

ZHANG Lei, LIU Shanhe, XIANG Jun, CHENG Penggao, TANG Na and WANG Xuekui, 2014. The Effect of Additives on Crystal Size of Sodium Sulphate. *Acta Geologica Sinica* (English Edition), 88(supp. 1): 401-403.

The Effect of Additives on Crystal Size of Sodium Sulphate

ZHANG Lei¹, LIU Shanhe¹, XIANG Jun¹, CHENG Penggao¹, TANG Na^{1,2} and WANG Xuekui¹

¹ College of Marine Science and Engineering, Tianjin University of Science & Technology, Tianjin 300457, China

² Tianjin Key Laboratory of Marine Resources and Chemistry, Tianjin 300457, P.R. China

1 Introduction

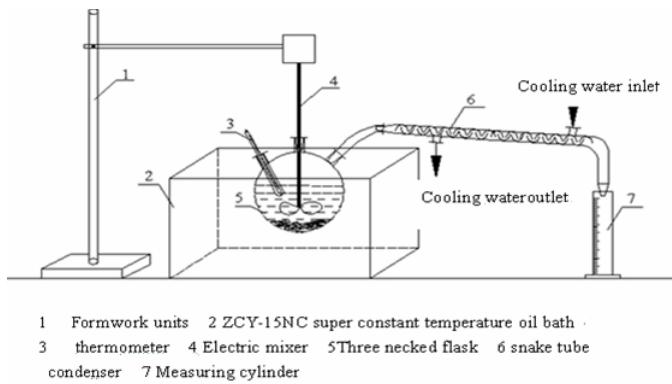
Anhydrous sodium, mainly produced in the United States, Canada, Japan, is indispensable commodities and raw materials in daily life and industry. In recent years, anhydrous sodium sulfate of general size was much oversupplied. However particles anhydrous sodium sulphate of large size is not adequate to the demand for its high purity, better quality, beautiful appearance, separating easily from mother liquor, easily electrolysis, lower solubility, convenient washing etc. As a result, the large particles anhydrous sodium sulfate process conditions should be investigate for commercial purpose.

Particle size and particle size distribution of crystal related was related with nucleation rate, the crystal growth rate and crystal residence time in the crystallizer. Trace amounts of surface active agent will lower surface tension and increasing the effect of the surfactant solution, which change crystallization rate of the solution, thereby controlling the particle size of the crystal product. The presence of additives may promote nucleation; it can slow down the rate of formation of crystal nuclei, or inhibit the growth of nuclei. Additives had different effects on the crystallization process in different systems. In this work, the effects of additives on the anhydrous sodium sulfate crystal size were explored with sodium dodecyl benzene sulfonate and potassium dichromate as additives.

2 Experimental Part

Experimental apparatus and procedures

Experimental apparatus was shown in Figure 1. Anhydrous sodium sulfate solution was formulated in three flasks under certain temperature by adding the



1 Formwork units 2 ZCY-15NC super constant temperature oil bath
3 thermometer 4 Electric mixer 5 Three necked flask 6 snake tube condenser 7 Measuring cylinder

Fig. 1. Experimental device.

different dose of additives and sodium sulfate crystal and conducting constant temperature evaporation.

The influence of additives and crystallization conditions on anhydrous sodium sulfate crystal size of solid sodium crystal was obtained with different crystallization conditions after been vacuum filtrated, dry and sized.

Table 1 Optimum condition determined by orthogonal experiment.

No.	A stirring speed (rad/min)	B seed mass (g)	C residence time (min)	D Oversize fraction over 80 mesh (%)
1	180	1	30	0.45
2	180	2	40	0.6571
3	180	3	50	0.4827
4	240	1	40	0.6443
5	240	2	50	0.5779
6	240	3	30	0.6248
7	300	1	50	0.4989
8	300	2	30	0.4994
9	300	3	40	0.4063
K ₁	1.590	1.593	1.574	1.434
K ₂	1.847	1.734	1.708	1.781
K ₃	1.405	1.514	1.560	1.626
L ₁	0.53	0.531	0.525	0.478
L ₂	0.616	0.578	0.569	0.594
L ₃	0.468	0.505	0.520	0.542
R	0.148	0.073	0.049	0.116
Factor main→auxiliary		A→D→C		
Optimum		A ₂ D ₂ C ₂		

* Corresponding author. E-mail: tjtangna@tust.edu.cn

3 results and discussion

As shown in Table 1, The optimum condition of Sodium sulfate crystallization was 240rad/min stirring speed, 40min residence time 40g seeded. And the grain size of anhydrous sodium sulfate greatest impact is the stirring speed, followed by retention time, and finally the amount of seed added.

Size and growth rate of the seed crystal particles interacted with each other. It showed that the crystal growth line rate increases as crystal size decreases. Adding a certain size and number seed crystal with gentle stirring to homogeneously suspend in solution can accelerate the rate of nucleation. In this study, the added sodium seed are 100-120 mesh (0.15 cm-0.125cm) particles unless other noted.

3.1 Effect of additives on the grain size of the sodium

The following experiments take sodium dodecyl benzene sulfonate and potassium dichromate as additives with OP emulsifiers and isopropanol as auxiliary agents. Effect of Additives on sodium crystal size was shown in Figure 2 when the experimental condition was 240rad/min stirring speed, 40min dwelling time, 40g seeded amount, OP emulsifier concentration of 25ppm, isopropyl alcohol concentration of 10ppm.

The additives of Sodium dodecyl benzene sulfonate influence more apparently than dichromate on sodium crystal size, when the concentration of sodium dodecyl benzene sulfonate was 30ppm, the amount of oversize fraction over 80 mesh of anhydrous sodium up to the most which was 67.18%, followed by a downward trend; With the increasing of the amount of potassium dichromate added, the crystal grain size also increased.

Analysis of Influencing Factors designed showed that Stirring rate have a significant impact on sodium crystal size. Under the experimental conditions in the

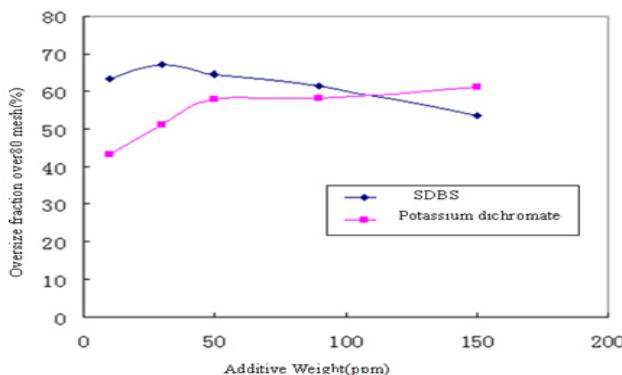


Fig. 2. Effect of Additives on sodium crystal size.

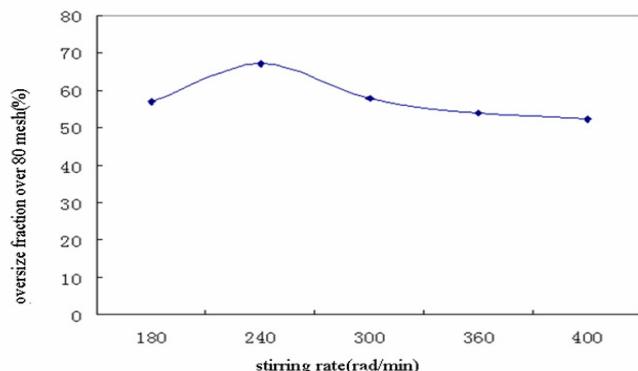


Fig. 3. Effect of stirring rate on sulfate crystal size.

concentration of sodium dodecyl benzene sulfonate 30ppm, 40min residence time, 40g seeded amount , OP emulsifier concentration of 25ppm, 10ppm concentration of isopropyl alcohol, the influence of stirring rate of sulfate crystal size was studied. The results shown in Figure3 stirring rate on have a significant impact on crystal nucleation rate, crystalline shape, and size etc

As shown in Figure 3, the sodium crystal product particle size is small when the stirring rate is large, whereas the larger particle size. The main source of secondary nucleation result of the contacting of mixing pulp and grain pulp, collision of crystals and crystal, the higher Stirring speed, the more energy and chances provided access to the collision, so as to nucleation rate and crystals size. If the stirring rate is too small, the mixed slurry crystal in the mold is not uniform leading to the existence of partly high concentrations, which accelerated nucleation rate and crystal became smaller. Experimental results show that oversize frictions over 80 mesh obtained up to the most of 67.18%.when the stirring rate is 240rad/min.

3.2 Effect of residence time on crystal size of sodium sulphate

The residence time of the effect on the crystal size of

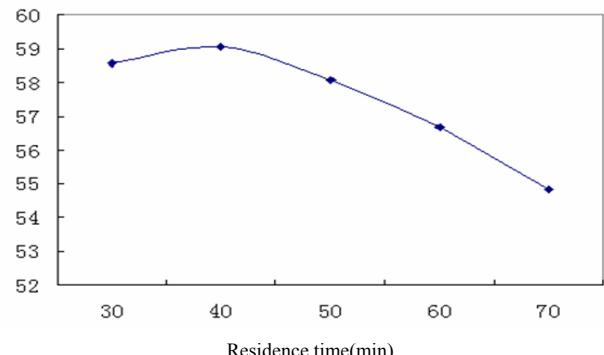


Fig. 4. residence time on sulfate crystal size.

sodium sulphate was studied when the experimental conditions was 30ppm SDBS added, 240rad/min stirring speed, 40g seeded quantity, 25ppm OP emulsifier concentration, 10ppm isopropanol added, as shown in Figure 4.

Figure 4 shows that the residence time of sodium crystal have a significant effect on product size. The amount of oversize frictions over 80-mesh was up to the most of 59.07% when the residence time was 40min in the study, However sodium crystal size may decrease significantly when residence time over 40min.

4 Conclusions

In this work, sodium dodecyl benzene sulfonate was regarded as an additive, the effects of stirring rate and residence time on sulfate crystal size was studied. The secondary nucleation rate increased and the sodium crystal size reduced with higher speed stirring. When stirring rate was 240rad/min, the sodium granularity was much larger

and the 67.18% anhydrous sodium sulfate were o 80 mesh. The residence time of sulfate crystals impact product size significantly. The sodium crystal granularity decreased as the residence time increased. 59.07% sulfate sodium crystal was above 80 mesh under 40 min residence time.

Key words: Additive, Sodium sulphate ; Crystallization, Crystal size

Acknowledgements

The authors thank for the financial support of National Nature Science Foundation (21376178), TIDA giant growth plan (2011-XJR13020), Tianjin Science and technology support program (12ZCDZSF06900), Tianjin University of Science and Technology fund for scientific research (20120119), Tianjin education commission program (20130509) and Research fund for the doctoral program of higher education of China (20131208120001).