Preparation of Chitin Microcapsules and Their Applications in Stability Improvement of Tobacco Aroma Components

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Abstract. Tobacco microcapsules based on chitosan and tobacco extract was prepared in this paper. The preparation yield of microcapsules, tobacco loading rate and embedding rate were studied by calculation. The structures of such microcapsules were characterized by scanning electron microscope and thermogravimetry analysis was used to studied their stability. Else, the taste comfort data of microcapsule samples was also added by Artificial evaluation and scoring. The result showed that microcapsule technology can effectively improve the stability of tobacco products. The amount of tobacco extract had a great influence on the structure of microcapsules. The optimization of tobacco extract content can effectively improve the taste comfort of microcapsules.

1. Introduction

With the rapid development of our society, the demand for tobacco is higher and higher. Excepting the traditional consumption demand, the taste comfort of tobacco smoking should also be improved and the irreversible damage to the body caused by the burning process of tobacco should be reduced [1]. Microcapsules composed of natural or synthetic polymer materials can encapsulate tobacco aroma components, flavors, and fragrances to form micron or nano particles, which can reduce the environmental influence to the tobacco, improve the stability of traditional tobacco products [4]. Spray drying, extrusion, complexation and condensation are the common methods for preparation of tobacco microcapsules [6]. The selection of polymer materials has an important influence on the properties of tobacco microcapsules for all these methods, including stability and safety.

Chitosan is a kind of polysaccharide after deacetylation of chitin by chemical or enzyme treatment [8]. It has good biological activity, special physical and chemical properties, such as antibacterial, hemostatic, anti-inflammatory, and so on [10]. Chitin are mainly prepared from many lower arthropod shells and marine organism. So, chitosan is a natural polymer and it is safer for the human body. Else, molecular weight and solubility can be accurate controlled by adjusting the corresponding prepared conditions, which provides favorable conditions for the preparation of tobacco flavor capsules with better quality. So, tobacco microcapsules based on chitosan were prepared and evaluated in this paper.

2. Experimental

2.1 Materials and Instruments

All the chemicals were purchased from Aldrich or Beijing Lanyi Chemical co. ltd and were used as received unless otherwise specified. All the organic solvents were distilled before use. Chitosan
(average Mw 5,000-10000) were purchased from Top science Chemical co. ltd. The tobacco extract was extracted from tobacco leaves according our previous papers [12]. Thermogravimetric analysis (TGA) was determined by a TA instrument Q50 with a heating rate of 10 oC min-1 under nitrogen. The surface morphology of the samples was carried out via Carl Zeiss Sigma field emission scanning electron microscopy (FESEM).

2.2 Preparation of Tobacco Microcapsules
8 g chitosan and 32 g distilled water were added into a round bottomed flask and stirred for 45 min at 500 r/ min to disperse chitosan. A certain number of tobacco extract including flavors, spices and nicotine was added into the aqueous solution of chitosan slowly. After the addition, the reaction was carried out at 50 °C for 4 h at 500 r/min. After the reaction, when the reaction system temperature drops to room temperature, the precipitated solid is filtered, washed, and freeze-dried affording microcapsules.

3. Results and Discussion
3.1 Evaluation of Embedding Effect of Microcapsules
To evaluate the embedding effect of such tobacco microcapsules, the yield of microcapsules, microcapsule loading rate and the embedding rate of microcapsules to tobacco extract were measured and listed in Table 1. Obviously, chitosan showed us high yield of microcapsules (above 82%), tobacco microcapsule loading rate (above 13%) and embedding rate (above 46.1%). As the increasing of tobacco extract, the yield and embedding rate have a little reduce and the tobacco extract loading rate increases greatly (from 13.0% to 18.7%).

Table 1. Yield, loading and embedding efficiencies of microcapsules

| Sample  | Chitosan (g) | Tobacco extract (g) | Yield of microcapsules (%)<sup>a</sup> | Microcapsule loading rate (%)<sup>b</sup> | Embedding rate of microcapsules<sup>c</sup> |
|---------|--------------|---------------------|----------------------------------------|------------------------------------------|------------------------------------------|
| sample1 | 8            | 2                   | 92.1                                   | 13.0                                     | 60.1                                     |
| sample2 | 8            | 3                   | 86.0                                   | 15.4                                     | 48.7                                     |
| sample3 | 8            | 4                   | 82.1                                   | 18.7                                     | 46.1                                     |

<sup>a</sup> The yield of microcapsules = mass of microcapsules after drying / (mass of chitosan + mass of tobacco extract) × 100%.

<sup>b</sup> Microcapsule loading rate = mass of tobacco extract in microcapsule / mass of microcapsule after drying × 100%.

<sup>c</sup> The embedding rate of microcapsules to tobacco extract = mass of tobacco extract in microcapsule / tobacco extract × 100%.

3.2 Analysis of Particle Size and Morphology of Microcapsules
The particle size and distribution of microcapsules were determined by nano particle size and potentiometry. The detection temperature was 25°C. Scanning electron microscope was used to observe and photograph the micro morphology of the microcapsules. The specific parameters were as follows: magnification 1000 times, accelerating voltage 15.0 kV, current 75 mA.
Figure 1. SEM micrographs of microcapsule samples and chitosan

Figure 1 shows the SEM results of chitosan and three tobacco microcapsule samples. It can be seen that the shape and size are different between chitosan and else three tobacco microcapsules samples. The surface of chitosan particles is rough and the edge is irregular, which indicates that it is amorphous; the surfaces of three tobacco microcapsules samples are smooth, showing a tight and regular crystal state. The difference of shape and size between chitosan and else three tobacco microcapsules samples indicate that new stable inclusion state was formed between tobacco extract and chitosan, and the embedding effect is ideal.

3.3 Thermal Stability Analysis of Microcapsules
The thermal stability of microcapsules was analyzed by thermogravimetry analysis (TGA). Analysis conditions: sample crucible material: alumina; test temperature range: room temperature ~ 500 °C; heating rate: 10 °C / min; protective reaction gas: nitrogen; flow rate: 50 ml / min.
Figure 2. TGA curves of microcapsule samples

The results of thermal stability of tobacco microcapsules are shown in Figure 2. It can be seen from Figure 2 that the thermogravimetric process of sample 3 occurs between 100-500 °C, and its mass loss is close to 100%. The weight loss is mainly caused by its violent volatilization before 250 °C. The weight loss is mainly caused by chemical decomposition of chitosan from 250 °C to 500 °C. Comparing with sample 3, thermogravimetric process of sample 1 and sample 2 are different. The weight loss starting temperatures were improved to 300 °C and their mass loss are also close to 100% around 500 °C. Such a phenomenon indicated that microcapsules with low tobacco extract loading rate have stable structure due to the strong interaction among the chitosan macromolecule.

3.4 Evaluation of Taste Comfort of Microcapsule Samples

Table 2. Sensory evaluation results of microcapsule

| sample | Aroma  | Harmony | Irritation | Off-flavour | Agreeable aftertaste | Total  |
|--------|--------|---------|------------|-------------|----------------------|--------|
| 1      | 18.06  | 15.31   | 17.03      | 12.38       | 13.23                | 76.01  |
| 2      | 18.68  | 13.52   | 16.31      | 16.07       | 14.09                | 78.67  |
| 3      | 19.01  | 11.07   | 14.22      | 16.01       | 13.07                | 73.38  |

The evaluation results of taste comfort of microcapsule samples were shown in Table 2. Sample 1 showed advantages in harmony and irritation among them. Sample 2 showed advantages in agreeable aftertaste and off-flavour. Sample 3 showed advantages in aroma. And for the total score, sample 2 performed the best. So, there is a certain balance between the loading rate of tobacco extract and the structural characteristics of microcapsules. This balance is of great significance for the preparation of high-quality tobacco capsules.

4. Conclusion

Tobacco microcapsules were prepared using chitosan as embedding polymer and tobacco extract as core materials. The yield of microcapsules can reach 92.1%. The highest embedding rate reached 46.1% and the tobacco extract loading rate in the microcapsules reached 18.7%. The result of scanning electron microscope showed that the samples have different shape and size, which indicated all the samples were on the status of microcapsules. Thermogravimetry analysis indicated that as the increasing of tobacco extract addition, the thermal stability reduced greatly. The taste comfort data of microcapsule samples indicated that microcapsule technology can effectively improve the stability of
tobacco products. The optimization of tobacco extract content can effectively improve the taste comfort of microcapsules.

5. Reference

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