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Study on mechanical behaviour of agar gel in compression mode

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Gels were widely used in food industry as food material or additives, the deformation of gels not only affect the design of the processing equipment, but also affect the formulation and optimization of the processing technology of this kind food materials. In this study, Stress relaxation and strain relaxation experiment were conducted aimed at the sample of gel concentration of 2.5%，5% and 7.5%with a texture analyzer. Based on the experimental data，utilizing Kevin and Maxwell model as basal model, established the four-element model that predict the mechanical behaviour of gels. Experimental results and theoretical models show that: the equilibrium stress of gels was positively correlated with gel concentration; four-element model can accurately describe the mechanical behaviour of food with different concentrations of agar gels and water in the compression, The mechanical model is of important significance for optimization not only in processing technology of food materials with different amounts of gels but also in packaging and shipping process.

Keywords: agar gels, compression velocity, stress relaxation, four-element model

INTRODUCTION

In the food industry, agar gels are commonly used as thickeners, coagulants, and stabilizers in various foods such as pastries, jelly and candies. As a kind of food additive, the agar gel will affect the texture and taste of the food, thus affecting the consumer's choice tendency. Therefore, the research on the mechanical properties of the agar gel is of great significance to the development of the food industry [1, 2].

Gel refers to the colloidal system dispersed phase particles connected to each other to form a network of semi-solid material formed in the gel system, the original dispersion medium filled in the network structure of the gap. The sol loses its original fluidity after gelling and gains elasticity, strength and yield values. For the mechanical properties of gels, lots of researches have been done by scholars. Forte et al. [3] studied the compression and wire-cutting characteristics of gelatin based on the strain rate, and established the fracture model of gels；Shokrieh M M et al. [4] studied the relationship between reinforced polymers and compressive strain rates and proposed a micromechanical model based on strain rate changes. Ma et al [5] studied the influence of agar solution concentration, pH, ionic strength, Na^+, K^+ Composition on the agar gel texture characteristics; Liu Shilin et al [6] discussed the factors affecting the agar gel strength and relaxation characteristics, and on the basis of analysis to research the gel mechanism. At present, scholars focus on the strain rate-related model of the gel mechanics, and the strain rate is constantly changing during the actual processing and storage. Some scholars focus on the relationship between the mechanical properties of the gel and the chemical composition. The relationship between the mechanical properties of the gel and the test parameters has not been studied.

In this paper, the mechanical properties of agar gel with different concentrations under different compression rates were studied by means of a texture analyser. Then the stress relaxation properties of the gel were obtained in the elastic range. The stress relaxation curve was obtained by using the four-element model. The curve has a good fit with the experiment data. The research is of great significance in the study of food texture and the guidance of food processing technology, product quality control and so on.

MATERIALS AND METHODS

Material preparation

(1) Material

Agar powder: food grade, Fujian LvQi Food Colloid Co., Ltd. production, 200 g /bag.

(2) Instruments and equipment

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Texture analyzer: TMS-pro, the United States FTC company; HH-4A digital constant temperature water bath, Jiangsu Jingda; electronic balance; thermometer; beaker.

(3) Preparation

Take 200ml of distilled water with a beaker and heat it to 95 °C in a constant temperature water bath, replenish the evaporated water in the heating process, weigh a certain amount of agar powder, weigh (m), pour into distilled water at 95 °C and stir 5 min after cooling, poured into a cylindrical mold at about 65 °C cooled to room temperature, placed in a 4 °C oven for 20h.

Experimental methods

Respectively, the concentration of 2.5%, 5.0%, 7.5% agar gel preparation, prepared as a cylindrical sample; TA11 universal cylindrical probe (diameter 25.4 mm; length 35.0 mm) experiments at room temperature, the main parameters are as follows:

(1) Uniaxial compression test

Test temperature: room temperature (28 °C); speed before test: 20.0 mm / min; test speed: 1.0 mm / min, 10.0 mm / min,

(2) Stress relaxation test

Test temperature: room temperature (28 °C); pretest speed: 20.0 mm / min; test speed: 10.0 mm / min; constant strain 0.15; stress relaxation time: 30 min.

The above compression experiments and stress relaxation experiments were performed on cylindrical, agar-gel samples of φ22 × 17 mm. Before each test, silicone oil was uniformly applied to the contact surfaces of the probe and the sample so as to minimize the effect of friction on the compression. The effect of the experiment [7], each sample measured 3 times, and finally take the arithmetic mean.

EXPERIMENTAL RESULTS AND DISCUSSION

Uniaxial compression

Assuming that the agar gel volume does not change during compression, the true compressive stress σ_T and the true strain ε_T are in the following relationship:

$$\sigma_T = \frac{F(H_0 - \Delta H)}{\pi R^2 H_0} \quad (1)$$

$$\varepsilon_T = -ln(1 - \frac{\Delta H}{H_0}) \quad (2)$$

Where:

F- compressive force of loading;

H_0- initial height of the experimental sample;

ΔH- compression distance;

R - radius of the experimental sample;

According to the above formula, the real stress σ_T and true strain ε_T of the agar gel in the compression process can be calculated by combining the compressive force F and the compression ΔH collected during the experiment of the texture analyzer.

Fig.1 shows the compressive stress-strain curves of agar gels at 2.5%, 5.0% and 7.5% concentrations at different strain rates (mean calculations for all curves and fitting completed in Origin 9.0). It can be seen that the compression process of colloids can be divided into a linear elastic phase, a non-linear phase of deterioration and a fracture phase. During the elastic phase, the compressive stress of the colloid is less than its critical value, and the stress-strain curve is nearly a straight line. In the nonlinear phase, the damage evolution begins to occur in the material. With the continuous increase of compressive stress, the crack continuously expands and the material stiffness continuously. When the stress reaches the maximum bearing stress, the colloid shows obvious rupture damage. At this moment, the stress drops rapidly. The highest point of the curve is the rupture stress and rupture strain corresponding to the colloid.

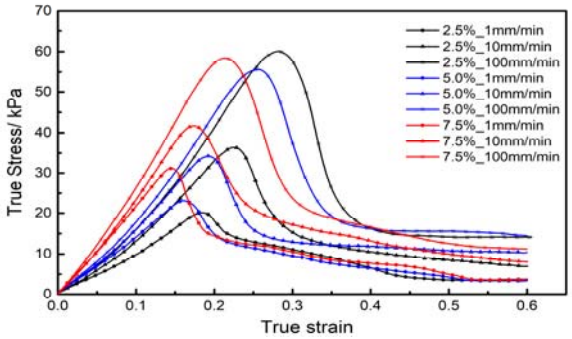


Fig.1. Stress-strain curves of agar gels in various concentrations

At a certain compression speed v_c, the strain rate of the colloid:

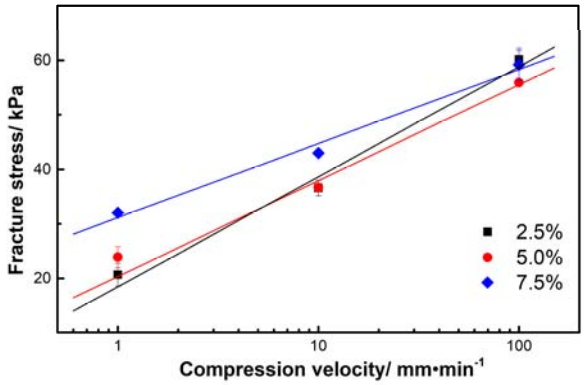
$$\dot{\varepsilon} = \frac{v_c}{H(t)} \quad (3)$$

$$H(t) = H_0 - v_c \cdot t \quad (4)$$

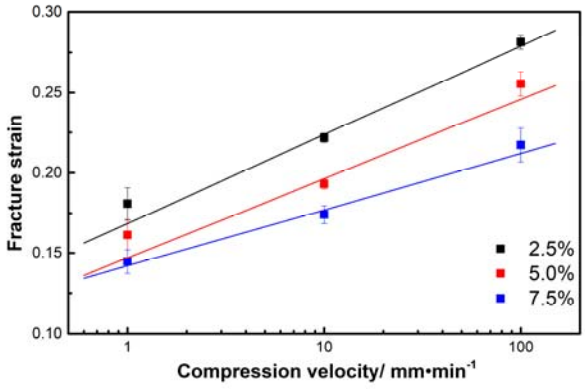
Eq.(3) shows that when the compression rate is a constant, as the sample height decreases, the strain rate of the compression process becomes larger and larger. In the dynamic loading process, due to the stress acting time is very short, there is not enough time to crack development, cannot

reach the energy required for material rupture, it must be higher stress, strain conditions to rupture failure, showing strain rate effect.

Fig.2 shows the fracture stress and strain curves of the colloid at different compression rates. For the compression rate, we use logarithmic coordinates. From the experimental results, the failure stress and failure strain are not only affected by the concentration of colloids, but also depend on the compression speed. As the compression speed increases, the damage stress and failure strain of the colloid are improved. This result is consistent with that of Gamonpilas [8, 9] and Haman [10] et al. It is noteworthy that with the increase of compression speed, the effect of colloidal concentration on fracture stress is getting smaller and smaller; on the contrary, colloids have more and more influence on fracture strain.



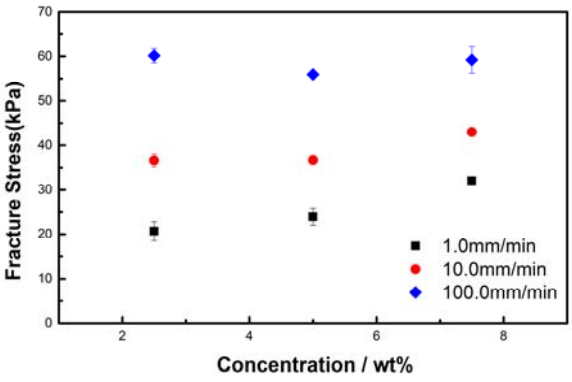
(a) fracture stress



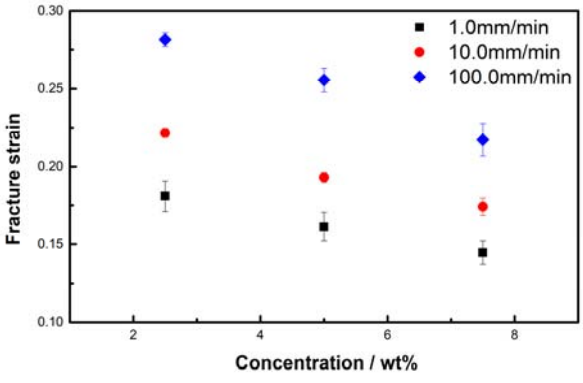
(b) fracture strain

Fig.2. Fracture strength of gels with various compression velocity

Fig.3 shows the relationship between rupture stress and rupture strain and colloidal concentration in the compression process. As the concentration increases, the proportion of dispersoid in the mixture system increases, which leads to the deterioration of the colloidal fluidity. At this time, the inherent toughness effect of the material is more and more. Therefore, at the same compression speed, the colloidal rupture strain decreases with increasing concentration. It can be predicted that even when the concentration reaches a certain level, the colloidal polymer will break even in brittle form. From the experimental results, when the compression speed reaches 100.0 mm / min, the rupture stress of the three concentrations of colloids are 60.16 kPa, 55.95 kPa and 59.22 kPa, respectively. Compared with the case of low compression rate, the effect of stress is weakened. Fig.4 shows the breakdown of 5.0% and 7.5% agarose gels at a compression rate of 10.0 mm / min. It can be seen from the figure that 5.0% colloidal fluidity is good and can be compressed during compression It is clearly observed that the material is extruded (the red frame indicates the outline of the colloidal body); for the 7.5% colloid, the fracture of the material occurs at a lower strain rate due to its reduced fluidity and earlier crack growth during compression (Black curve indicates crack).

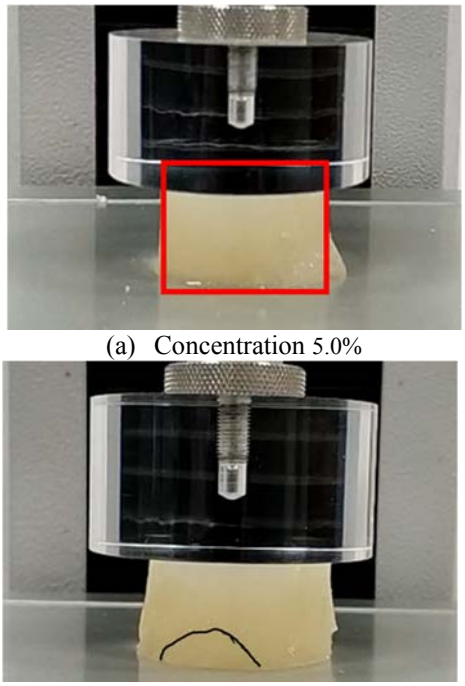


(a) fracture stress



(b) fracture strain

Fig.3. Fracture strength of gel with various concentrations



(a) Concentration 5.0%

(b) Concentration 7.5%

Fig.4. Fracture situation of gels with various concentrations at 10.0mm/min compression velocity

Stress relaxation

Stress relaxation refers to the constant temperature and deformation, stress gradually decay with increasing time. For the colloidal polymer, under the action of external force, the polymer chain is forced to move, resulting in internal stress; in the segment reaches a new equilibrium, the internal stress gradually eliminated. Compared with the linear polymer, the stress of the crosslinked polymer only decays to a certain value σ_∞without falling to zero.

This paper intends to use the four-element model as shown in Fig.5 for an approximate description of the stress relaxation behavior of agar gel. It consists of two Maxwell models in parallel with a spring unit that is designed for cross-linked polymers, Can make internal stress will not drop to zero.

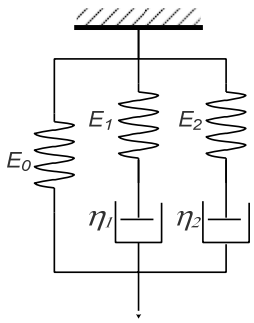


Fig.5. Schematic diagram of four-element model

In the case of constant strain $d\varepsilon/dt = 0$, the stress in the four-element system is distributed to three elements, at this time the equation of motion is:

$$\sigma(t) = \varepsilon_0 E_0 + \varepsilon_0 E_1 e^{-t/\tau_1} + \varepsilon_0 E_2 e^{-t/\tau_2} \quad (5)$$

Where:

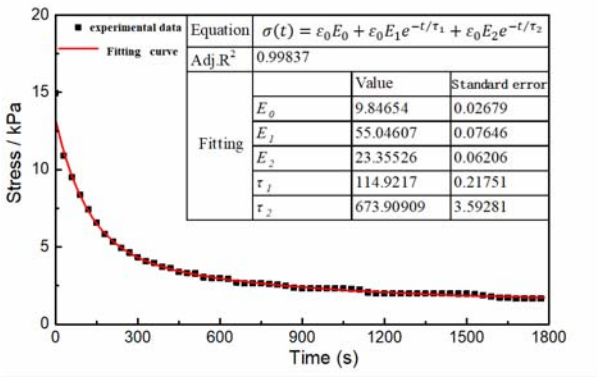
$\sigma(t)$- relaxation stress;

ε_0- the initial strain;

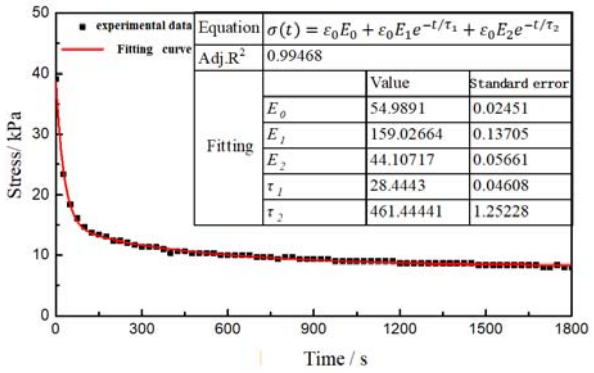
E_i- the spring modulus of the corresponding unit;

η_i -viscosity of the viscous kettle in the corresponding cell;

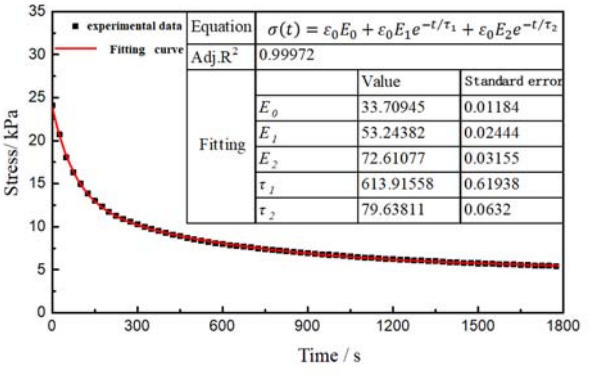
$\tau_i = \eta_i/E_i$-the relaxation time of the ith unit.



(a) Concentration 2.5%



(b) Concentration 5.0%



(c) Concentration 7.5%

Fig.6. Stress relaxation curves and fitting results of gals at 0.15 strain

Based on the elastic range of the agar gel obtained by the compression experiment, the stress relaxation curve of the agar gel can be obtained by keeping the compression probe stationary in this elastic range. Agar gel stress relaxation curves were obtained using a texture analyzer maintaining strain agar at 2.5%, 5.0% and 7.5% at strain rates of 0.15 at different compression rates. Fig.6 shows the experimental data collected at a compression rate of 10.0 mm / min and the fitted results of the fit were customized by Origin using equation (5).

From the fitting results, the coefficient of determination R2 is above 0.99, indicating a good fitting effect; from the characteristics of the curve, the stress balance value was positively correlated with the concentration of colloids, the colloidal concentration is higher, the balance of stress relaxation achieved The value is bigger.

The relaxation time t is defined as the time required for the stress to decrease to 0.368 times the initial stress σ (0), and the relaxation time of the three concentrations of colloid is calculated: $t_{2.5\%}$ = 248.04,$t_{5.0\%}$= 469.4,$t_{7.5\%}$ = 102.7. The concentration of 5.0% of the longest relaxation time of the colloid, the slowest downward trend in stress; 7.5% of the colloidal stress decreased the fastest, in the shortest possible time to reach equilibrium value.

CONCLUSIONS

In this paper, different concentrations of agar gel compression and stress relaxation experiments, the following conclusions:

1. Agar gel is divided into linear elastic phase, non-linear phase and fracture phase in the compression process, and the rupture strength (rupture stress and rupture strain) of gel are affected by concentration and compression rate.

2. Both the rupture stress and the rupture strain of agar gel increase with the increase of the compression rate. However, under the same compression rate, the higher the concentration of colloid, the poorer the fluidity is, the smaller the corresponding rupture strain is. However, There is no clear correspondence between stress and concentration at high compression rates.

3. The defined four-element model can well describe the stress relaxation behavior of agar gel; the higher the concentration, the greater the equilibrium stress, while the relaxation time and concentration have no obvious corresponding relationship.

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