The Effect of Seeding on the Crystallization of Sodium Chloride in Vacuum Pans

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ABSTRACT

This work describes the results of tests in which salt seeds were introduced into full plant scale vacuum pans to control nucleation and growth of salt crystals. The salt seeds were produced by wet grinding in a vibrating ball mill. It is shown that seeding has a very strong effect on the particle size of the salt produced. The salt produced during seeding operations has a 25-40% smaller average crystal size than salt produced without seeding. It is also shown that, with seeding, a high suspension can be maintained in the vacuum pans while still producing salt of a relatively fine crystal size.

INTRODUCTION

In internal Calandria-type salt crystallizers, it is commonly necessary to keep sufficient salt in suspension to prevent salting of the lower ends of the tubes. Such relatively high salt suspensions tend to produce crystals of large particle size. Thus a limitation is placed on flexibility of operation with regard to the crystal size of the salt produced. In theory, providing nuclei as sites for growth will permit the growth of smaller crystals at high salt suspensions. This work was done to determine whether nuclei, or seeds, produced mechanically, could be used to control particle size.

EXPERIMENTAL

The tests described were performed on full-scale plant operating equipment. An Allis-Chalmers 15-inch vibrating ball mill charged with one half- to one-inch steel balls was used to produce salt seeds. A portion of the salt slurry from the combined vacuum pan discharge was diverted to feed the ball mill. The ball mill discharge was injected back into the circuit where desired. The point of injection was changed from time to time as will be pointed out later.

Salt samples taken in slurry form from the vacuum pans were filtered on a vacuum filter funnel, washed twice with methanol and once with carbon tetrachloride to displace all mother liquor. The samples were then air dried and worked gently to prevent agglomeration of crystals. Screen analyses were performed on the dry samples.

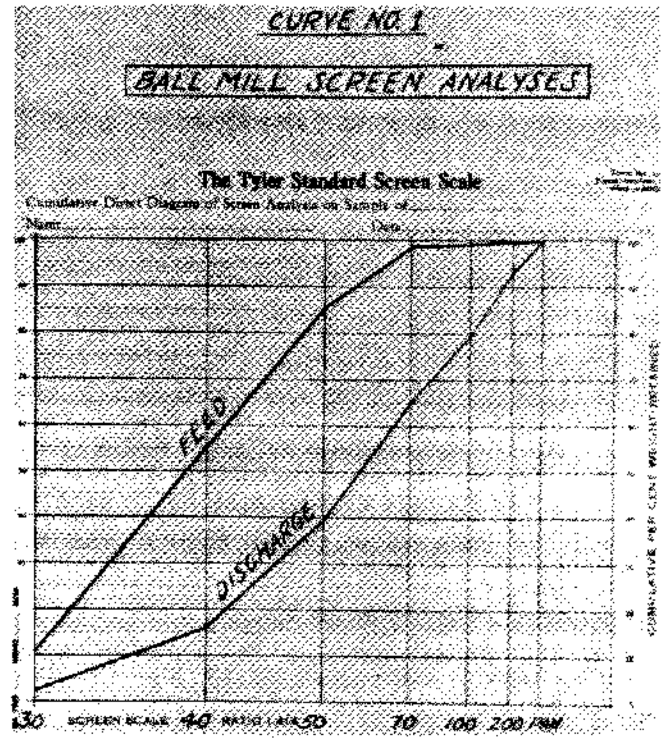
Salt suspensions were determined by drawing one liter of slurry directly from the pan. The salt was allowed to settle. The volume of settled salt is taken as a direct measure of percent salt in suspension.

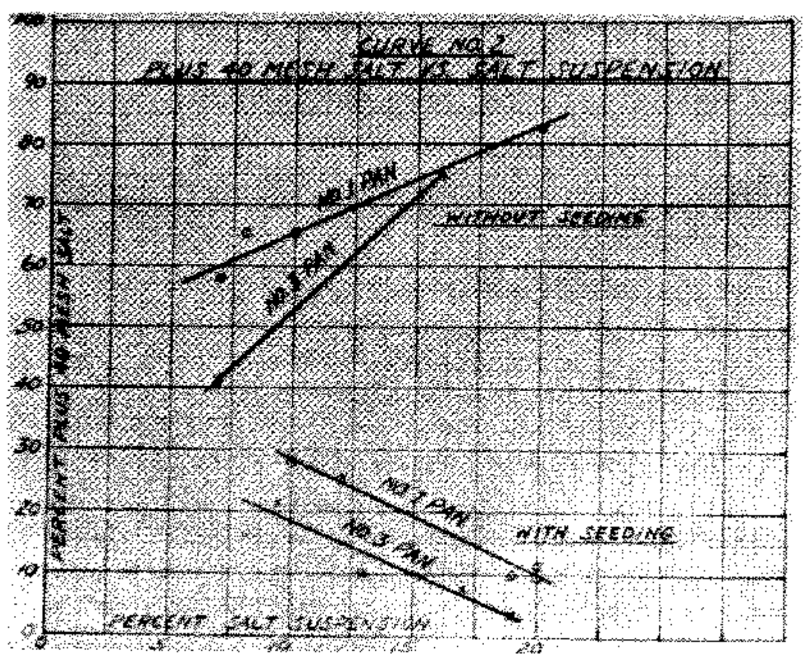
RESULTS

Curve No. 1 shows the screen analyses of the ball mill feed and discharge. The feed has almost nothing passing a 70-mesh screen while the discharge has 35 percent passing this screen.

The most effective seeds are the finer screen fractions, so probably only a portion of the ball mill discharge is providing useful nuclei.

Curve No. 2 shows the effect of operating with and without seeding. Here the percent plus 40-mesh salt is plotted as a function of salt suspension for two pans in a set of 16-foot quadruple effect pans. Rate from the ball mill was 800 pounds per hour. The No. 1 pan was seeded above the brine level and the No. 3 pan below the brine level. These data are for separate runs, seeding each pan individually.





As will be shown later, there is a direct correlation between average particle size and percent plus 40-mesh salt. The latter is used as a convenient and perhaps more meaningful way of expressing crystal size. Curve No. 2 shows the sharp increase in crystal size with increasing salt suspension when operating without seeding. With seeding, the percent plus 40-mesh salt is sharply reduced, and crystal size continues to fall with increasing salt suspension.

Curve No. 3 also shows data on 16-foot pans at 800 pounds per hour of seeds. Here the No. 1 and No. 2 pans were seeded simultaneously through the pan legs. This plot shows a sharp drop in the percent plus 40-mesh salt shortly after commencing to add seeds to the system, and after three to four hours seeding time the plus 40-mesh salt has been reduced to one-third to one-sixth of the original value.

Curve No. 4 represents data on a set of 18- to 22-foot triple effect pans fed 1,500 pounds of seeds per hour. Seeds were injected into the main brine line feeding the pans through the pan legs. These data show a rapid drop in plus 40-mesh salt shortly after starting ball mill operation, and a rapid increase in crystal size after cutting the ball mill off. The general effect is similar for all three pans, although somewhat more pronounced for the No. 1 pan.

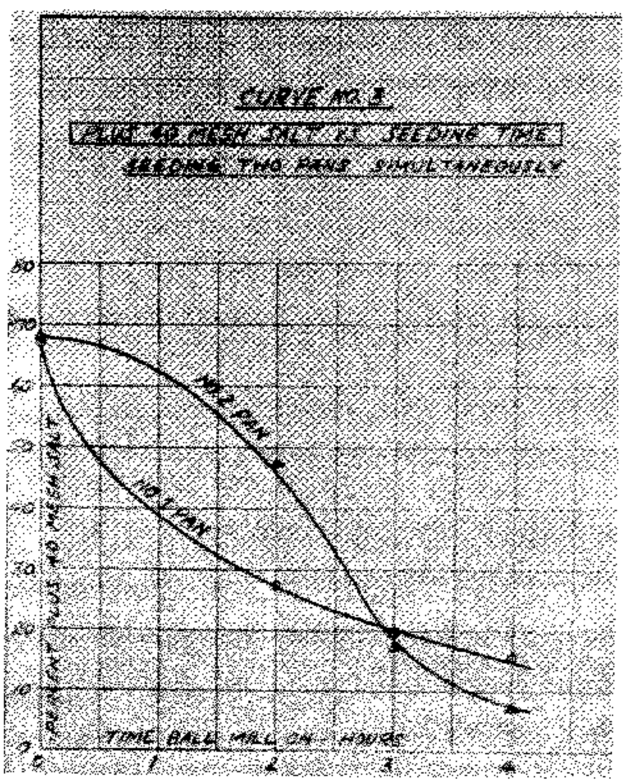
Curve No. 5 is for a 20-foot pan of a set of triples being fed 800 pounds of seeds per hour directly into the pan under the brine level. This shows the typical decrease in size with ball mill operation. On this run the salt suspension was allowed to continue to rise to very high levels, but in spite of this, the amount of plus 40-mesh salt remained very low.

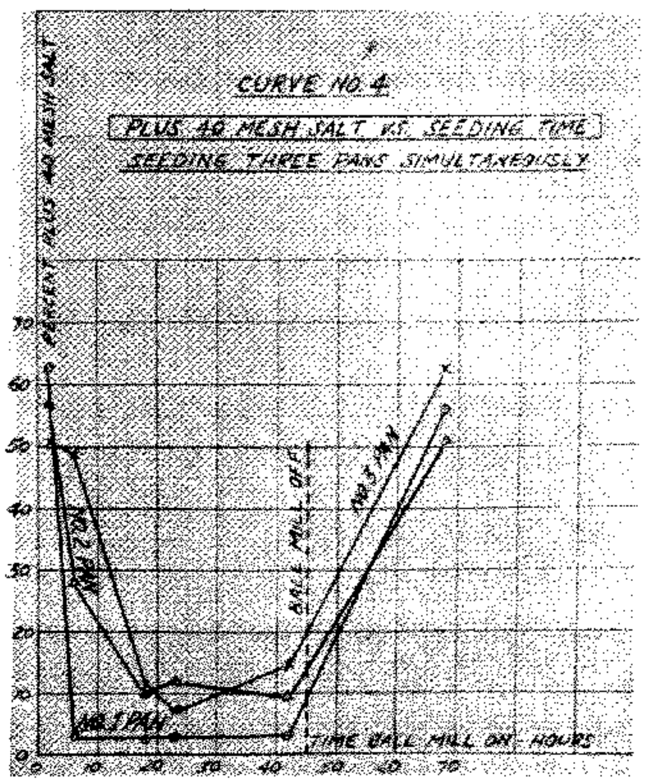
Curve No. 6 shows the correlation between plus 40-mesh salt and average particle size. This relationship demonstrates that the amount of plus 40-mesh salt is a good indicator of the crystal size.

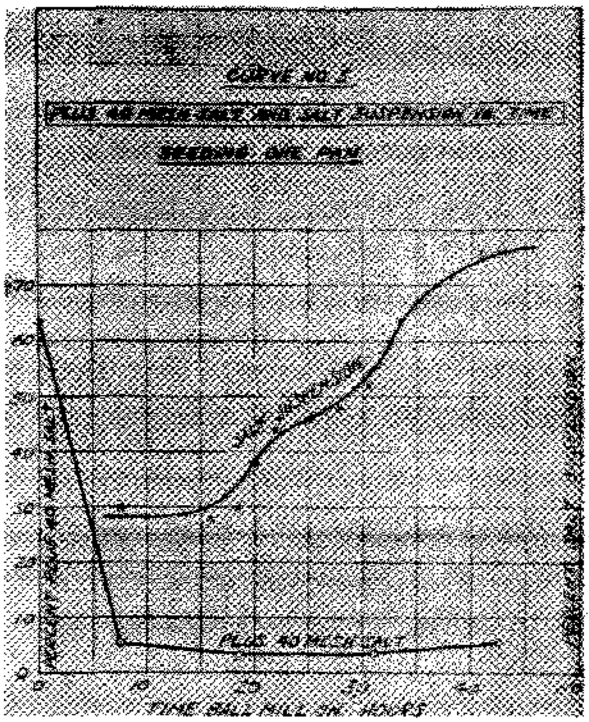
Table I summarizes some typical salt screen analyses with and without seeding. Also shown is the calculated percent reduction in average crystal size due to seeding.

DISCUSSION

The seed crystals produced in a ball mill are fragmented and irregularly shaped. However, microscopic examination shows that the salt produced is normal in appearance, and there is no







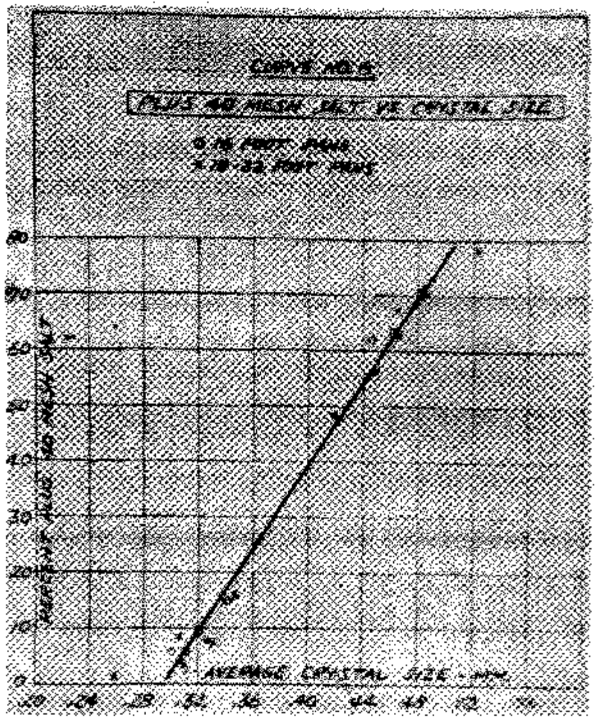


TABLE I

TYPICAL SCREEN ANALYSES -- WITH AND WITHOUT SEEDING

U.S. Std. Screens	Percent Retained						Average Crystal Size, mm.	Percent Reduction in Crystal Size
	30	40	50	70	100	Pan		
Without Seeding	28.5	52.5	17.5	3.4	0.5	0.3	0.523	
With Seeding	0.0	8.4	45.8	33.0	8.4	4.4	0.307	41
Without Seeding	3.4	44.8	37.9	11.2	2.0	0.7	0.419	
With Seeding	0.0	1.6	19.4	57.2	15.4	6.4	0.257	39
Without Seeding	2.0	49.2	44.8	3.6	0.4	0.0	0.433	
With Seeding	0.0	9.2	54.8	29.8	5.6	0.6	0.320	26
Without Seeding	7.6	56.0	30.6	5.0	0.8	0.0	0.462	
With Seeding	0.0	5.8	38.0	44.0	10.6	1.6	0.298	36

evidence of fragmented seeds remaining. This indicates that the seeds are healing and growing into typical cubic-shaped sodium chloride crystals.

The question may be raised as to whether injecting seeds at a rate of 800-1,500 pounds per hour would, in itself, contribute to reducing over-all crystal size. It has been calculated that merely mixing seeds and salt of a size normally produced without seeding would result in an average particle size reduction of less than 2%. Since the actual particle size reduction obtained is on the order of 25-40 percent, the effect of seeding on crystal size is real.

CONCLUSIONS

This work has definitely demonstrated that salt seeds produced mechanically in a wet ball mill will control nucleation and growth in internal Calandria-type pans. The point of injection of seeds into the system is not critical. Similar effects are obtained injecting seeds above or below the brine level and into the pan legs. It is shown that high salt suspensions can be maintained without growing excessively large crystals.