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The Influence of Pan Evaporation Conditions on NaCl Crystallization

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1 Introduction

Crystallization is one of important unit operations in the chemical production process, which requires not only the crystal product with high purity and yield, but also the available particle size of the crystal products to ensure product quality. Crystallization process is affected by many factors, including supersaturation, crystallization temperature, suspension density, and crystallization time, collision energy, stirring rate, seed and impurity. The product should be of uniform crystal size, no agglomeration, appropriate hardness and high transparency. Shape, crystal size and distribution of products all affect the select of crystallizer.Salt refining is one of the methods of manufacturing salt crystal products by recrystallization. Some single crystal salt, such as vacuum salt, could be manufactured through the control of the conditions of the salt recrystallization. The flaky or the dendritic salt crystals could also be manufactured by regulating the recrystallization conditions. The conditions of the refining salt process have an important impact on the quality of the salt crystal.In this study, the effect of evaporation rate, retention time and stirrer on NaCl properties was investigated in the recrystallization process. Then, the lamellar structure and particle size of product were also studied in recrystallization process.

2 Experimental results and discussion

Boiling evaporation and non-boiling evaporation at specific evaporation rate were used in the experiment. The effect of evaporation rate on the salt product bulk density and water content was shown in Table 1 (residence time of the pan evaporation process was 7h without stirring). The crystal habit and size distribution of the refined salt products were obtained at different evaporation rates.

As shown in Figure 1, the salt crystal produced by boiling evaporation was small and cubic. While piece shape salt crystal was obtained at the condition of boiling till salting-out (floating flowers) following rate-controlled evaporation. Moreover, the piece became thicker with the decrease of evaporation rate.

Table 1 The effect of pan evaporation rate on the characteristics of refined salt

Average evaporation rate ($mm \cdot h^{-1}$)	Bulk density ($g \cdot ml^{-1}$)	Refined salt water content (%)
28	0.881	9.636
18	0.679	8.640
14	0.653	8.998
10	0.625	8.936
6	0.601	8.854

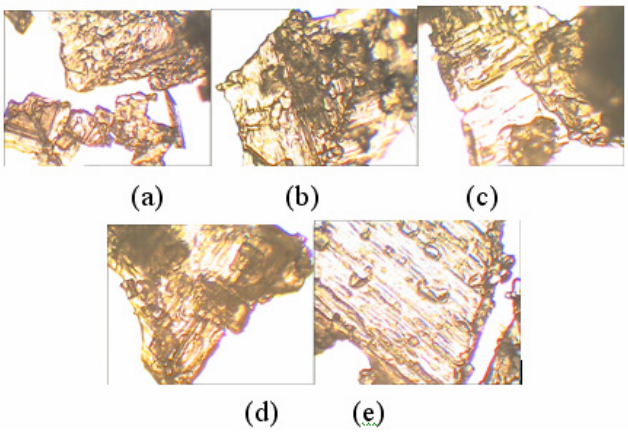

Fig. 1. The effect of evaporation rate on the crystal habit of refined salt.Average evaporation rate is (a) 28 mm/h (b) 18 mm/h (c) 14 mm/h (d) 10 mm/h (e) 6 mm/h.

The average particle size of salt produced by boiling evaporation was only 497.7μm. In the rate-controlled evaporation crystallization, crystal size increased with evaporation rate decreased. The crystal size was higher than 800μm when the average rate of evaporation was equal or less than 18mm/h, however, the average size of refined salt was 800μm when the evaporation rate was 14mm/h.

The influence of residence time on the refined salt deposit density and water content influence under condition of 14 mm/h evaporation rate and stirring twice

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Table 2 The influence of risidence time on the characteristics of refinded salt

Retention time (h)	bulk density (g·ml^{-1})	Refined salt water content(%)
7	0.679	8.998
8	0.687	9.031
9	0.705	8.967
10	0.749	8.930

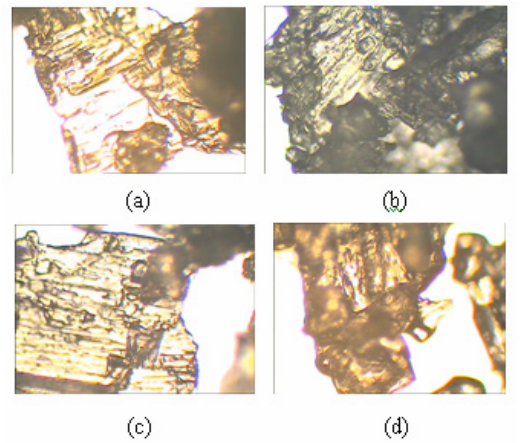

Fig. 2. The effect of residence time on the crystal habit of refined salt. Retention time (a) 7h (b) 8 h (c) 9 h (d) 10 h.

per hour (each stirring time was 3 min) was listed on Table 2. The crystal habit and particle size distribution of refined salt crystal were obtained in different residence time as shown in Figure 3and Figure 4.

As shown in Figure 2, the plat crystal became thicker and the mechanical stability of crystal was improved with longer retention time. Moreover, the average particle size increased from 812.8 μm to 827 μm with the retention time increased from 7h to10h.

In Table 3, the influence of stirrer frequency (each stirring time was 3 min) on salt deposit density and water content was listed under the condition of evaporation rate 14 mm/h and residence time 10h. The crystal habit and particle size distribution of refined salt crystal products under different stirrer frequency was detected by SEM and laser diffraction particle size analyzer as shown in Figure 3.

The square and small refined salt crystal was prepared under relative high stirrer frequency. However, thick plat refined salt crystal was prepared without using the stirrer. The average size of refined salt decreased with the increase of the stirrer frequency as shown in Figure 7. The average particle size of salt crystals changed from the 416.7 μm to 533.6 μm when the stirrer frequency of the refined salt crystallization process changed from 4 times/h to 1 time/h, while, the average particle size of refined salt was 820.7 μm without stirring

Table 3 The effect of stirrer frequency on the characteristics of refined salt

Stirrer frequency(h)	Bulk density(g·ml^{-1})	Refined salt water content(%)
4	0.882	9.258
3	0.877	9.239
2	0.864	9.201
1	0.853	9.197
0	0.749	8.930

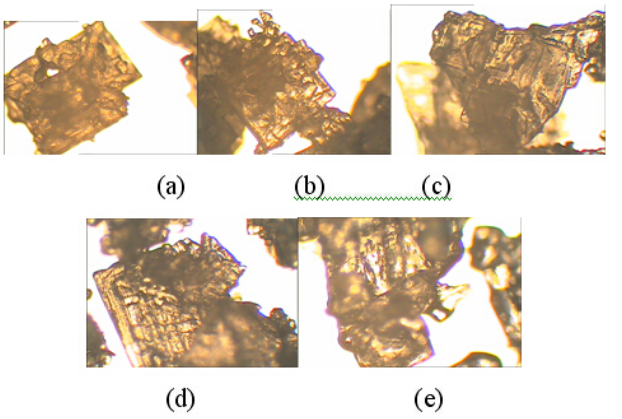

Fig. 3. The effect of stirrer frequency on the crystal habit of refined salt. Stirrer frequency (a) 4 times/h (b) 3times/h (c) 2times/h (d) 1 time/h (e) Without stirrer.

3 Conclusion

In this study, the effect of evaporation rate, retention time and stirrer on NaCl properties were investigated in the recrystallization process. Then, the lamellar structure and particle size of product were also studied in recrystallization process. The crystal product was in an uniform particle size (average size of refined salt was 800μm) under the condition of 14mm/h evaporation rate. Higher crystal nucleus rate was obtained in boiling evaporation process, and most NaCl was with cube crystallization. The retention time had a significant effect on the thickness of NaCl plat crystallization, and the plat crystal production was thicker with longer retention time. The average size of NaCl crystallization was smaller with higher frequency of stirrer, and cube crystal productions were available with higher frequency of stirrer. When retention time was 10h without stirrer, the average size of NaCl production was 827.0 μm，the stack density was 0.749 g/L, and the size distribution was 500 μm~1500 μm.

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