

## High Purity Sodium Chloride Production from Impurities Salt using Crystallization Method

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**Abstract:** Production of high purity sodium chloride from impurities salt was investigated with a suitable solvent using a 250 mL agitated batch crystallizer. Experimental mode, feed to solvent ratio, salt concentration and number of stage calculation were determined to extract pure NaCl from the saturated solution. Feed to solvent ratio at 1:3, NaCl crystal was produced around 50 wt% from impurities salt solution at 25% salt concentration. There was no significant effect on the basis of experimental modes to produce NaCl crystal. In the three-stage extraction process, this indicates that it is possible to utilize the rest of the waste solution as solvent instead of freshly added solvent in the 2nd stage for the production of NaCl. This means that the sufficient amount of solvent remained in the rest of the waste solution in this stage. In the characterization of raw salt and the product, sodium and potassium ions estimated by Flame Photometer, chloride and sulfate ions estimated by Ultraviolet-Visible Spectrophotometer, and calcium and magnesium ions estimated by Atomic Absorption Spectrophotometer. The raw salt contained 35.15% Na<sup>+</sup>, 62.02% Cl<sup>-</sup>, 0.58% Ca<sup>2+</sup>, 0.55% Mg<sup>2+</sup>, 0.12% K<sup>+</sup> and 1.58% SO<sub>4</sub><sup>2-</sup> ions. Ascribing all the chloride ions to sodium ions, the purity of NaCl in the raw salt was 97.2%. After treating the raw salt with solvent, the product contained negligible amounts of impurities ions as well as Ca<sup>2+</sup>, Mg<sup>2+</sup>, K<sup>+</sup> and SO<sub>4</sub><sup>2-</sup> ions, and the purity of NaCl in the product was 99.8%.

**Keywords:** NaCl; High purity; Salt, Solvent; Crystallizer.

**Introduction:** In Bangladesh, sodium chloride (NaCl) is produced from seawater by evaporation and vacuum evaporation processes. This NaCl is not highly pure, and it contains other chemicals including calcium, magnesium, and potassium in the forms of chlorides, sulfates, carbonates, and bromides [1-4]. It is mainly used for table salt, food preservation, animal feed, skin preservation, melting snow, etc [1,2,5].

Solid NaCl is dissolved only in polar solvents because of Na<sup>+</sup> and Cl<sup>-</sup> ions are formed. When impurities salt is dissolved in water, the sodium chloride framework is disintegrated as the Na<sup>+</sup> and Cl<sup>-</sup> ions surround the polar water due to relatively large difference in the electro-negativity. Therefore, NaCl is highly soluble in water but insoluble or only slightly soluble in most other non-polar solvents [6-16].

Analytical grade NaCl is highly pure (>99.5%) and it is widely used in the areas of chemical industries, pharmaceutical industries, analytical research and laboratory [17,18]. NaCl is used in a variety of pharmaceutical products to produce isotonic solutions. It is used in normal nasal saline sprays, intravenous lock flush solutions, and eye washes or solutions. NaCl tablets are also available to replace salt lost through excess sweating to help prevent muscle cramps. NaCl solution may also be used to dilute medications for mobilization and inhalation. Eye lotion, intravenous infusion, oral rehydration salt, mouthwash, etc. are prepared from high purity NaCl. Moreover, NaCl has a wide range of synthetic uses. It is the valuable raw material for the synthesis of many industrial chemicals. It is a raw material used for the manufacturing of caustic soda, chlorine gas, soda ash, sodium peroxide, sodium hypochlorite, and many other chemicals. NaCl is also used in the manufacturing of metallic sodium, sodium peroxide (bleaching agent used in textile industries), sodium sulfate, and many more products. Utilization of sodium chloride can be increased after getting its purity and large scale production indigenous raw material and this can be achieved by using this process of manufacturing of NaCl [3,16,19-22].

However, high purity NaCl is not produced in Bangladesh. Mostly, this NaCl is imported from China, Japan, Korea, Germany, Australia and USA with high costs [23-25]. Therefore, in this study, we have investigated the production of high purity NaCl from impurities salt with solvent using crystallization method which would fulfill in national demand.

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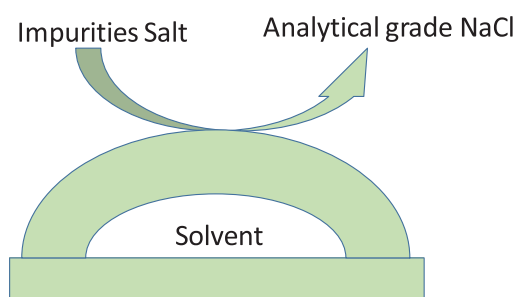
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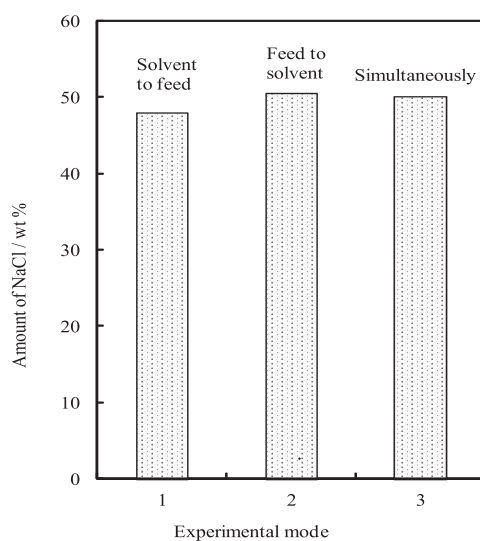
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**Methodology:** A 250 ml Pyrex glass beaker fitted with a magnetic stirrer was used as an agitated batch crystallizer. At first, saturated salt solutions were prepared by impurities salt which was collected from local market. Then the saturated salt solution and NB1 solvent (Merck, India) were added in an agitated batch crystallizer with continuous stirring. After stirring, the crystals were allowed to settle for 30 minutes. The wet crystals were separated by filtering through the centered glass crucible under suction. The crystals were washed several times with excess of NB1 solvent. Finally, the crystals were dried in an oven at 105°C for half an hour, and cooled in room temperature and weighed. The process was repeated using different experimental modes, feed to solvent ratio, salt concentration, and stage calculation with waste solution. All the runs were carried out at room temperature and atmospheric pressure. Impurities salt and the product were characterized in the Institute of National Analytical Research and Service (INARS), Bangladesh Council of Scientific and Industrial Research (BCSIR), Dhaka for the measurement of NaCl purity. Schematic diagram of crystallization method is shown as below.

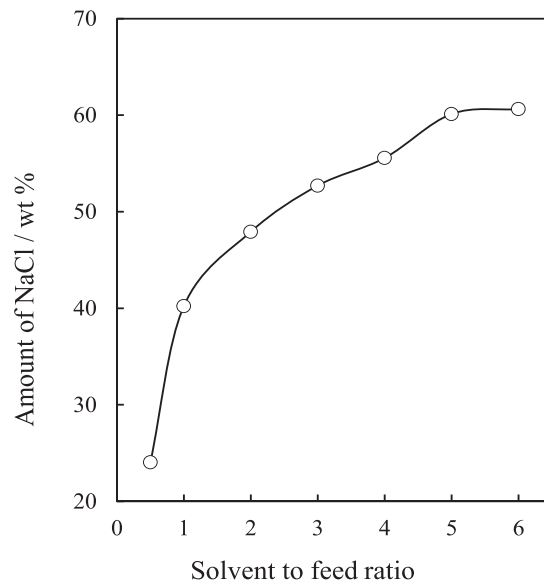


**Results and Discussion:** The effect of experimental mode between feed and solvent on the amount of NaCl using impurities salt and solvent is presented in Figure 1. Here, feed to solvent ratio was 1:3 and feed concentration was 25.4%. In this experiment, the performance of experimental mode among solvent added to feed, feed added to solvent and simultaneously added feed and solvent were observed in the agitated crystallizer. It is seen that, the amount of NaCl production was slightly higher on the addition of feed to solvent mode than that of solvent to feed mode. Figure 1 shows that the amounts of NaCl were almost similar yields over feed to solvent mode and simultaneously addition mode. The amount of NaCl on solvent to feed addition mode was 48%. Over feed to solvent mode and simultaneously mode, the amount of NaCl was almost same about 50%. Therefore, there was no significant effect on the basis of experimental mode for the production of NaCl. This result is also supported by previous report [16].

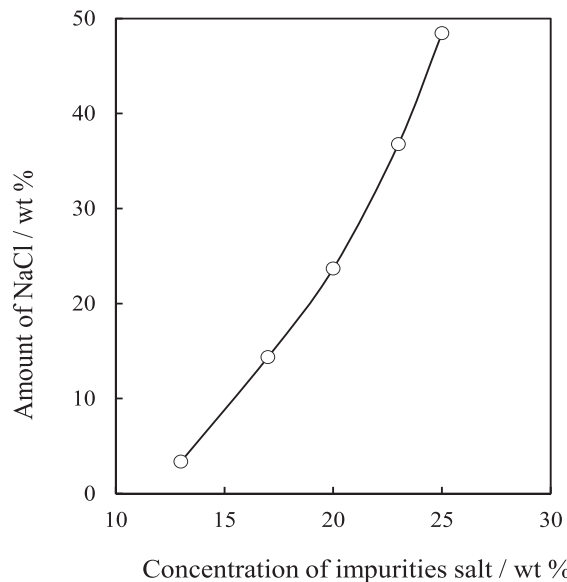


**Fig. 1:** Effect of experimental mode for the production of NaCl. Feed= impurities salt, Solvent= NB1, Feed to solvent ratio= 1:3, Settling time = 30 min.

Figure 2 shows the effect of solvent to feed ratio on the amount of NaCl production at room temperature. In this experiment, 25.4% concentration of impurities salt solution was used as feed. Settling time and room temperature were 30 min and  $(25 \pm 1)^\circ\text{C}$ , respectively. It is seen that the solvent to feed ratio increased along with increasing the amount of NaCl up to 60%. At 5 and 6 of solvent to feed ratio, the amount of NaCl was almost the same and there was no significant effect after adding more solvent. In this case, a large amount of solvent was needed, about 201 g, compared to other solvent to feed ratio. When maintaining solvent to feed ratio at 0.5, the amount of NaCl produced only 24%. However, solvent to feed ratio at 3 (100 g), the amount of NaCl was 53% and it shows suitable value compare to other values on the basis of solvent addition [18].



**Fig. 2:** Effect of solvent to feed ratio for the production of NaCl. Feed = impurities salt, Solvent= NB1, Salt concentration= 25.4%, Settling time = 30 min.



**Fig. 3:** Effect of concentration of impurities salt for the production of NaCl. Feed= impurities salt, Solvent= NB1, Feed to solvent ratio= 1:3, Settling time = 30 min.

Figure 3 represents the effect of impurities salt concentration with the amount of NaCl production at 1:3 feed to solvent ratio. It is seen that the salt concentration increased with the increase of production of NaCl sharply and it tends to attain a maximum value. At 1:3 feed to solvent ratio, we obtained maximum value of NaCl about 48% at 25% salt concentration. Above this concentration, the solution exceeded the supersaturated condition at room temperature and it became difficult to extract pure NaCl crystal in the crystallizer [9].

Data for the different impurities salt concentration for the production of NaCl is given in Table 1. In this table, we observed that the negligible amount of NaCl was produced to be 3 wt% at 13% salt concentration. This means that it is not possible to extract NaCl at low concentration of salt in the feed solution. When salt concentration in the solution increased from 17 to 25%, the production rate of NaCl was increased significantly from 14 to 48 wt%. Therefore, we obtained maximum amount of NaCl at 25% supersaturated salt concentration.

**Table 1.** Concentration of impurities salt in the solution.

No. of Observation	Wt. of salts (g)	Concentration of impurities salts (wt %)	Wt. of solvent (g)	Amount of NaCl	
				(g)	(wt %)
1	15	13	115	0.67	3.36
2	20	17	120	2.86	14.36
3	25	20	125	5.78	23.67
4	30	23	130	11.03	36.78
5	34	25	134	16.46	48.45

Salt= Impurities salt

Feed= Impurities salt solution

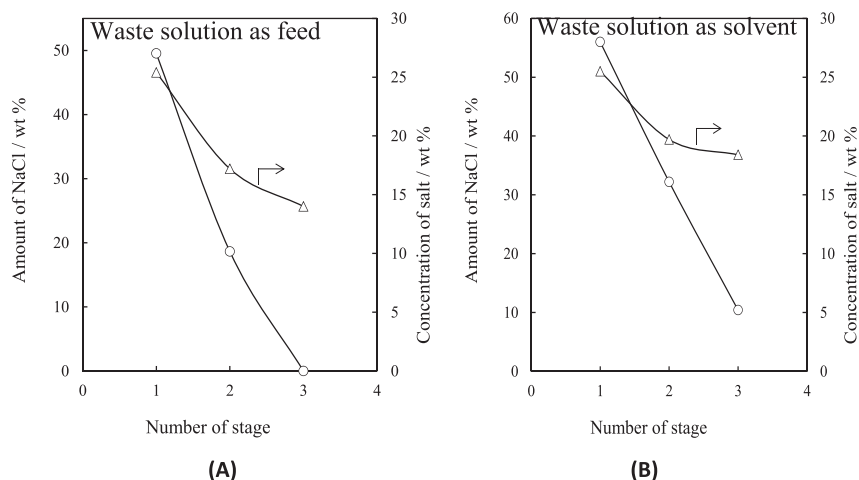
Solvent = NB1

Feed to solvent ratio = 1:3

Settling time = 30 min

Room temperature =  $(25 \pm 1)^\circ\text{C}$

Stage calculation data for the utilization of waste solution as feed and as solvent to produce NaCl crystal are presented in Figure 4 and Tables 2 and 3. Impurities salt and NB1 solvent were used in this experiment and initial salt concentration was 25.4%. Figure 4(A) shows the amount of NaCl production with the number of stage on waste solution as feed at 1:2 feed to solvent ratio. It is seen that the amount of NaCl and concentration of salt decreased as the number of stage increased. At the 3<sup>rd</sup> stage, amount of NaCl was negligible, due to the low amount of salt concentration in the feed where it used lots of NB1 solvent. This data is shown in detail in Table 2. Initial concentration of salt in feed was 25.4% and it was taken in the 1<sup>st</sup> stage with NB1 solvent where production of NaCl crystal was 50 wt%. After filtration, the rest of the waste solution was taken in the 2<sup>nd</sup> stage as 17.2% feed concentration and it was treated with NB1 solvent. Only 19 wt% NaCl crystal was extracted in the 2<sup>nd</sup> stage. After filtration, the rest of the waste solution was taken in the 3<sup>rd</sup> stage as low as 14% feed concentration and it was again treated by NB1 solvent with a maintained feed to solvent ratio. However, it was very difficult to extract NaCl crystal in this stage and the amount of NaCl was almost zero. It is indicated that the waste solution is not suitable as feed to utilize in the various stages.



**Fig. 4:** Number of stage calculation for the utilization of waste solution. Feed= impurities salt solution, Solvent= NB1, Initial concentration of salt= 25.4 %, Settling time = 30 min. (A): Waste solution used as feed, Feed to solvent ratio = 1:2. (B): Waste solution used as solvent, Feed to solvent ratio = 1:5.

**Table 2.** Number of stage calculation using waste solution as feed.

No. of observation	Number of stage	Wt. of feed (g)	Concentration of salt (wt %)	Wt. of solvent (g)	Amount of NaCl	
					(g)	(wt %)
1	1st	20.87	25.4	41.74	2.62	49.53
2	2nd	Rest of 1 <sup>st</sup> stage waste solution	17.2	90	0.49	18.63
3	3rd	Rest of 2 <sup>nd</sup> stage waste solution	14	244	0	0

Feed= Impurities salt solution.

Solvent= NB1

Initial concentration of salts= 25.4 %

Feed to solvent ratio = 1:2

Settling time = 30 min

Room temperature = (25 ± 1) °C

On the other hand, Figure 4(B) shows the amount of NaCl production with number of stage on waste solution as solvent at 1:5 feed to solvent ratio. It is seen that the amount of NaCl and concentration of salt decreased as the number of stage increased. At the 3<sup>rd</sup> stage, the amount of NaCl was extracted about 10 wt% due to the low amounts of NB1 solvent which remained in the solution. This data is also presented in Table 3. Initial concentration of salt in feed was 25.4% and it was taken in the 1<sup>st</sup> stage with maintained NB1 solvent where production of NaCl crystal was 56 wt%. After filtration, the rest of the waste solution was taken in the 2<sup>nd</sup> stage as solvent, and this solvent was treated with freshly added feed as 19.5% salt concentration. 32 wt% NaCl crystal was extracted in this 2<sup>nd</sup> stage. This indicates that it is possible to utilize the rest of the waste solution as solvent instead of NB1 solvent addition in the 2<sup>nd</sup> stage for the production of NaCl crystal. This means that the sufficient amount of NB1 solvent remained in the rest of the waste solution in this stage.

**Table 3.** Number of stage calculation using waste solution as solvent.

No. of observation	Number of stage	Wt. of feed (g)	Concentration of salt (wt %)	Wt. of solvent (g)	Amount of NaCl	
					(g)	(wt %)
1	1st	5	25.4	25	0.71	56.0
2	2nd	5	19.5	Rest of 1 <sup>st</sup> stage waste solution	0.59	32.2
3	3rd	5	18.4	Rest of 2 <sup>nd</sup> stage waste solution	0.26	10.4

Feed= Impurities salt solution.

Solvent= NB1

Initial concentration of salts= 25.4 %

Feed to solvent ratio = 1:5

Settling time = 30 min

Room temperature = (25 ± 1) °C

After filtration, the rest of the waste solution was taken in the 3<sup>rd</sup> stage as low as solvent concentration and it was treated with freshly added feed as 18.4% salt concentration. Amount of NaCl crystal was extracted about 10 wt% in this stage. This means very low concentration of NB1 solvent remained in the 3<sup>rd</sup> stage and it is not sufficient for NaCl extraction. Therefore, it is clear that we can utilize the rest of the waste solution as solvent to produce NaCl crystal only for the 2<sup>nd</sup> stage. On the basis of these experiments, we can indicate that the process is beneficial in economic value for the perspective of industrial production.

Both impurities salt and the product were analyzed by Flame Photometer, Ultraviolet-Visible Spectrophotometer and Atomic Absorption Spectrophotometer in the Institute of National Analytical Research and Service (INARS), Bangladesh Council of Scientific and Industrial Research (BCSIR), Dhaka. In this characterization, the raw impurities salt contained 35.15% Na<sup>+</sup>, 62.02% Cl<sup>-</sup>, 0.58% Ca<sup>2+</sup>, 0.55% Mg<sup>2+</sup>, 0.12% K<sup>+</sup> and 1.58% SO<sub>4</sub><sup>2-</sup> ions, and the purity of NaCl was estimated to be 97.2%. After treating the raw impurities salt by solvent, the product contained negligible amounts of impurities ions and these values were 38.54% Na<sup>+</sup>, 61.23% Cl<sup>-</sup>, 0.07% Ca<sup>2+</sup>, 0.04% Mg<sup>2+</sup>, 0.02% K<sup>+</sup> and 0.10% SO<sub>4</sub><sup>2-</sup> ions. The purity of NaCl in the product was 99.8%.

**Conclusions:** In the experimental mode: solvent added to feed, feed added to solvent, and feed and solvent added simultaneously in the crystallizer. There was no significant effect on the basis of experimental modes to produce NaCl crystal. Production rate of NaCl increased with increasing the ratio of feed to solvent. At 1:3 feed to solvent ratio, NaCl crystals were produced around 53 wt% from impurities salt solution with solvent. Concentration of impurities salt, salt concentration increased while increasing the production of NaCl sharply and it tends to attain a maximum value of NaCl at 25% salt concentration. Three-stage extraction diagram showed that waste solution from the 1<sup>st</sup> stage can be utilized as a solvent in the 2<sup>nd</sup> stage for the production of NaCl. In the characterization, it was found that the purity of NaCl in the raw salt was 97.2%, and the purity of NaCl in the product was 99.8%.

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