

The Effectiveness of Sodium Hydroxide Precipitation in Impurity Removal during NaCl Recovery from Bittern Waste Water

Indah Maharani Puspitasari^{1✉}, Andreansyah Brilliant Putra², Caecilia Pujiastuti³, Renova Panjaitan⁴

^{1, 2, 3, 4} Department of Chemical Engineering, Faculty of Engineering & Science, Universitas Pembangunan Nasional "Veteran" Jawa Timur, Indonesia

✉ **Corresponding Author** : Indah Maharani Puspitasari (e-mail : indahmaharani.sr@gmail.com)

Article Information	ABSTRACT
Article History Received : September 18, 2025 Revised : October 03, 2025 Published : October 10, 2025	Recovery of sodium chloride (NaCl) from bittern, a by-product of salt production, is often hindered by impurities such as Mg^{2+} and Ca^{2+} , which reduce NaCl purity. This study evaluated the effect of sodium hydroxide (NaOH) concentration and stirring time on NaCl recovery, using NaOH concentrations of 4.6 until 8.02 N and stirring durations of 20 until 60 minutes at a constant stirring speed of 300 rpm. The process involved NaOH addition, stirring, filtration of precipitates, and NaCl crystallization. Parameters such as impurity levels, pH, NaCl purity, and recovery percentage were analyzed. Results indicated that higher NaOH concentrations and longer stirring times generally decreased impurity levels and improved NaCl recovery. Optimal conditions 5.8 N NaOH and 50 minutes stirring yielded an impurity level of 0.0359%, NaCl purity of 89.61%, and a recovery rate of 85.57%. This study presents a novel approach by optimizing NaOH concentration and stirring time parameters to effectively minimize divalent ion contamination, thereby enhancing NaCl recovery from bittern. The findings have direct applicability in improving the efficiency of salt recovery processes in coastal salt industries, contributing to more sustainable salt production systems.
Keywords: <i>Bittern;</i> <i>Sodium Chloride;</i> <i>Impurities;</i> <i>Sodium Hydroxide;</i> <i>Recovery.</i>	

INTRODUCTION

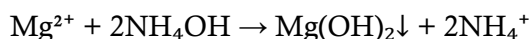
Indonesia is one of the world's largest consumers of salt, with demand driven not only by household needs but also by various industries such as glass, textiles, paper, and petrochemicals. Despite its vast coastal areas, domestic salt production remains unable to meet national demand—particularly for high-purity salt containing more than 97% sodium chloride (NaCl). Data from the Central Bureau of Statistics (BPS) show that between 2017 and 2021, Indonesia's salt imports increased from 2.5 million to 2.8 million tons, indicating a persistent gap between production and consumption. Salt is a strategic commodity for both the consumption sector and various industries, including food processing, chemicals, pharmaceuticals, and water treatment (Jumaeri et al., 2021). It primarily consists of sodium chloride (NaCl) in the form of white cubic crystals, which can be obtained either from seawater or rock salt deposits. However, naturally produced salt typically contains impurities such as magnesium chloride ($MgCl_2$), magnesium sulfate ($MgSO_4$), calcium chloride ($CaCl_2$), and calcium sulfate ($CaSO_4$), which lower its chemical purity and industrial usability (Martina et al., 2019).

In Indonesia, conventional salt production relies on solar evaporation of seawater in open ponds, producing bittern as a by-product after NaCl crystallization. Although often regarded as

waste, bittern still contains recoverable NaCl along with dissolved minerals such as MgCl_2 , CaCl_2 , and MgSO_4 (Rahem & Kartika, 2020). High concentrations of Mg^{2+} and Ca^{2+} act as impurities that lower NaCl quality, making it unsuitable for industrial use without further treatment (Martina et al., 2019). To enhance purity, methods such as recrystallization, which dissolves and re-forms the salt to remove surface and internal impurities and hydromilling, which mechanically breaks down crystals to release entrapped impurities (Umam, 2019) have been applied successfully.

Salt production by solar evaporation leaves behind a concentrated residual solution known as bittern, which still contains sodium chloride (NaCl) along with impurities such as magnesium chloride (MgCl_2) and calcium chloride (CaCl_2) that reduce product purity (Hoiriyah, 2019). Bittern typically has a salinity above 28° Baumé ($^\circ\text{Bé}$), reaching up to $30\text{--}32^\circ\text{Bé}$, with a pH of 6–8 (Laili et al., 2022). The Baumé degree serves as a key parameter for assessing density and readiness for crystallization (Megawati et al., 2021). In Indonesia, bittern production in regions such as Sampang, Pamekasan, and Sumenep reaches about 766,809 m^3 per season, indicating its high potential for further utilization in industrial and food applications (Nuzula et al., 2020). Given its relatively high NaCl content, bittern can be further processed to recover salt via recrystallization, which requires the removal of magnesium and calcium through appropriate separation techniques such as chemical precipitation. The pH range and Baumé degree of bittern are critical parameters that influence both the effectiveness of impurity separation and the quality of the final NaCl product (Radja et al., 2021).

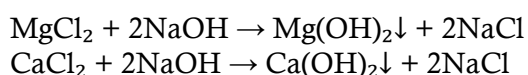
The extraction of dissolved minerals from brine or bittern can be accomplished through chemical precipitation or electrochemical methods. Chemical precipitation is often preferred due to its operational simplicity, the availability of inexpensive reagents, and cost-effectiveness. This method relies on increasing the solution's saturation level beyond its solubility threshold to induce nucleation and the subsequent formation of solid salts. Several precipitating agents have been investigated, each with distinct outcomes. Ammonium hydroxide (NH_4OH) offers high recovery of $\text{Mg}(\text{OH})_2$, achieving yields of 97.6–98.3% (Darwis et al., 2022). In precipitation using NH_4OH , the main reaction is as follows:



However, the filtrate produced contains residual ammonia (NH_3), which is harmful and requires specialized treatment (Pujiastuti et al., 2016). Precipitation with calcium carbonate (CaCO_3) is slower and generally produces $\text{Mg}(\text{OH})_2$ of lower purity, while leaving dissolved carbonate in solution (Morgante et al., 2022). In addition, excess CaCO_3 may co-crystallize as impurities in the final NaCl product (Yulastuti & Cahyono, 2020). A combination of sodium hydroxide (NaOH) and sodium carbonate (Na_2CO_3) has also been applied to simultaneously reduce Mg^{2+} and Ca^{2+} concentrations. NaOH precipitates $\text{Mg}(\text{OH})_2$, whereas Na_2CO_3 precipitates CaCO_3 or $\text{Ca}(\text{OH})_2$. However, carbonate residues may persist as impurities during NaCl crystallization.

Electrochemical methods such as electrolysis provide an alternative that avoids excess chemical reagents. In this process, $\text{Mg}(\text{OH})_2$ forms as a paste on the cathode and can be dried into a solid. Nonetheless, product purity remains a challenge. For example, Amrulloh, (2017) reported a $\text{Mg}(\text{OH})_2$ purity of only 81.735% from Pamekasan bittern, with NaCl and CaCO_3 identified as the main impurities.

In contrast, sodium hydroxide (NaOH) has demonstrated superior performance. As a strong base, it efficiently precipitates both Mg^{2+} (as $\text{Mg}(\text{OH})_2$ at pH 9–11) and Ca^{2+} (as $\text{Ca}(\text{OH})_2$ at pH >12,4), and is reported to produce $\text{Mg}(\text{OH})_2$ with a purity exceeding 99% (Astuti et al., 2016). The primary reactions are as follows:



In conclusion, NaOH offers high efficiency and product purity; NH_4OH provides high yield but poses challenges related to ammonia waste; CaCO_3 and Na_2CO_3 are cost-effective yet risk leaving

carbonate impurities; whereas electrolysis presents a reagent-free alternative, albeit with lower product purity that requires further optimization. The approach to purifying NaCl from bittern is sodium hydroxide (NaOH) based precipitation. In principle, the addition of NaOH increases pH and provides hydroxide ions (OH^-) to precipitate impurity ions as insoluble hydroxides.

Based on this background, this study was conducted to evaluate the effect of variations in NaOH concentration and stirring time on the impurities, purity, and NaCl recovery levels of bittern, with constant stirring speed. The results of this research are expected to contribute to the development of bittern processing technology into high-quality industrial salt products, as well as increase the use of liquid waste from salt production into economically valuable resources.

RESEARCH METHODS

Materials and Equipment

The main material used was bittern obtained from PT. Garam (Persero), Sampang, East Java, Indonesia, with a concentration of 32°Baumé which containing NaCl as the primary component; magnesium (Mg^{2+}), and calcium (Ca^{2+}) as major impurities. Analytical grade sodium hydroxide (NaOH, >98% purity) served as the precipitating agent, while distilled water was used for reagent preparation. The equipment used in this study includes a series of recovery NaCl from Bittern with precipitation shown in Figure 1, consisting of a Stand (Retort stand/Support stand), Thermometer, Beaker glass, Power outlet, Magnetic stirrer cable, Magnetic stirrer, as well as other tools such as a Baumémeter, magnetic stirrer with heating capability, analytical balance, pycnometer, filtration apparatus with Whatman No. 42 filter paper, and oven for drying the recovered salt.

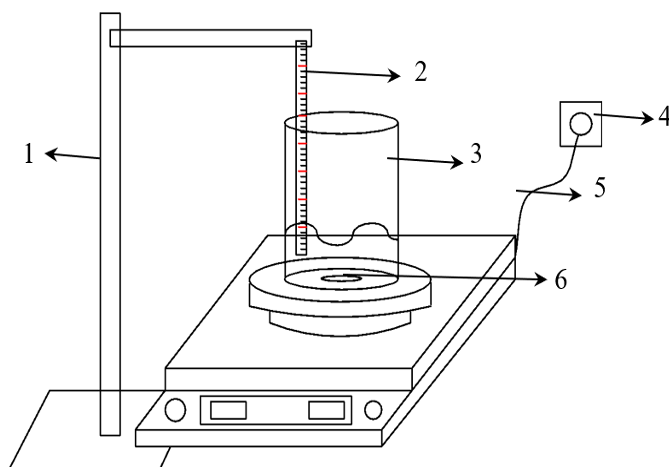


Figure 1. Experimental setup for NaCl recovery from bittern using NaOH precipitation

Procedure Experimental

The experimental procedure was divided into three main stages: pre-treatment, precipitation, and crystallization. In the pre-treatment stage, bittern waste with an initial concentration of 32° Baumé was filtered using standard filter paper to remove suspended solids, sludge, and other insoluble impurities. During the precipitation stage, sodium hydroxide (NaOH) solution was added to the filtered bittern. The process employed NaOH concentrations of 4.6 N, 5.3 N, 5.8 N, 6.6 N, and 8.02 N, with stirring times of 20, 30, 40, 50, and 60 minutes at a constant speed of 300 rpm, until a white slurry-like precipitate was formed. The mixture was then left to stand at room temperature for 24 hours to allow complete sedimentation. The precipitate was separated from the filtrate by vacuum filtration using Whatman No. 42 filter paper.

In the crystallization stage, the filtrate was heated to 90 °C and stirred at 350 rpm until a supersaturated state was reached, initiating salt crystal formation. The resulting NaCl crystals were collected and dried in a laboratory oven at 105 °C for 15–20 minutes to remove residual moisture. The drying and weighing process was repeated until a constant mass was achieved. The final product was analyzed using titrimetry to determine NaCl purity, while magnesium (Mg) and calcium (Ca) contents were quantified using Atomic Absorption Spectrophotometry (AAS).

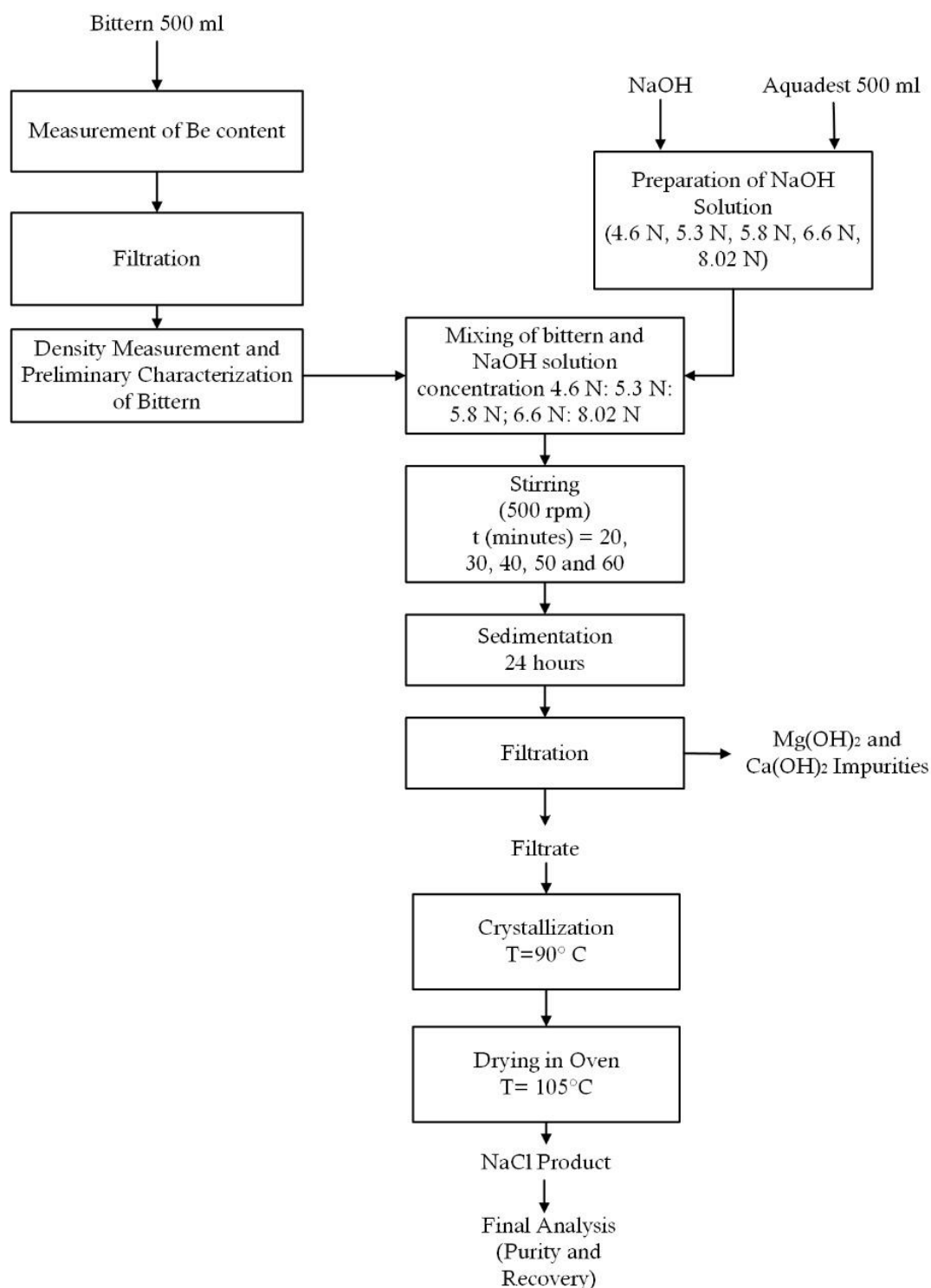


Figure 2. Experimental procedure for NaCl recovery from bittern.

RESULTS AND DISCUSSION

The raw material utilized in this study was bittern waste sourced from PT. Garam (Persero) in Sampang, Madura, Indonesia. An initial compositional analysis was conducted to serve as a baseline for the subsequent purification process. The analysis revealed that the bittern is primarily composed of sodium chloride (NaCl), with magnesium (Mg) and calcium (Ca) as its major impurities. The complete results of this chemical analysis are presented in Table 1

Table 1. Initial Content Analysis of bittern waste

Compound	Composition (%)	method
Mg	6,3531	AAS
Ca	1,227	AAS
NaCl	30,44	Titrimetric

As shown in Table 1, the initial bittern contains a high concentration of impurities, particularly magnesium, which necessitates a purification step to increase the quality of the final NaCl product.

Effect of NaOH Concentration and Mixing Time

Table 2. Analysis of NaCl Recovery and Purity

NaOH Concentration (N)	Mixing Time (minute)	Impuritis Content (%)	NaCl Recovery	NaCl Puritiy (%)
4,60 N	20	0,1597	62,53	72,65
	30	0,1059	65,48	76,87
	40	0,0803	67,65	73,94
	50	0,0516	70,83	75,43
	60	0,0481	73,36	78,19
5,30 N	20	0,1556	68,76	75,28
	30	0,0916	71,14	78,65
	40	0,0717	74,24	78,83
	50	0,0481	76,46	80,08
	60	0,0432	78,20	82,63
5,80 N	20	0,1366	77,52	79,96
	30	0,0859	78,77	81,32
	40	0,0673	83,16	83,43
	50	0,0435	85,57	89,61
	60	0,0359	83,69	87,38
6,61 N	20	0,1261	74,32	79,76
	30	0,0847	72,97	81,42
	40	0,0686	80,23	83,12
	50	0,0631	81,36	86,88
	60	0,0549	82,23	84,71
8,02 N	20	0,1212	71,06	79,03
	30	0,0763	72,52	79,76
	40	0,0701	77,63	80,83
	50	0,0634	76,14	83,43
	60	0,0491	79,32	81,92

Table 2 shows that increasing NaOH concentration and stirring time reduces impurity levels while enhancing NaCl recovery and purity. The optimum condition was achieved at 5.80 N

with 50 minutes of stirring, resulting in the lowest total impurities 0.0359%, the highest recovery 85.52%, and maximum purity 89.61%. In contrast, at 4.60 N with 20 minutes of stirring, impurities remained high 0.1597%, with the lowest recovery 60.18% and purity 72.65%. These results indicate that both the concentration of NaOH and the duration of mixing play a critical role in the precipitation process. Higher concentrations of NaOH provide sufficient hydroxide ions (OH^-) to effectively precipitate magnesium and calcium impurities, while longer stirring times promote more complete reactions and enhance crystal growth. However, excessively high concentrations or prolonged mixing may not yield significant additional improvements, suggesting the existence of an optimum operating range. The findings therefore highlight the importance of balancing reagent dosage and mixing conditions to maximize NaCl recovery and purity from bittern.

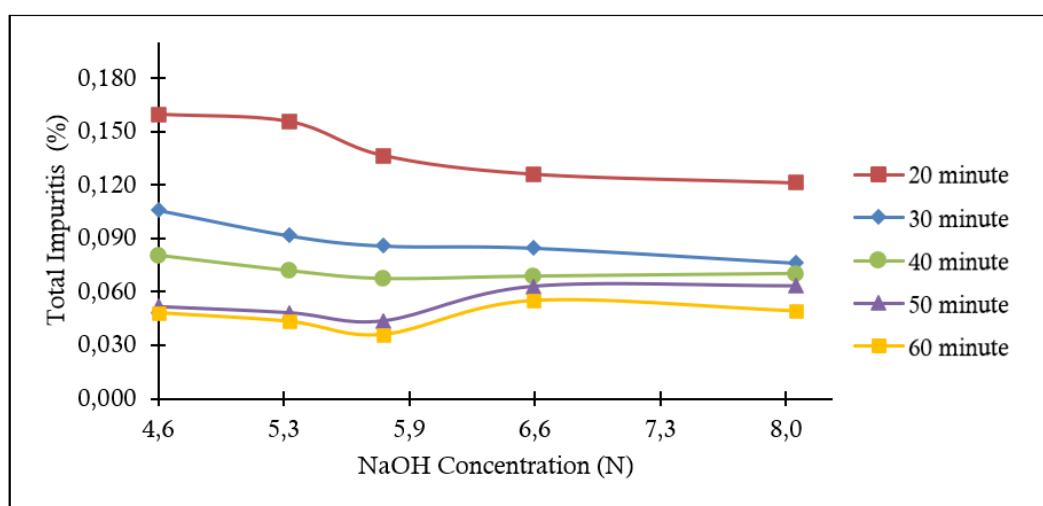


Figure 3. Effect of NaOH concentration and mixing time on total impurity content

Figure 2 shows that impurity levels decrease with increasing NaOH concentration, with the lowest value of 0.0359% obtained at 5.8 N and 60 minutes of stirring. At 4.6 N and 5.3 N, a clear reduction is also observed, while at higher concentrations 6.6 N and 8.02 N the precipitation efficiency becomes unstable under longer stirring times, leading to a slight increase in impurity levels. This trend aligns with previous findings by Trisanti et al., (2021) and Bouaziz et al., (2024) reported that increasing alkalinity promotes the precipitation of divalent ions Mg^{2+} and Ca^{2+} as $\text{Mg}(\text{OH})_2$ and $\text{Ca}(\text{OH})_2$ due to enhanced nucleation and higher OH^- ion availability. Liu et al., (2020) and (Harjono et al., 2024) further explained that NaOH addition increases solution alkalinity, which raises the pH and accelerates the nucleation of $\text{Mg}(\text{OH})_2$ and $\text{Ca}(\text{OH})_2$ formation, facilitating impurity removal. According to (Spiridonov et al., 2024) higher NaOH concentrations can accelerate the reaction up to 2.5 times faster; however, at pH values above 11, $\text{Mg}(\text{OH})_2$ tends to redissolve, increasing impurity levels under prolonged stirring (Almoussa et al., 2024). Overall, high NaOH concentrations accelerate impurity removal but require precise control of pH and mixing time to prevent re-dissolution. These findings emphasize the importance of optimizing operational parameters to maintain a balance between precipitation rate and product stability in NaCl recovery from bittern.

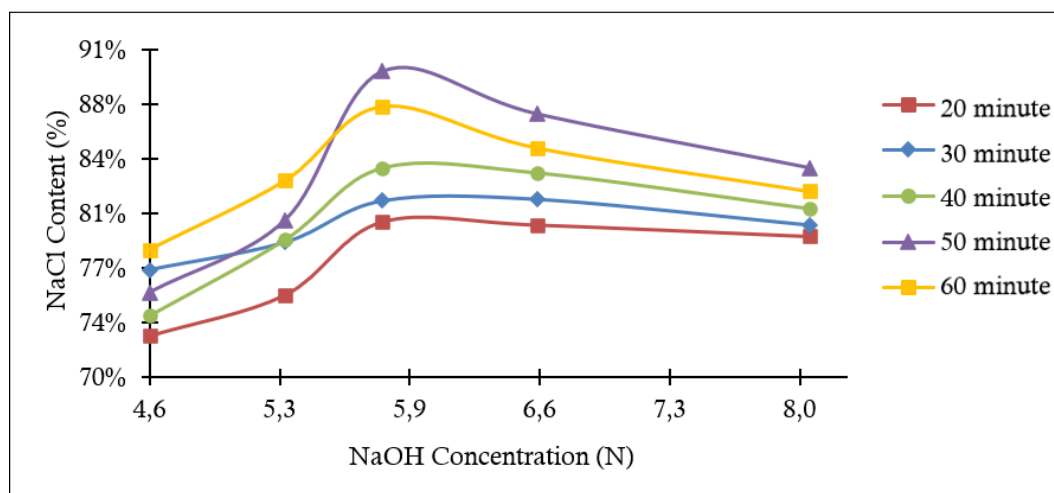


Figure 4. Effect of NaOH concentration and mixing time on NaCl purity.

Figure 3 shows that increasing NaOH concentration up to 5.8 N improves NaCl purity, reaching the highest value of 89.61% at 50 minutes of stirring, while the lowest purity of 72.65% occurs at 4.6 N and 20 minutes. The low purity at 4.6 N is attributed to insufficient OH^- ions to bind divalent impurities, leading to incomplete precipitation (Spiridonov et al., 2024). Similar results were observed by Tristani et al. (2021), where higher NaOH concentrations enhanced the removal of Mg^{2+} and Ca^{2+} , resulting in better NaCl crystallization and higher purity. However, at concentrations 6.6 N and 8.02 N, NaCl purity decreases to 86.88% and 84.71%, respectively. This decline is consistent with findings by (Battaglia et al., 2022) showing that excessive alkalinity leads to the formation of very fine particles ($<2.5 \mu\text{m}$) that pass through filtration media. Under these highly supersaturated conditions, nucleation dominates over crystal growth (Romano et al., 2023), producing numerous small nuclei that fail to develop into larger crystals. Consequently, some impurities remain dissolved and co-crystallize with NaCl, reducing overall purity NaCl (Widjaja et al., 2024). Overall, these findings indicate that maintaining NaOH concentration near the optimal value (5.8 N) ensures a balance between impurity removal and controlled crystal growth. This supports the importance of precise process optimization to achieve high NaCl purity while preventing impurity co-precipitation during bittern treatment.

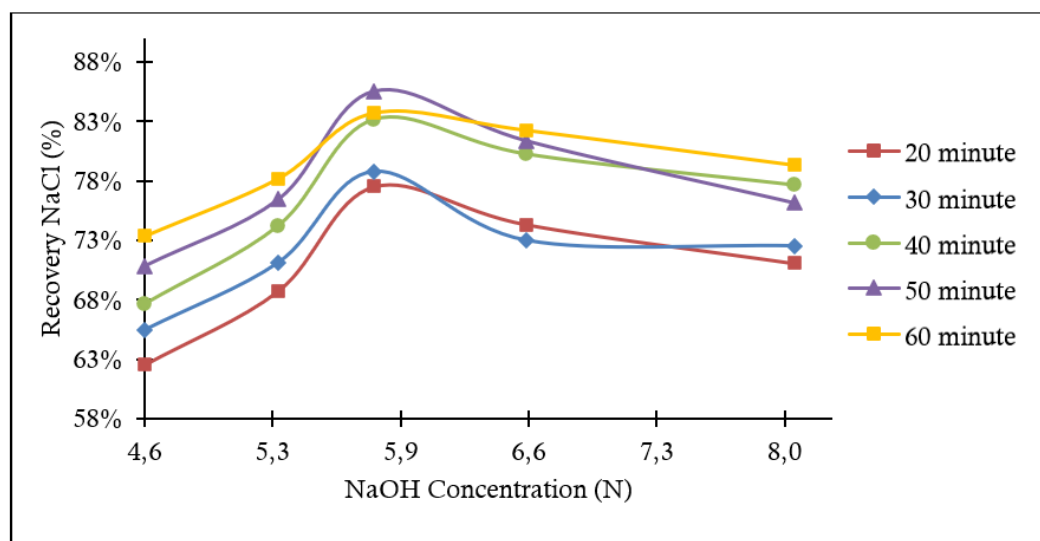


Figure 5. Effect of NaOH concentration and mixing time on NaCl recovery.

As shown in Figure 4, NaCl recovery increased with higher NaOH concentrations, reaching a maximum of 85.57% at 5.8 N and 50 minutes of stirring. At lower concentrations (4.6 N and 5.3

N), recovery ranged from 62.53% to 73.36%, mainly due to insufficient OH^- ions to fully precipitate Mg^{2+} and Ca^{2+} , allowing residual impurities to remain in solution and reduce yield (Redjeki et al., 2024). This finding supports the results of Rizqi Mubarak et al., (2021), who noted that lower impurity levels correlate directly with higher product recovery. When NaOH concentration exceeded the optimum level 6.6 N and 8.02 N, recovery declined to 74.32 until 82.23% and 71.06 until 79.32%, respectively. This pattern is consistent with (Romano et al., 2023) and Liu et al., (2020), who reported that excessive alkalinity promotes redissolution of $\text{Mg}(\text{OH})_2$ and $\text{Ca}(\text{OH})_2$, leading to incomplete precipitation and impurity retention. Widjaja et al., (2024) also observed that remaining impurities can be entrapped within the NaCl lattice during crystallization, reducing purity and effective yield. Furthermore, Gemati et al., (2023) emphasized that fine particulates formed under high pH conditions are difficult to separate, causing additional recovery losses despite prolonged stirring. These results indicate that precise control of NaOH concentration and stirring duration is essential to maximize precipitation efficiency and prevent impurity co-crystallization, thereby improving NaCl recovery from bittern.

CONCLUSION

Based on the results of the study, the recovery of sodium chloride (NaCl) from bittern is strongly influenced by sodium hydroxide (NaOH) concentration and stirring time. Optimal conditions were achieved at 5.80 N NaOH and 50 minutes of stirring, yielding the lowest impurity level (0.0359%), a recovery rate of 85.57%, and NaCl purity of 89.61%. These findings confirm that hydroxide ions effectively precipitate Mg^{2+} and Ca^{2+} as $\text{Mg}(\text{OH})_2$ and $\text{Ca}(\text{OH})_2$, thereby enhancing NaCl purity and yield. Although the recovered salt meets household salt standards, it does not yet qualify for food-grade or pharmaceutical applications. This study introduces an optimization-based novelty by systematically determining the most effective NaOH concentration and stirring duration to minimize divalent ion contamination in bittern-derived NaCl. The results demonstrate improved process efficiency compared to conventional methods and provide a low-cost, scalable, and sustainable purification approach suitable for coastal salt industries. Practically, the findings contribute to the valorization of bittern waste into a higher-value salt product while supporting sustainable salt production practices. Future research may explore alternative purification agents, crystallization improvements, and process scaling to achieve higher-purity NaCl suitable for broader industrial applications.

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