Study on Preparation and Stability of Ginger Oil o/w microemulsion

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Abstract. In order to enhance the stability of ginger oil emulsions, ultrasound-assisted emulsification method was used for the preparation of ginger oil emulsions in this paper. The optimization was based on single factor and response surface optimization experiments. Four main factors in the preparation process (amount of emulsifier, ultrasonic power, time and temperature) were investigated. According to the visual analyses of the result, the optimum condition was obtained. Based on the optimal condition in the response surface optimization experiments, the preparation process was then conducted under ultrasonic emulsification. (The effects of ultrasound amplitude and irradiation time on the encapsulation process were also studied). The microemulsion type, grain size, and phase transition temperature were used to characterize the resulting sample. Finally, the average particle size of ginger oil nano-emulsion attained 94.49 nm in the condition of ultrasonic power 250W, ultrasonic time 5.8 min, ultrasonic temperature 50 °C, emulsifier concentration of 1.0 g / 10 ml, implying that the ultrasonic emulsification technology can solve the difficulty of macromolecular oil microemulsion preparation.

1. Introduction

Ginger oil is a general term for a type of ginger extract composed of volatile ginger essential oil and non-volatile resin oil[1]. Ginger oil has not only the faction of anti-oxidation, ant-bacteria, anti-inflammatory analgesic, anti-cancer, but it can protect the stomach and improve the cardiovascular system[2]. Ginger oil has a strong aromatic scent and contains almost no high-boiling ingredients. It was mainly used for the seasoning and flavoring of foods and beverages. Meanwhile, it was a kind of extremely expensive medicinal and fragrance raw material which is required by domestic and foreign markets[3]. At present, it is widely used in food and drug industry. However, due to the astringent taste of ginger oil, its taste is not good. At the same time, the hydrophilicity of the oil makes it swell and condense in water, so that its solubility and dispersion are poor and there are a number of obstacles to use it under water.

Ultrasonic emulsification technology[4] is an emerging technology for the preparation of nano-emulsions in the food field. Compared with the traditional emulsification technology, the ultrasonic emulsification technology has the advantages of small emulsion particle size, narrow distribution, stable emulsion, less dem and for surfactant, low production cost, easy operation, low pollution, high efficiency and energy saving.
In this paper the optimal preparation process of ginger oil microemulsion was obtained by ultrasonic emulsification method using response surface optimization experiment. Therefore, a nano-scale ginger oil microemulsion can be improved not only to solve the difficulty of preparation of macromolecular oil microemulsion [5, 6], and expand the application range of ginger oil, but to provide a referance method for the further development of the nutrient and nano-functional food as well.

2. Experimental

2.1. Material
Ginger oil, Food grade Guangzhou qianqiu cosmetics co., LTD; Distilled water, Polyethylene glycol (peg) 400, span, span 80, twain 20, 80, twain food-grade, petroleum chemical plant in jiangsu province; D - 95 distillation glycerin monostearate, food grade, plus its additives (MSC) co., LTD.; SE - 11 sucrose fatty acid ester, food grade, hangzhou gonadorelin chemical co., LTD.; Polyglycerol fatty acid ester, food grade, shandong superior chemical technology co., LTD.; Rosin glyceride, food-grade, wuxi double chemical co., LTD.

2.2. Process optimization

2.2.1. The test of single factor. In order to reduce the emulsion preparation time, improve the experimental effect and the production efficiency, the response surface optimization experiment method was used to optimize the process flow based on the range of reaction conditions determined by single factor test.

Ultrasonic power, ultrasonic time, ultrasonic temperature and emulsifier concentration were taken as the investigation factors. The average particle size of the prepared ginger oil microemulsion was determined by single factor test.

| level | Amount of emulsifier/g/10ml | Ultrasonic power/W | time /min | Temperature /℃ |
|-------|-----------------------------|--------------------|-----------|-----------------|
| 1     | 0.2                         | 0                  | 5         | 30              |
| 2     | 0.4                         | 100                | 10        | 35              |
| 3     | 0.6                         | 150                | 15        | 40              |
| 4     | 0.8                         | 200                | 20        | 45              |
| 5     | 1.0                         | 250                | 25        | 50              |

2.2.2. The response surface optimization test. Based on the results of the single factor test, Design Expert 8.0 software was used to design and analyze the response surface tests. The average particle size of the nano-emulsion was used as the response value, and the center combination test Box-Behnken Design was used to select the four factors and three levels for the experimental design.

3. Result

3.1. The test of single factor
As shown in fig1. (a), when the ultrasonic power was 150 W, the microemulsion had the lowest average particle size of 207 nm. In the occasion of ultrasonic power was increased from 150W to 200W, the average particle size was slightly increased. This phenomenon was called the over-treatment effect. take into consideration the polymerization of emulsion particles was caused by the high-intensity ultrasonic effect[7], the range of optimal ultrasonic power should be selected from 100W to 200W.

As shown in fig1. (b), ultrasonic treatment within 15 min can significantly reduce the particle size of the emulsion, so the ultrasonic treatment time should be from 5 min to 15 min.
As shown in fig1. (c), the average particle size of the emulsion reached the lowest point at the degree of 35, which was 284 nm, indicating that the influence of temperature may be non-significant as other factors, so the best optimization range of the temperature was from 35 to 50.

As shown in fig1. (d), when the mass concentration of emulsifier was 0~0.6g/10mL, the average particle size of the emulsion tends to decrease. And the average particle size of the emulsion decreases the most. When the concentration of the emulsifier was more than 0.8 g/10 mL, the average particle diameter increases as the concentration of the emulsifier increases. This was because the concentration of the emulsifier was too large and the excess emulsifier interacts to cause polymerization of the emulsion particles [8, 9]. This phenomenon can also be explained by the theory of repulsive flocculation [10, 11], that was, when the concentration of the polymer emulsifier exceeds the critical value the modified starch which was not adsorbed in the continuous phase would cause flocculation of the droplets due to the osmosis, thereby causing the molecular chain of the modified starch to be The surface of the two droplets repels out, indicating that the presence of excess emulsifier affected the uniformity of the emulsion, which in turn affected the stability of the emulsion[12]. Considering comprehensively, the mass concentration of emulsifier of 0.6~1.00g/10mL was most suitable for the preparation of ginger oil micro-emulsion.

3.2. The test of response surface optimization
The response surface optimization test design and results are shown as follow. The results were analyzed by using Design Expert 8.0 software to obtain the regression model equation.

\[ R=-513.45+1001.66A+7.53B-31.08C-9.90D-1.12AB-3.25AC-6.00AD+0.03BC-0.02BD+0.31CD-280.20A^2-0.01B^2+0.77C^2+0.16D^2 \]

(And a, b, c, d indicates the emulsifier mass I concentration, ultrasonic power, time temperature coded, values). The regression analysis and variance analysis results were shown in as follow. The f-value of the model was 9.24 and the p-value<0.001 meanted that the model was significant. The model R2=90.23%, indicating that the mode fit was good and statistically significant. The missing term f=0.19, p=0.5322>0.05, and the coefficient of variation was small. CV=7.71%<10% indicating that the model was not missing, the experimental error was small, and the actual situation could be fully reflected. The mode was established.
The results of the other party's difference analysis were centrally standardized. It could be seen that the primary term b, c, d and quadratic b² and c² in the equation were extremely significant (p<0.01) and the other effects were not significant (p>0.05). Using proc glm to process the model [13] and retaining the term of p<0.04 could get an optimized regression model:

\[ R = -513.45 + 7.53B - 31.08C - 9.90D - 0.01B^2 + 0.77C^2 \]

The order in which the variables affect the main effect was the ultrasonic action temperature > ultrasonic power > ultrasonic time > emulsifier addition.

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**Figure 2.** 3D surface plots and contour plots of the aqueous separation after hot storage

It can be seen from Fig.2a that as the ultrasonic power increases, the average particle size of the emulsion increases at first and then decreases, reaching a maximum at 20W, while the effect of the addition of emulsifier and ultrasonic time is not significant. In Figure 2d, 2e in the middle, when the ultrasonic power is low, the ultrasonic temperature and time have little effect on the average particle size of the emulsion. When the ultrasonic power is high, the interaction between the ultrasonic temperature and the ultrasonic temperature (ultrasound time) is more obvious. With the ultrasonic power increaseing, the higher the ultrasonic temperature (the less the ultrasonic time)is, the smaller the average...
particle size of the emulsion would be. Figures 2b and 2c show that the mass concentration of emulsifier has no significant effect on ultrasonic temperature if time is in the range of 0.6~0.8g/10ml; in figure 2f, the interaction between ultrasonic time and ultrasonic temperature is obvious. The decrease in time and the increase in ultrasonic temperature result in a significant decrease in the average particle size of the emulsion.

The conditions were optimized by Design Expert 8.0 software. The optimization result was ultrasonic power of 250W, ultrasonic time of 5.8min ultrasonic temperature of 50℃ and emulsifier mass concentration of 1.0g/10mL. The average particle size of the nano-emulsion prepared under these conditions was predicted to be 94.4957m. According to this condition, the average particle size of the emulsion was (94+/-10.23) nm, which was close to the predicted value, which proved that the model was reasonable.

3.3. The study of the property
A. The type of emulsion was identified by staining[14]. Sultan red dye and methylene blue dye were selected. According to the oil-soluble characteristics of sultan red, the dyes of Sultan red and methylene blue dye were added into the emulsion. The result showed that the diffusion rate of the oily dye Sultan red in the emulsion was lower than that of water (alcohol) of the methylene blue dye which proved that the obtained emulsion was of an oil-in-water type.

B. The particle size distribution of the obtained micro-emulsion was measured by a laser particle size analyzer, and its average particle diameter was 94.4957 nm.

C. 40 mL of the newly prepared microemulsion was taken and stirred in a thermostatic magnetic device while controlling the temperature of the water bath to uniformly heat the temperature at a rate of about 5℃/min. The conductivity meter electrode was placed therein to determine the change in conductivity of the micro-emulsion between 35 and 100℃. As it is shown in Fig. 2, the conductivity increases at 35~85℃ and then decreases rapidly, indicating that 85℃ is its phase transition temperature, so that the prepared micro-emulsion has good thermal stability at 85℃.

4. Conclusion
In order to overcome the water insoluble drawback of ginger oil in this paper, the ginger oil nano-emulsion was prepared by ultrasonic emulsification method. The optimum experimental conditions were obtained as follows: ultrasonic power 250W, ultrasonic time 5.8 min, ultrasonic temperature 50℃, emulsifier concentration of 1.0 g/10 ml, the best ginger oil nano-emulsion is achieved with the average particle size is 94.4967 nm.

The study of the property analysis demonstrated that ginger oil nano-emulsion possess a good thermal stability under 85℃ temperature, which has practical significance in the production.

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