06. The Role of Na 2 CO 3 as Precipitating Agent in Salt Purification Process

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The Role of Na_2CO_3 as Precipitating Agent in Salt Purification Process

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ABSTRACT

Salt is an essential compound in life. It is used for food preservation. It is also used for health or isotonic drink, additive in soap production, dialysate, and use an infusion liquid. In the industrial sector, the minimum purity of NaCl in salt was 98.5%. Therefore, the impurities in raw salt from coarse salt should be removed to increase the purity of NaCl and increase the economic value of raw salt. The extraction and recrystallization method can separate the impurities in raw salt by adding the precipitating agent Na_2CO_3. The mol ratio of Na_2CO_3 and the stirring time were varied to optimize the weight of Ca extracted and give the high purity. The result showed that using Na_2CO_3 mol ratio of 1-2.5 will drastically reduce the Ca content (0.03-0.01%) in stirring time 60-150 min. The maximum purity of NaCl obtained from the extraction and recrystallization process was 98.66% using a ratio mol of 1:1 and stirring time of 150 min.

Keywords: Recrystallization, raw salt, NaCl, Na2CO3

Introduction

Salt is one mineral mainly composed of NaCl. Salt is an essential nutrient compound in human life as an electrolyte and osmotic solute. Salt is used as food preservation, food flavoring, health/isotonic drink, as an additive in soap production, dialysate fluid, and an infusion fluid. In general, salt is produced from salt mines by seawater evaporation in the salt fields. The seawater evaporation may cause a high concentration of seawater, and at a specific level of salt concentration, the crystallization of salt happens gradually (Ihsan et al., 2002).

In the industrial process, salt is the primary commodity as the raw materials in chlorine (Cl_2) production and caustic solution (sodium hydroxide (NaOH) and Potassium hydroxide (KOH), which is produced by the electrolysis process of salt solutions decomposition. Nowadays, 95% of chlorine production in the world uses the electrolysis method. The salt impurities such as Ca^{2+} and Mg^{2+} can cause disturbance ion Na+ crossing on cell membranes. Based on Indonesian National Standard (SNI), the minimum purity of NaCl for salt industries was 98.5%, and the maximum content of Ca^{2+} and Mg^{2+} cation were 0.1% and 0.06%, respectively (Gemati et al., 2013).

The improvement of NaCl purity in crude salt can be made by the recrystallization method. The recrystallization method purifies the raw materials by the precipitation of solid impurities and further heated and evaporation processes to produce high purity crystals (Rusiyanto et al., 2013). The impurities in coarse salt can be removed by NaOH addition to transforming $MgCl_2$ and $MgBr_2$ to precipitated $Mg(OH)_2$. The addition of Na_2CO_3 will remove $CaCl_2$ in forming precipitated $CaCO_3$ (Rahman et al., 2010). The cation of Ca^{2+} can be removed by graded crystallization with the first fraction was $CaCO_3$ and $CaSO_4$ as the precipitated salt (Mishra et al., 2010). In this research, the Ca^{2+} cation as the impurities in coarse salt was removed by recrystallization using Na_2CO_3 as the precipitating agent. The effect of the Na_2CO_3 mol ratio and the stirring time was investigated in this study.

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Material and Methods

Materials

The coarse salt as raw salt materials used in this research was acquired from Blawi, Lamongan, East Java Province.

Methods

The mol ratio of $CaSO_4$ and Na_2CO_3 used in this study was detailed in Table 1. The extraction process was conducted by mixing 300g of coarse salt with 1000 mL of demineralized water. Na_2CO_3 (100 mL) was added gradually to the mixture and stirred with 150 rpm for 30, 60, 90, 120, and 150 min, followed by a precipitation process for 90 min. Then, the solution is filtered to separate $CaCO_3$ from the solution. The filtrate is evaporated and crystallized. The salt crystal was dried in an oven at 100-110 °C for 4 hours. The process was repeated by using different mol of Na_2CO_3, as shown in Table 1. The products were further analyzed for the level impurities of Ca and NaCl purity.

Table 1. Mol ratio and the weight of $CaSO_4$ in coarse salt and Na_2CO_3

gmol/Ratio	**$CaSO_4$**	**Na_2CO_3**
1 : 0.5	10.74	4.18
1 : 1	10.74	8.36
1 : 1.5	10.74	12.54
1 : 2	10.74	16.72
1 : 2.5	10.74	20.90

Results and Discussion

The coarse salt still containing impurities such as Ca^{2+}, Mg^{2+}. Al^{3+}, Fe^{3+}, $SO4^{2-}$, I^-, Br^-, $CaSO^4$, and $MgSO_4$. The coarse salt needs to be processed to reduce the impurities content and increase the purity of NaCl. The role of Na_2CO_3 as the precipitating agent of Ca^{2+} was investigated in this study. The effect of Na_2CO_3 mol and stirring time can be optimized to obtain the highest purity of NaCl.

Firstly, the physicochemical properties of coarse salt were investigated using titrimetry and gravimetry methods. The analysis results were tabulated in Table 2. Based on titrimetry analysis, coarse salt has low purity of NaCl (49.17%). The purity of NaCl in coarse salt is not eligible for consumption and industrial process. $CaSO_4$ and $MgCl_2$ as the impurities in coarse salt were detected in gravimetry analysis.

Table 2. The physicochemical properties of Coarse Salt

No	Testing parameter	Result	Method
1.	NaCl	49,17%	Titrimetry
2.	$CaSO_4$	3,58%	Gravimetry
3.	$MgCl_2$	2,12%	
4.	H_2O	7,60%	
5.	Insoluble	980 ppm	

The effect of Na_2CO_3 mol and stirring time was investigated in this study. The results were summarized in Table 3 as the amount of Ca extracted from Coarse Salt. The results showed that the mol of Na_2CO_3 and the stirring time influence the Ca that removed from Coarse Salt. To achieve the standard for industrial salt with the NaCl purity of 98.5%, the Ca content should be 0.1% in maximum. As shown in Table 3, with the increase of Na_2CO_3 mol ratio, the weight of Ca extracted will also increase. This result was similar to the rise of stirring time. The increasing stirring time will also increase the weight of Ca extracted from Coarse Salt. The impurities of Ca^{2+} were precipitated in the form of carbonate as follows the chemical reaction of Ca^{2+} cation with $Na2CO_3$ (Gemati et al., 2013).

$CaCl_2 + Na_2CO_3 \rightarrow CaCO_3 + 2NaCl$ (1)
$CaSO_4 + Na_2CO_3 \rightarrow CaCO_3 + Na_2SO_4$ (2)
$Ca(OH)_2 + Na_2CO_3 \rightarrow CaCO_3 + NaOH$ (3)

Table 3. The weight of Ca extracted (g) in the variation of mol ratio and stirring time

Mole Ratio / Stirring-time (min)	1 : 0,5	1 : 1	1 : 1,5	1 : 2	1 : 2,5
30	9.198	9.759	10.680	10.686	10.686
60	9.417	10.641	10.686	10.698	10.695
90	9.648	10.686	10.695	10.698	10.710
120	9.774	10.695	10.701	10.701	10.710
150	9.798	10.710	10.707	10.707	10.710

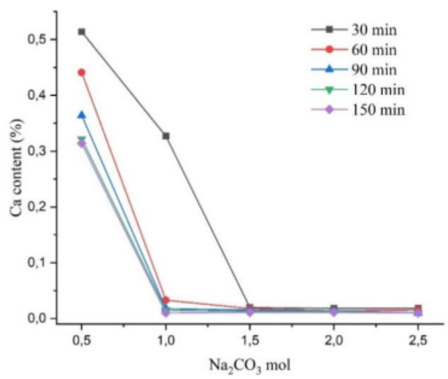

Figure 1. The effect of Na_2CO_3 mol in Ca content (%) of salt produced

The Ca content in salt produced after the recrystallization process is shown in Figure 1 and Figure 2. The correlation between the mol of Na_2CO_3 with the Ca content (%) in salt produced was clearly shown in Figure 1. Theoretically, if the mol of Na_2CO_3 is equivalent to Ca^{2+} mol in coarse salt, the salt produced will have 0% Ca. Based on the analysis, the addition of 1:1 Na_2CO_3 with the stirring time of 30 min, the Ca content was 0.327%. Furthermore, in the mol ratio of Na_2CO_3 0.5, the Ca content still has a high value (0.3-0.5%) in 30-150 stirring time. The Ca content did not meet the requirement in SNI standard.

Interestingly, increasing the number of Na_2CO_3 up to 1.5 moles can significantly reduce the Ca content up to 0.02% by stirring time 30 minutes. It was worthy to note that using Na_2CO_3 mol ratio of 1-2.5 will drastically reduce the Ca content (0.03-0.01%) in stirring time 60-150 min. These results agreed with the previous study by Ihsan et al. (2002), which increases the precipitating agent Na_2CO_3 wil the stirring time also plays an important role in coarse salt purification using the recrystallization process. The correlation between the stirring time with the Calcium content is shown in Figure 2. In mol ratio Na_2CO_3 of 1:0.5, the increasing of stirring time only gives a little impact in reducing Ca content from 0.5% in 30 min become 0.3% in 150 min. Meanwhile, in mol ratio Na_2CO_3 of 1:1, the increasing stirring time to 60 min gives a significant impact for Ca content reduction from 0.33% in 30 min become 0.01% in 60 min of stirring time. Furthermore, in mol ratio Na_2CO_3 of 1:1.5, 1:2, and 1:2.5, the increasing stirring time has no impact on Ca content reduction since the Ca content was 0.01% in 30 min of stirring time.

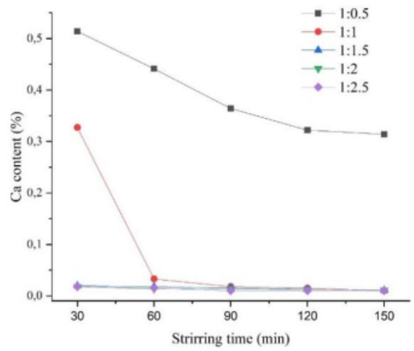

Figure 2. The effect of stirring time in Ca content (%) of salt produced

The salt produced by recrystallization proceeded using coarse salt was further analyzed for the NaCl content as shown in Table 4. The highest purity of NaCl was obtained in stirring time of 150 min with the Na_2CO_3 mol ratio of 1:1. The increasing stirring time will increase the NaCl purity in salt produced from coarse salt.

Table 4. The NaCl content in the variation of stirring time

Stirring time (min)	NaCl content (%)
30	88.490
60	91.900
90	92.690
120	94.680
150	98.660

Conclusion

The improvement of NaCl purity in coarse salt can be implemented by the extraction process using Na_2CO_3. The role of Na_2CO_3 as the precipitating agent of Ca^{2+} was investigated in this study. The increasing mol of Na_2CO_3 in the extraction process will reduce the Ca content in CoarseSalt. Furthermore, the increasing stirring time will also increase the Ca extracted and reduce the Ca content. The maximum purity of NaCl obtained from the extraction process was 98.66% using ratio mol Na_2CO_3 of 1:1 and stirring time of 150 min.

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