Analysis of Influencing Factors on Viscosity of Agar Solution for Capsules

YU Zhenhua, ZHAN Jianbo*, WANG Hao, ZHENG Han, XIE Jiao, WANG Xu
R&D Centre, China Tobacco Yunnan Industrial Co., Ltd., Kunming, China
yuzh@ynzy-tobacco.com

Abstract—The viscosity of agar solution for capsules depends on the molecular structure and chemical composition of agar. In addition, many external factors also affect its viscosity, such as concentration, temperature, metal ions, electrolyte and non electrolyte, mechanical stirring shear. In this paper, the factors that affect the viscosity of the liquid, which mainly include concentration, temperature and pH, are studied. The results showed that the order of the influence on the viscosity of agar solution was agar concentration > water hardness > solution temperature > pH. Within the range of this experiment, agar concentration, water hardness and solution temperature have significant effects on the viscosity of agar solution, while the pH of solution has no significant effect on the viscosity.

1. Introduction
The aroma compensation technology of cigarettes is mainly the design of flavoring and additive formula and the development of tobacco flavor, and the application mode is mainly cut tobacco flavoring. In recent years, capsule flavor addition has been developing rapidly in the industry with its unique advantages. The technology of capsule addition is a forming technology of cigarette filter. In the production process of filter tip, one or more grains can easily be pinched and broken, so as to achieve the controlled release of special flavors in the process of cigarette smoking. Such a process reduces the influence of external environment on the smell absorption and the loss of flavor, enriching the taste of cigarette smoking, which achieves the humidification in the process of inhalation. Compared with other flavoring technologies, the explosive bead cigarette can stabilize the unstable molecules, realizing the artificially controllable aroma release in the process of cigarette smoking, enriching the taste, reducing the environmental impact on the taste, increasing the aroma.

Capsules own obvious advantages in the transfer rate of spices. It has been reported that aldehydes and ketones with low boiling point are easier to transfer to mainstream smoke than cut tobacco. In addition, compared with cut tobacco flavoring, the flavoring substances do not participate in the combustion and will not produce chemical changes such as cracking, which can realize the direct transfer of spices, which is more intuitive and convenient in the deployment and application of spices. (1) the concentration or content of flavoring or moistening substances carried by capsules can be selected in a wide range during cigarette design, (2) the contents of capsule products less enter into the side stream smoke with high utilization efficiency, (3) the contents of capsule products are stable without participation in combustion, which will not produce harmful smoke, (4) the volatile components can be stored and locked, and the capsules can be squeezed when they need to be released.

Agar is a polysaccharide extracted from seaweed, which is one of the most widely used seaweed gels in the world. Agar is widely used in food industry, medicine industry and bioengineering. Agar can
obviously change the quality of food and improve the grade of food. It has the characteristics of solidification, stability and other physical and chemical properties, and can form complex with some substances. It is a common wall material for capsules. The influence factors of agar solution concentration on the preparation of capsules were studied, and the influencing factors by orthogonal design was analyzed.

2. Experimental

2.1. Materials
Agar, carrageenan, sodium alginate, modified starch, Arabic gum, gellan gum, polyethylene glycol, glycerol, sorbitol. (Analytically pure. Sinopharm Chemical Reagent Co., Ltd.)

2.2. Equipments
Electronic balance (PL-602S, Mettler Toledo Co., Ltd.), heating magnetic stirrer (MS-H-Pro+, Dalong Xingchuang experimental instrument (Beijing) Co., Ltd.), viscometer (NDJ-5S, Shanghai fangruiyiqiao Co., Ltd.), electric stirrer (Germany IKA), electric constant temperature water bath pot (DHG-9070A, Shanghai Jinghong Experimental Equipment Co., Ltd.), Fourier transform infrared spectrometer (NICOLET 5700, Thermonikol Instruments Co., Ltd. USA), X-ray fluorescence probe (EAQLEIII, EDAX Inc., USA).

2.3. Methods
Firstly, the single factor analysis method is used to find the optimal level of each factor, and finally the optimal test scheme of all factors is found. On the basis of this, the orthogonal test was designed to determine the main factors affecting the viscosity and gel strength of the bead wall material, and to explore the effects of each component of the wall material on the viscosity and gel strength of the capsule.

3. RESULTS AND DISCUSSION

3.1. Effect of Concentration on the Viscosity of Agar Solution
At the temperature of 50 ℃, the apparent viscosity of agar solution with different concentrations was measured by rotational viscometer. As shown in Fig.1, when the concentration of agar was 0-3%, the viscosity of the solution increased linearly and slowly with the increase of concentration. When the concentration of agar was greater than 3%, the viscosity of solution increased sharply. The main change is that in the low concentration range, agar colloidal particles have fewer entanglement nodes, which makes it difficult to form large molecular clusters, as characterized by low viscosity. When the concentration increases to a certain extent, the chance of agar molecules contacting each other greatly increases, and macromolecular aggregates are easily formed, which increases the viscosity of the solution significantly.
3.2. Effect of pH on the Viscosity of Agar Solution

Agar solution was prepared with deionized water with acid or alkali solution, and immediately the viscosity of the solution at 65 °C was measured, and the influence of different solutions on the viscosity was investigated. The test results are shown in Fig. 2. It can be seen that with the increase of pH value, the pH value of agar solution firstly increases and then decreases, and reaches the peak viscosity of 59 mPa·s at pH = 7.

3.3. Effect of Temperature on the Viscosity of Agar Solution

The viscosity change of agar solution at different temperatures was measured. The test results are shown in Fig. 3. It can be seen that the viscosity of agar solution decreases with the increase of solution temperature. The higher the temperature is, the greater the decrease range of viscosity is. The reason is that with the decrease of solution temperature, the molecular thermal movement slows down, and the double helix structure is gradually formed, which is conducive to the formation of macromolecular aggregates and the increase of viscosity.
3.4. Effect of Water Hardness on the Viscosity of Agar Solution

According to the research results reported in a large number of previous literatures, metal ions can reduce the viscosity of solution. The reason is that the existence of metal ions will compress the diffusion double layer of charged agar particles, resulting in the decrease of electric potential and electrostatic repulsion force between charged particles, resulting in the curling of molecular chain, which causes the reduction of molecular size and the decrease of viscosity. The higher the electrolyte concentration, the stronger the ability to compress the electric double layer, and the higher the valence of metal ions, the greater the degree of viscosity decrease. Water is the solvent of agar solution. Daily tap water often contains a certain amount of metal salt ions, such as calcium, magnesium, aluminum or iron. Among them, calcium and magnesium ions are the main impurities, and the content is much higher than other metal ions. They are the main metal ions that constitute the hardness of water. According to the provisions of drinking water health standards, the maximum allowable hardness of tap water for daily use is, therefore, this paper mainly studies the influence of water hardness on the viscosity of agar solution in this range.

In the experiment, calcium chloride and magnesium chloride were mixed in proportion (agar concentration 2%) and dissolved in water to prepare agar solution with various hardness. The measurement results of solution viscosity are shown in Fig. 4. With the increase of water hardness, the viscosity of solution decreases rapidly at the beginning. When the hardness of water exceeds, the decrease of viscosity slows down. The experimental results show that a small amount of metal ions, especially high valence metal ions, can make the viscosity of agar solution decrease rapidly. This is because a small amount of metal ions can make its counter ions tend to saturate, and the effect of continuous increase on the viscosity becomes smaller.
3.5. Orthogonal Test Analysis on the Influence of Agar Solution Viscosity

The results of orthogonal experiment design are shown in Table 1.

| No. | Agar concentration(%, w/w) | Temperature (℃) | Water hardness (mg/kg, CaO) | pH | Viscosity (mPa·s) |
|-----|---------------------------|----------------|-----------------------------|----|------------------|
| 1   | 1                         | 1              | 1                           | 1  | 36.5             |
| 2   | 1                         | 2              | 2                           | 2  | 27               |
| 3   | 1                         | 3              | 3                           | 3  | 15               |
| 4   | 2                         | 1              | 2                           | 3  | 73.5             |
| 5   | 2                         | 2              | 3                           | 1  | 52.5             |
| 6   | 2                         | 3              | 1                           | 2  | 74.5             |
| 7   | 3                         | 1              | 3                           | 2  | 469              |
| 8   | 3                         | 2              | 1                           | 3  | 481              |
| 9   | 3                         | 3              | 2                           | 1  | 473              |
| K1  | 78.5                      | 579            | 592                         | 562|
| K2  | 200.5                     | 560.5          | 573.5                       | 570.5|
| K3  | 1423                      | 562.5          | 536.5                       | 569.5|
| K1  | 26.2                      | 193            | 197.3                       | 187.3|
| K2  | 66.8                      | 186.8          | 191.2                       | 190.2|
| K3  | 474.3                     | 187.5          | 178.8                       | 189.8|
| R   | 448.1                     | 6.2            | 18.5                        | 2.9 |

The results of single factor test showed that the concentration, pH value and temperature of agar solution would affect the viscosity of agar solution. In order to compare the influence of these factors
on the viscosity, L9 (3⁴) orthogonal table was selected to set up the orthogonal test. The test results are shown in Table 2.

According to the results of orthogonal test Table 2, comparing the range values of each column, we can see that the order of influence on the viscosity of agar solution is agar concentration > water hardness > solution temperature > pH. Further analysis shows that the concentration of agar, the hardness of water and the temperature of the solution have significant effects on the viscosity of agar solution, but the pH of the solution has no significant effect on the viscosity.

3.6. Stability Control Effect of Capsules

The technical indexes such as capsule size, compressive strength and brittleness are the important basis for judging the quality and stability of bead blasting. Due to the great difference of temperature and humidity in four seasons of the year, how to maintain the stability of various technologies of bead blasting has become a difficult technical problem.

By studying the change of temperature and humidity in different seasons in different years, the temperature in summer is high, the air humidity is large, and the duration is long. Meanwhile, the temperature in winter is low, the air is dry and the humidity is small; the temperature and humidity in spring and autumn are moderate, among which, the ability of anti-moisture absorption is the most important to solve the technical breakthrough. Therefore, on the basis of the previous research on the wall material system, we changed the content of one or two substances in the formula system to determine the agar alginate wall material system, so as to ensure the stability of the technical indicators such as the particle size, compressive strength and brittleness of the explosive beads in summer and winter. Under the condition of standard temperature and humidity, the change rate of hardness and brittleness is within 10%. When the temperature is -15-65 ℃ and humidity is 25-75%, the physical indexes of hardness and brittleness are controlled within the effective embrittlement effect.

Fig.5 Schematic diagram of molecular gel for capsule wall materials

4. Conclusion

The order of the effect on the viscosity of agar solution was agar concentration > water hardness > solution temperature > pH. Within the range of this experiment, agar concentration, water hardness and solution temperature have significant effects on the viscosity of agar solution, while the pH of solution has no significant effect on the viscosity.

The agar alginate wall material system was selected to ensure the stability of technical indexes such as particle size, crushing strength and brittleness in summer and winter. Under the condition of standard temperature and humidity, the change rate of hardness and brittleness is within 10%. When the temperature is -15-65 ℃ and humidity is 25-75%, the physical indexes of hardness and brittleness are controlled within the effective embrittlement effect.

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