Experimental Study on the Effect of High Pressure Micro-jet Parameters on Homogeneity Performance

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Abstract. High-pressure micro-jet homogenization technology is one of the commonly used methods in the preparation of liquid chemicals. The study on the influence of the parameters of high pressure micro jet on the homogeneity of the agential is helpful to improve the efficacy and safety of the agential. In this paper, using high pressure homogenizer as experimental platform, the oil-water emulsion as homogeneous material, the variation law of homogeneous particle size under the conditions of homogeneous pressure, initial temperature and oil / water ratio was studied. The experimental results show that the particle size of oil droplets decreases with the increase of homogenization pressure; the particle size of oil droplets increases with the increase of initial temperature; and the particle size of oil droplets decreases with the decrease of oil/water ratio. The experimental basis is provided to improve the homogeneity of high pressure micro jet homogenizer.

Introduction

High pressure micro-jet is under the action of ultra-high pressure, through the tiny diameter of the valve core, resulting in several times the speed of the fluid. The technology was first proposed in the 1970s, but it was not fully studied until the 1990s. The formation of micro-jet is mainly in two forms, one is closed cavity of a small opening on one side forming jet actuator. During operation, the opposite side of the opening vibrates, and the external fluid enters and discharges the cavity, to form the micro-jet; The other is to directly put the diaphragm into the environmental fluid, the diaphragm vibration as long as its amplitude is large enough, but also along the normal direction of the formation of jet. During operation, the opposite side of the opening vibrates, and the external fluid enters and discharges the cavity, forming the micro-jet. The other is to directly put the diaphragm into the environmental fluid, the diaphragm vibration as long as its amplitude is large enough, also along the normal direction of the formation of jet.

High pressure micro jet application in aerodynamics, chemical industry, food, pharmaceutical preparations and other fields. Micro-jet method is also a new method of preparation of micro-nanoemulsion. In the pharmaceutical, with no damage to the active ingredients of drugs in the case of reducing the size of the drug to the drug particle size distribution and so on. Siah et al. [1] prepared an aspirin nanoemulsion by combining ultrasound with high pressure micro jetting, with an average particle size of 146.1 nm and a uniform particle size distribution in the field of pharmaceutical preparation. Kotyla et al. [2] were prepared by high-pressure micro-jet method and the traditional method of the same proportion of δ-vitamin E cream. The average particle size of nanoemulsion prepared by high pressure micro jet method is 65 nm, and the average particle size of microemulsion prepared by traditional method is 2 788 nm. Zhao et al. [3] prepared the injection of fat emulsion by combining the piston-slit homogenization method with the high-pressure micro-jet, and studied the factors influencing the particle size and particle size distribution. Barnadas-Rodriguez et al. [4] prepared phospholipid liposomes, Saheki et al. [5] prepared egg yolk and soybean oil mixed lipid nano-dispersion, Chen et al. [6] prepared coenzyme Q10 nano lipid carrier, respectively, the use of high pressure micro jet Technology under different pressures and different processing times to get a different micro-nano particle size and particle size distribution. LI Guangji [7] used the micro-jet technology combined with the ultra-high pressure technology and the impinging stream technology, proposed a new method of emulsification to overcome the
complexity of the traditional mechanical method, low energy utilization, insufficient shear force, difficult control of the working process and other shortcomings.

Domestic and foreign scholars have applied high-pressure micro-jet technology to many fields, and have studied the micro-jet process. However, there are still some shortcomings in the preparation of emulsion of micro-jet, mainly on the impact of homogeneous performance of micro-jet parameters, improve the efficiency of micro-jet homogenizer, etc. also need to further study.

**Experimental Materials and Methods**

Taking phospholipid b g, glycerol c g, in the mortar is grinded uniformity. Distilled water d ml, first putting a small amount of distilled water to the mortar is grinded, and then put in all the remaining distilled water to dilute. Taking soybean oil a g, with a mixture of mortar in high speed shearing machine, is sheared 15×2 times with 10000r/min, and then initial emulsion is obtained. According to the different formulations, a, b, c, d take different values.

**Effect of Homogenization Pressure**

(1) According to the formula: soybean oil 5g, phospholipid 1g, glycerin 1.5g, and water 50ml preparation of 5 initial emulsion.

(2) The prepared initial emulsion is putted in the high pressure homogenizer, and the working pressure of the homogenizer is adjusted. The 5 initial emulsions are operated in accordance with table 1. The emulsion is cooled to room temperature after each homogenization, and then the next homogeneous.

<table>
<thead>
<tr>
<th>Initial Emulsion</th>
<th>Pressure(MPa)</th>
<th>Initial Temperature(℃)</th>
<th>Homogenization times</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>30</td>
<td>20</td>
<td>8</td>
</tr>
<tr>
<td>2</td>
<td>40</td>
<td>20</td>
<td>8</td>
</tr>
<tr>
<td>3</td>
<td>50</td>
<td>20</td>
<td>8</td>
</tr>
<tr>
<td>4</td>
<td>60</td>
<td>20</td>
<td>8</td>
</tr>
<tr>
<td>5</td>
<td>70</td>
<td>20</td>
<td>8</td>
</tr>
</tbody>
</table>

(3) The particle size distribution of the emulsion was measured by Malvin laser particle sizer.

**Effect of Initial Temperature**

(1) According to the formula: soybean oil 5g, phospholipid 1g, glycerin 1.5g, and water 50ml preparation of 4 initial emulsion.

(2) The 4 initial emulsions are operated in accordance with table 2. The initial emulsion is heated in the beaker to the appropriate temperature and then placed in high pressure homogenizer. The emulsion is cooled to the appropriate temperature after each homogenization, and then the next homogeneous.

<table>
<thead>
<tr>
<th>Initial Emulsion</th>
<th>Pressure(MPa)</th>
<th>Initial Temperature(℃)</th>
<th>Homogenization times</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>40</td>
<td>20</td>
<td>8</td>
</tr>
<tr>
<td>2</td>
<td>40</td>
<td>30</td>
<td>8</td>
</tr>
<tr>
<td>3</td>
<td>40</td>
<td>40</td>
<td>8</td>
</tr>
<tr>
<td>4</td>
<td>40</td>
<td>50</td>
<td>8</td>
</tr>
</tbody>
</table>

(3) The particle size distribution of the emulsion was measured by Malvin laser particle sizer.

**Effect of Oil/Water Ratio**

(1) Initial emulsion is prepared according to the formulation of Table 3

(2) The 6 initial emulsions are placed in the high pressure homogenizer respectively, and the homogeneous pressure is adjusted to 50MPa, which is homogenized for 8 times. The emulsion is cooled to room temperature after each homogenization, and then the next homogeneous.
The particle size distribution of the emulsion was measured by Malvin laser particle sizer.

Table 3. Experimental Parameters of Oil /Water Ratio.

<table>
<thead>
<tr>
<th>Initial Emulsion</th>
<th>O/W</th>
<th>Oil (g)</th>
<th>Phospholipid (g)</th>
<th>Glycerol (g)</th>
<th>Water (ml)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1:6</td>
<td>5</td>
<td>3.5</td>
<td>1.75</td>
<td>30</td>
</tr>
<tr>
<td>2</td>
<td>1:8</td>
<td>3.75</td>
<td>3.375</td>
<td>1.6875</td>
<td>30</td>
</tr>
<tr>
<td>3</td>
<td>1:10</td>
<td>3</td>
<td>3.3</td>
<td>1.65</td>
<td>30</td>
</tr>
<tr>
<td>4</td>
<td>1:12</td>
<td>2.5</td>
<td>3.25</td>
<td>1.625</td>
<td>30</td>
</tr>
<tr>
<td>5</td>
<td>1:15</td>
<td>2</td>
<td>3.2</td>
<td>1.6</td>
<td>30</td>
</tr>
<tr>
<td>6</td>
<td>1:20</td>
<td>1.5</td>
<td>3.15</td>
<td>1.575</td>
<td>30</td>
</tr>
</tbody>
</table>

Results and Discussion

Conclusion and Analysis of Homogeneous Pressure Experiment

The greater the working pressure provided by the homogenizer, the greater the velocity of the liquid in the homogeneous chamber. High-speed fluid has a high turbulent kinetic energy, resulting in a strong impact effect. At the same time, the velocity gradient of the fluid in the near wall region is larger, and the shear effect increases. At high pressure, the transient pressure decrease and pressure increase of the fluid produce a strong hole effect.

As shown in Figure 1, with the increase of homogeneous pressure, the general trend of average particle size is decreased. It can be seen from Fig. 2 that with the increase of homogeneous pressure, the general trend of PDI is first reduced and then increased, and the inflection point appears at 40MPa. The smaller the PDI, the more uniform the particle size distribution. So with the increase of pressure, the particle size is smaller and smaller, but the particle size distribution is uneven. If the particle size distribution in the homogeneous emulsion is not uniform, because of the difference of the interfacial energy between the large droplets and the small droplets, the large droplets have the tendency to merge small droplets. Therefore, in the emulsion with the same average particle size, the emulsion of uniform particle size distribution has a better stability than that of uneven distribution. When the emulsion is prepared by adjusting the homogenization pressure, the homogenization pressure should not be too high in order to obtain a stable emulsion, in the case where the particle size meets the requirements.

Conclusion and Analysis of Initial Temperature Experiment

With the increase of temperature, the viscosity of oil droplets is decreases and the surface tension is increases. When other parameters are constant, the breakup of oil droplets is affected by viscosity and surface tension. While the temperature increases, the movement of molecules within the emulsion is exacerbated, increasing the probability of oil droplets polymerization. As shown in Fig. 3, with the increase of initial temperature, the general trend of average particle size is increased, but a gentle phase occurs at 30-40°C. With the increase of temperature, the emulsion viscosity decreased, resulting in the increase of diffusion coefficient of oil droplets, so the homogeneous oil
drops after the collision probability increases, leading to drop polymerization again, thereby increasing the homogeneous particle size. At the same time, due to the decrease of viscosity, oil droplets are more vulnerable to external shocks. At 30-40°C, the breakup and coincidence of the droplets in the homogeneous cavity are in equilibrium, so the curve is relatively gentle. As can be seen from Fig. 4, the particle size distribution PDI at 30°C is high, indicating that the particle size distribution at a temperature of 30°C is extremely uneven, affecting the stability of the emulsion. Therefore, when the emulsion is prepared by homogeneous mechanism, the emulsion temperature is controlled at room temperature under the condition that other parameters are constant.

![Figure 3. Particle Size Varies with Temperature.](image)

![Figure 4. Particle Size Distribution PDI.](image)

**Conclusion and Analysis of Oil /Water Ratio Experiment**

The strength of the interfacial film between oil and water decreases with the increase of oil / water ratio, which can easily lead to the formation of larger droplets\(^8\). At the same time, with the increase of oil/water ratio, the space between oil droplets is reduced and the interaction between oil droplets is enhanced. The probability of collision and merging between oil droplets is increased. As shown in Fig 5, with the decrease of oil/water ratio, that is, with the decrease of the volume fraction of the oil phase, the average size of the particles is decreased. When the oil/water ratio is reduced to 1: 12-1: 20, the curve tends to be gentle. This is because that the volume fraction of the oil phase is much smaller than that of the water phase. The volume fraction of the oil phase in 1: 12-1: 20 changes to homogenization. The impact is not the main factor. The effect of volume fraction change of 1: 12-1: 20 in oil on the homogeneous effect is not a major factor. When the oil/water ratio continues to decrease, the emulsion is extremely unstable and the emulsion cannot be formulated. As can be seen from Fig.6, with the smaller oil/water ratio, the particle size distribution PDI increases and then decreases. And the inflection point occurs at 1:8 and 1:12, and PDI increased significantly after 1:12. Therefore, the oil/water ratio control in 1:12 can improve the homogenization effect, in the case of other conditions unchanged.

![Figure 5. Particle size varies with O/W.](image)

![Figure 6. Particle size distribution PDI.](image)

**Conclusion**

In this paper, the effects of homogenization pressure, initial temperature and oil/water ratio on
homogenization were studied by experiment. Analysis of particle size changes, the following conclusions: with the increase of homogeneous pressure, homogeneous particle size decreases; with the increase of initial temperature, homogeneous particle size increase; with the decreases of oil/water ratio, homogeneous particle size decreases.

In the field of pharmaceuticals, high pressure micro-jet technology still has many problems, the existing research is not enough, also need a lot of experimental research. The effect of the interaction between the emulsifier content and the initial temperature on homogeneous properties is also required to be studied in depth.

References