasing the values of G' and G'' with the increase of temperature; which is not the typical characteristic of gel.
- ✓ Finally, it can be mentioned that the acquired knowledge about rheological properties of DSS-GG gel may be useful during the characterization of similar kinds of other medicated gel.

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