New type crystallizer for particle size control of sodium chloride

Fumihiko Yamada and Takaomi Tsukamoto
DIASALT Co., Ltd. Sakito Manufacturing Factory , Sakito-cho , Nagasaki 857-3101 , Japan

In Japan, due to high humidity during the rainy season, salt products tend to cake due to deliquescence of microcrystals in the salts. In order to prevent the caking of crude salt by eliminating microcrystals, we performed laboratory-scale slurry classification using a crystallizer with an incorporated classifier. In the classification test, a liquid was introduced at the bottom of the crystallizer, the liquid was drawn upward and the slurry was classified. By these procedures, the following results were obtained. 1) The amount of microcrystals in salt products was reduced to one-half that found before classification ; 2) the mean particle size of crystals tended to increase due to the increase in the effective contact time of the microcrystal-containing slurry which was returned to the crystallizer after classification and the accompanying suppression of the generation of primary nuclei.

1 . INTRODUCTION

In Japan, evaporative crystallizers are used in the manufacture of salt. However, the crystal sizes distribution (CSD) obtained by such equipment exhibits a particular pattern that depends on the characteristics of the crystallizer and the operating conditions. Vacuum salt (medium particle size salt) produced with vacuum equipment has a wide distribution of particle sizes and contains microcrystals that affect the caking of salt products. Figure 1 shows a schematic diagram of a crystallizer is an "Up-flow" type, which features a long contact time for the crystals in the supersaturated zone, and the ability to increase the crystal growth rate. The mean particle size of the product is approximately 800 microns. The grown crystals settle down into the salt leg and are collected as product. It is suspected that microcrystals are mixed with the product during this process, creating a broader particle size distribution. To alleviate this mixing problem, we constructed a crystallizer into which a classification system is integrated that can classify microcrystals in the salt leg and return them to the crystallizer. We also studied the behavior of microcrystals that were returned to the crystallizer after classification.

2. TEST AND RESULTS

2.1 Laboratory test

2.1.1 Apparatus

Figure 2 shows the test equipment which is modeled on the lower part of an actual crystallizer with a classification system at 1/5 the actual size. To observe the flow pattern of the crystals, the salt leg, made of an acrylic pipe, was constructed at the outlet of the recycle loop of the crystallizer. In addition, a line was installed at the lower end of the salt leg, through which the classification liquid flows upward.

2.1.2 Experimental methods

We filled the main body of the test equipment with salt crystals (mean particle size : 790 microns) up to the outlet, and then introduced the classification liquid (a saturated saline solution) to the lower part of the salt leg to observe the flow behavior of the salt crystals. Table 1 lists the test conditions.

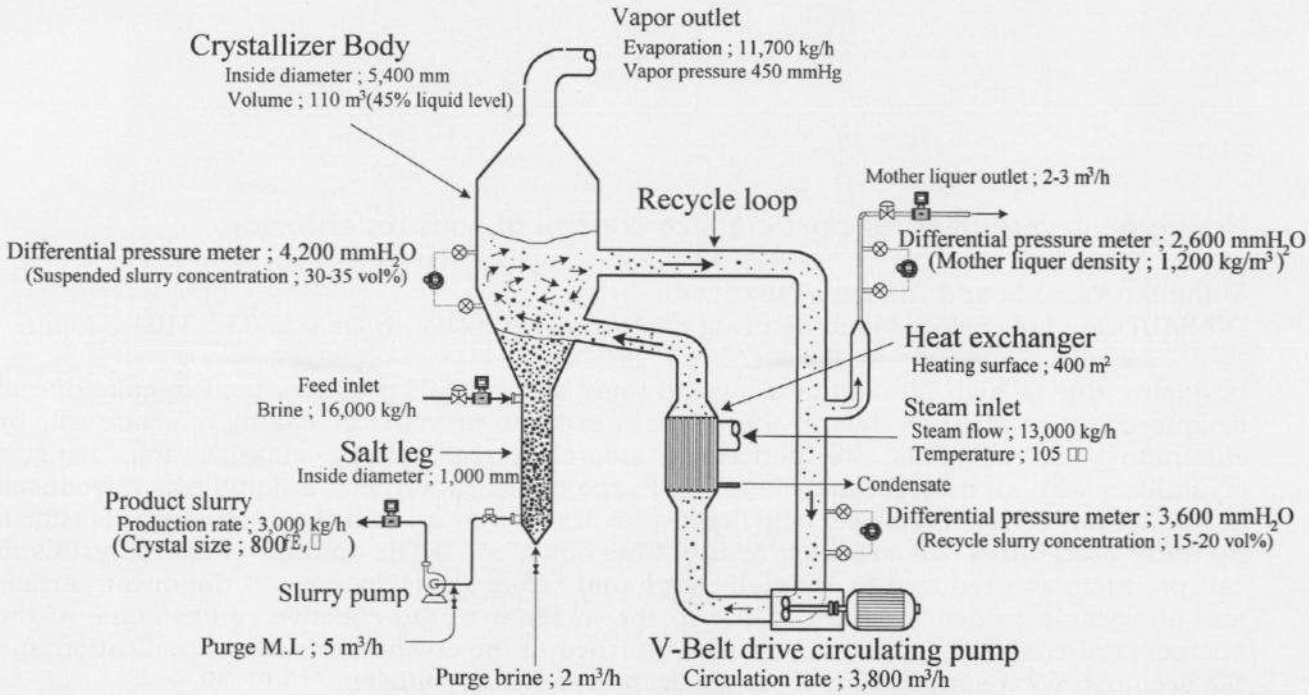

Fig.1 Schematic view of evaporative crystallizer

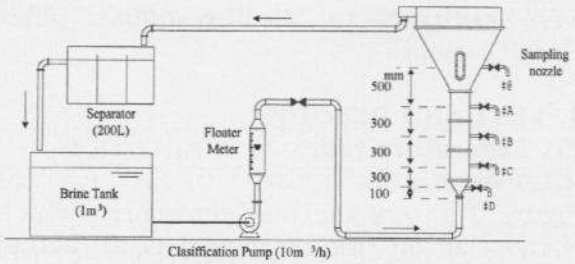

Fig.2 Classification test equipment

Table 1. Classification test conditions

RUN No.	Up-flow rate (cm/s)
1	2
2	4
3	6
4	8

2.1.3 Results

Figure 3 shows results observed at various flow rates. The microcrystals are lifted to a point close to the outlet of the recycle loop where there is a supersaturation, and then grow further. We found that for this phenomenon an up-flow rate of 6 cm/sec was required; determining this value was one of the purposes of this study. Furthermore, we studied the effect of the up-flow rate on crystal classification. Figure 4 shows the relationship between the relative height of the sampling nozzle from the bottom of the top equipment, HR, and the ratio of the mean particle size of added salt crystals to the mean particle size at each sampling nozzle after classification, (Ds/Df). Table 2 shows the measurements of CSD for each run. When the normalized mean particle size Ds/Df was larger than 1, classification of the added crystals occurred. The HR value indicates the height of a sampling point, and it decreased as the position of H descended to the lower part of the salt leg. At sampling point (4) which is positioned at the same height as in the actual crystallizer, the mean particle size was always 1.06 time that of the added crystals, regardless of the up-flow rate. If we define crystals smaller than 425 microns as microcrystals, the amount of microcrystals becomes less than half that of the added crystals. Based on the above observations and classification effects, we concluded that if the flow rate in the classification line of the actual equipment

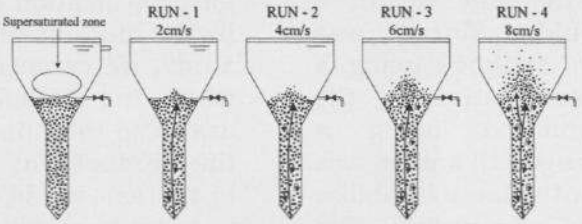

Fig.3 Observation results of flow behavior

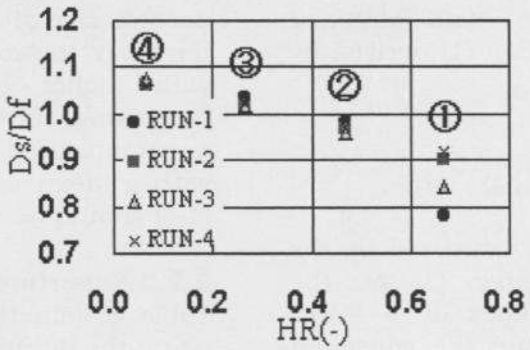

Fig.4 Correlation between the height HR and the normalized mean size
HR : Relative height of sampling nozzle
Ds (micron) : Mean particle size of added salt crystals
Df (micron) : Mean particle size at each sampling nozzle (After classification)

Table.2 Results of measured CSD and mean particle size

Sieve analysis (micron)	Added crystals (wt%)	RUN-1(2cm/s) ①	②	③	④	RUN-2(4cm/s) ①	②	③	④	RUN-3(6cm/s) ①	②	③	④	RUN-4(8cm/s) ①	②	③	④
		(wt%)				(wt%)				(wt%)				(wt%)			
75-212	0.61	7.78	0.65	0.44	0.23	1.46	0.48	0.22	0.16	2.98	0.54	0.34	0.09	0.70	8.68	0.29	0.09
212-425	3.12	16.7	4.16	2.37	1.51	7.62	4.54	2.42	1.34	12.4	4.08	2.10	1.13	6.80	3.32	1.82	1.09
425-500	3.19	7.52	3.91	2.66	1.64	5.61	4.06	2.70	1.88	7.28	4.18	2.63	1.61	5.48	3.47	2.42	1.61
500-600	10.34	15.2	11.4	8.63	7.24	14.7	12.2	9.70	7.12	16.1	12.7	9.30	7.06	14.6	11.3	9.26	6.68
600-850	44.18	35.3	43.9	41.7	40.2	44.3	45.2	44.1	41.2	41.4	46.2	44.4	40.0	45.0	45.7	44.2	40.9
850-1,000	25.35	12.6	24.2	28.4	30.9	19.8	23.6	26.7	29.6	15.1	23.2	28.6	30.8	20.3	20.3	28.1	30.8
1,000-1,180	10.79	4.12	9.87	12.9	15.1	5.94	8.29	11.9	15.0	4.01	7.92	10.7	16.1	6.32	6.32	11.8	16.0
above 1,180	**2.42**	0.79	1.86	2.83	3.29	0.65	1.70	2.25	3.72	0.64	1.14	1.87	3.23	0.87	0.87	2.02	2.88
Mean particle size (micron)	**790**	619	777	819	845	712	767	804	841	667	761	806	850	729	779	810	842

were maintained at 6 cm/sec, the mean particle size of a final product would become 1.06 times larger than that in the case of ordinary operations, and it would contain half or fewer microcrystals smaller than 425 microns.

2.2 Full-scale equipment

2.2.1 Apparatus

Figure 5 shows the classification system integrated into the actual equipment. The classification pump is capable of pumping at a rate of 200 m^3/h to generate the standard flow rate of 6 cm/sec ($170m^3/h$).

The mother liquor from the outlet was used as classification liquid. Slurry was collected by the batch method, using a timer. The slurry concentration in the crystallizer was monitored using a differential pressure gauge (1) and it was maintained at a constant value to stabilize the mean particle size of crystals. The slurry concentration, measured in terms of the differential pressure, was obtained using the following equation (1) derived by Hasegawa[1].

$$(1-\varepsilon) = (\rho_s - \rho_m) / (\rho_c - \rho_m)$$
$$= (\Delta H / H - \rho_m) / (\rho_c - \rho_m) \quad (1)$$

Where ρ_m is the density measured by the differential pressure gauge (3) at the mother liquor outlet. Values of ρ_c =2.15 and H=2,850 were set and the collection time was controlled so that the value of (1-ε), set by the computer, became constant. The timer for salt collection was set at 20 sec for sample removal with a waiting time for classification of approximately 100 sec. In the classification system in the current study, we constructed a control system in which the classification mother liquid was drawn to the upper part of the salt leg and the product was removed after the slurry in the salt leg had precipitated completely, in order to stabilize the collection of slurry. In the case of the production of a mean particle size of 800 microns, there was the tendency toward an increased crystal size with higher (1-ε) which leads to an increasing contact time for slurry in the supersaturated zone. To obtain a product with a mean particle size of 800 microns, (1-ε) should be set between 0.35 and 0.40.

2.2.2 Experimental

Table 3 lists the experimental conditions using the actual crystallizer. We measured the variation in mean particle size before and after classification by changing the

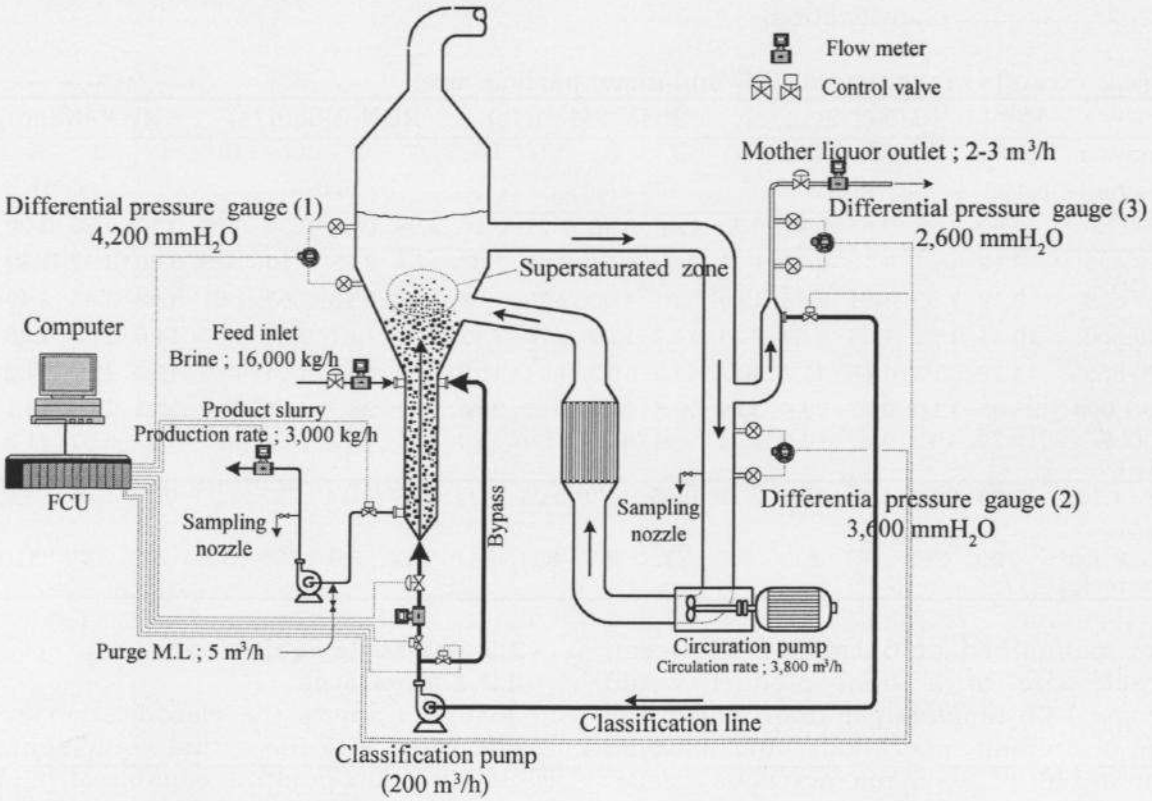

Fig.5 Schema of the classification line and the control system

The amount of classification liquid. Samples were collected from the salt leg at the product outlet and from the recycle loop at intervals of 8 hours, and mean particle sizes were measured.

Table 3. Experimental methods by full-scale equipment

RUN No.	Clqssification rate (m3/h)	Upwards rate (m/s)
1'	0	-
2'	100	3.5
3'	150	5.4
4'	200	7.1

2.2.3 Results

Figure 6 shows the changes of mean particle size in the salt leg and the recycle loop. Figure 7 shows changes of differential pressure values in the crystallizer and the recycle loop. There was a tendency for the mean particle size to increase in the salt leg and to decrease in the recycle loop soon after the classification process started. This indicates that slurry is classified in the salt leg. After the start of classification, slurry that accumulated in the upper region of the salt leg was drawn upward into the recycle loop the; differential pressure at the crystallizer decreased and the differential pressure in the recycle loop tended to increase, as reflected by variations in the differential pressure value. These changes also tend to be crystal size in RUNs 2,3 and 4 was 1.12 times that obtained in RUN-1. The amount

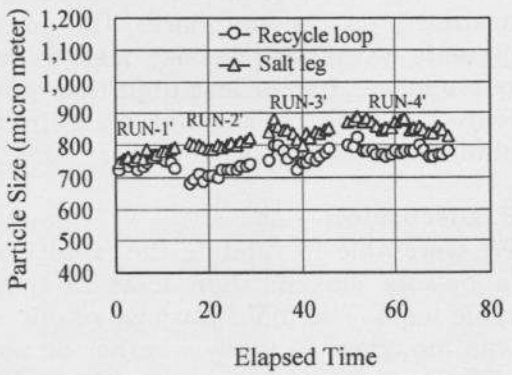

Fig.6 Changes of mean particle size

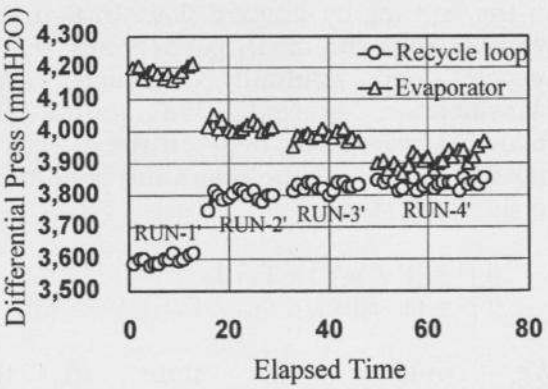

Fig.7 Changes of differential pressure

Table 4. Results of measured CSD and mean particle size

Sieve analysis (micron)		RUN-1'		RUN-2'		RUN-3'		RUN-4'	
		Recycle loop	Salt leg	Recycle loop	Salt leg	Recycle loop	Salt leg	Recycle loop	Salt leg
212-425	(wt%)	1.12	0.65	1.04	0.38	0.94	0.55	0.78	0.51
425-500	(wt%)	3.78	2.12	4.11	1.11	2.98	0.84	2.93	1.07
500-600	(wt%)	16.56	13.86	21.39	9.33	16.92	7.05	16.04	6.34
600-850	(wt%)	51.50	51.63	53.52	51.58	46.99	40.92	47.07	38.78
850-1,000	(wt%)	18.18	12.09	15.53	26.52	23.29	33.80	22.64	31.94
1,000-1,180	(wt%)	8.47	10.23	4.01	10.32	7.94	14.49	9.33	18.22
Above 1,180	(wt%)	0.40	0.42	0.39	0.75	0.93	2.35	1.20	3.14
Mean particle size	(micron)	747	764	703	797	755	845	763	857

clearer at high classification liquid flow rate. Table 4 shows the measured crystal size for each run. RUN-1, which did not involve the classification process, is assumed as the standard. The mean of crystals with particle size less then 425 microns was half or less than that found in RUN-1. These results are close to those obtained in laboratory tests.

3. Discussion

We were able to validate the results of the laboratory classification tests in the full-scale tests. The main purpose of our study was, however, to verify whether or not the microcrystals present in the product obtained from the original crystallizer could be eliminated by force classification in the salt leg by upward flow. In figure 6, we note that the mean particle size in the recycle loop gradually increased after classification started. We found the following correlation between the growth of particle sizes and the operating conditions in the fixed stage.[2]

$$(l^*)^3 = (P / \rho_c V') / F_v' I_1 I_2 \qquad (2)$$

$$(l^*)^4 = (1 - \varepsilon)(d\, l/d\, \theta)_{av} / F_v' I_1 I_2 \qquad (3)$$

We consider the state in the supersaturated zone before and after classification in terms of the above equation. The generation of nuclei in the supersaturated zone is suppressed by the slurry lifted up during classification. Thus the value of Fv' in eqs. (2) and (3) decreases. As a results, the mean particle size of product l* increased.

$$l_p = \theta(d\, l/d\, \theta)_{av} \qquad (4)$$

Also, as is clear from eq (4), the contact time for slurry in the crystallizer θ increased after classification started. As a results, mean particle size lp increased.

4. Conclusion

In order to eliminate microcrystals and increase the mean particle size in the product, we performed laboratory tests to examine the correlation between classification effectiveness and mean particle size. The results obtained in these experiments were applied in actual scale equipment and we confirmed that relatively steady operation can be carried out at an up-flow rate 6 cm/sec in the salt leg. After classification, the mean particle size in the product increased by 100 microns and the amount of microcrystals smaller than 425 microns decreased to less than half. Furthermore, we confirmed that microcrystals lifted into the supersaturated zone are re-grown. As a results, the mean particle size is increased.

Nomenclature

$(1 - \varepsilon)$ [-]	Suspension density
ρ_s [kg/m^3]	Density of slurry in the crystallizer
ρ_m [kg/m^3]	Density of mother liquor
ρ_c [kg/m^3]	Density of crystal
ΔH [mmH_2O]	Values of differential press
H [mm]	Distance of gauges
l* [m]	Crystal size
$(d\, l/d\, \theta)_{av}$ [m/h]	Average growth rate of crystal
P [kg/h]	Production rate
V' [m^3]	Volume of a crystallizer
Fv' [m^{-3}h^{-1}]	Nucleation rate per unit crystallizer
I [-]	Crystal volume shape factor
lp [m]	Crystal size
θ [h]	Time

References

1) M.Hasegawa Bull. Soc. Sea Water Sci., Japan, 44, 56(1992)
2) Toyokura, K. and Aoyama, Y., JACE Design Manual, Crystallization