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ENHANCEMENT OF SALT QUALITY WITH RECRYSTALLIZATION METHOD FROM CRUDE SALT RAW MATERIAL ON A LABORATORY SCALE

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ABSTRACT

Indonesia has significant maritime potential, particularly in salt production. However, national salt production has not yet met domestic demand, especially for industrial-grade salt, which requires a minimum NaCl content of 97%. Crude salt produced using traditional methods often contains only 88-94% NaCl and includes impurities such as Ca, Mg, and sulfate. This research aims to produce recrystallized salt that meets industrial standards and to determine the optimal conditions in a laboratory-scale simulator. The experimental method was conducted by simulating evaporation on a recrystallization table. Salt solutions were prepared from crude salt with varied concentrations of 22%, 25%, and 30%. The observed parameters included solution density, evaporation rate, and the content of NaCl, total hardness, and sulfate in the salt product. The results showed that recrystallization significantly increased the NaCl content and reduced impurities. Optimal conditions were achieved with a 22% salt solution, where crystal formation produced recrystallized salt with an NaCl content of 99.33%, total hardness of 0.47%, and sulfate content of 8.56 ppm. This quality has the potential to meet industrial salt standards. This study can serve as a reference for industrial salt production on a larger scale.

Keyword: *Folk salt, Industrial grade salt, Laboratory scale, Recrystallization.*

1. INTRODUCTION

Indonesia possesses great potential in the utilization of its marine sector, one aspect of which is salt production. Three-quarters of Indonesia's territory, covering 5.9 million km² is ocean and it has a coastline of 95,161 km, the second longest after Canada [1]. However, this geographical data does not align with Indonesia's capacity to meet its domestic salt needs. In 2023 the domestic salt requirement was 4.9 million tons, while national production only reached 2.5 million tons [2]. To date, salt production is widespread across many regions in Indonesia [3]–[5].

Most of salt in Indonesia is still produced through traditional methods, using seawater as the raw material, which is processed via evaporation in ponds or basins equipped with tunnels [3], [6]. This evaporation process increases the density of the seawater, and under certain conditions, it undergoes recrystallization to form salt [7], [8]. The product resulting from this method is often called crude salt due to its quality not meeting industrial standards, particularly its NaCl content. This is because crude salt contains only 88-94% NaCl, failing to meet the industrial standard which requires an NaCl content of no less than 97% [9], [10].

The failure of crude salt to meet industrial requirements is due to several factors [11]. One contributing aspect is the timing of harvesting salt crystals from the evaporated seawater, known as "air tua" (old water), which is done immediately after the crystals form. Additionally, bittern is added to the old water to increase the Baumé degree, thereby accelerating the recrystallization process. This practice causes many impurities to co-precipitate, leading to a high impurity content in the final salt product. Impurities found in crude salt include Ca, Mg, and sulfate. The presence of these impurities directly contributes to the low NaCl content in the salt.

Industrial salt is crucial in various sectors, especially in the food and petrochemical industries [12]. The imbalance between the demand for industrial salt in Indonesia and domestic production is a problem that needs to be addressed. Therefore, producing industrial-grade salt from crude salt raw material is a study of great potential for solving one of the issues related to food material needs. The common method for producing industrial salt from crude salt involves recrystallizing the crude salt solution after its impurities have been precipitated as calcium, magnesium, or sulfate compounds [13]. The drawback of this method is that the addition of chemical precipitating agents increases production costs, which is considered inefficient.

An alternative method that can be implemented is direct recrystallization from the old water without any precipitating agents. This method can produce industrial-standard salt based on its NaCl content by carefully controlling the operating conditions, one of which is the Baumé degree or density [14]. These operating conditions can be simulated in a small-scale laboratory system that mimics the conditions in the tunnels and ponds of a salt farm. The objective of this research is to produce a recrystallized salt product that meets industrial standards in terms of NaCl content using an evaporation and recrystallization method. The second objective is to provide an overview of the optimal conditions in a laboratory-scale simulator evaporation pond for producing industrial salt. The quality trend of the product as an industrial salt, based on NaCl and impurity content, will be periodically analyzed to assess the consistency of the salt product's quality. The optimal condition is observed from the measured Baumé degree and its relationship with the recrystallization capability of the crude salt solution. The product quality is measured by its NaCl content, total hardness (representing Ca and Mg content), and sulfate content in the recrystallized salt. The results of this study are expected to serve as a benchmark for field conditions, particularly the Baumé degree, as a reference point for harvesting recrystallized salt that meets industrial standards in terms of its NaCl content.

2. RESEARCH METHODOLOGY

This research was conducted using a laboratory experimental method that simulated the evaporation process occurring on a recrystallization table at a salt farm. The simulator set consisted of an artificial salt solution evaporation pan, which served as the recrystallization table. The evaporation pan was placed in a closed evaporation chamber with measured temperature and humidity, simulating the tunnel at a salt farm. The system was constructed on a small scale with dimensions as shown in Figure 1.

The temperature inside the evaporation chamber was maintained between 30-35°C using four 40-Watt light bulbs (Electra). The relative humidity in the evaporation chamber was kept below 60% using two 21-Watt exhaust fans (Winner). The room temperature, salt solution temperature, and relative humidity were adjusted to match the actual conditions measured in the tunnels of a salt farm [4], [7], [15].

Raw material for the recrystallized salt was prepared by dissolving crude salt in distilled water. The concentration value of the salt solution varied at 22%, 25%, and 30% (w/v). The prepared salt solution was placed in the recrystallization table simulator set under the previously described conditions. The parameters observed from the salt solution during the recrystallization process were the solution's density and evaporation rate, which were monitored periodically. The recrystallized salt formed from the process was analyzed for its NaCl content as a salt quality parameter. Total hardness and sulfate as impurity parameters of the salt product was also analyzed. The analysis of the product's quality and impurities was also conducted periodically. The complete research workflow is shown in Figure 2.

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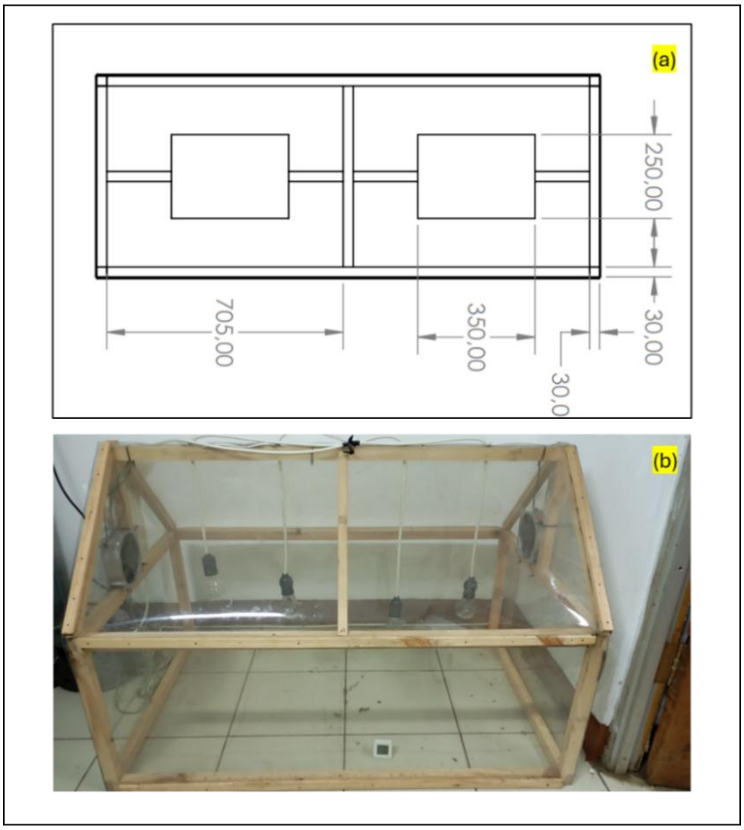

Figure 1. Schematic and dimension of evaporation and recrystallization simulator from above (a) and full-shaped evaporator and recrystallization simulator

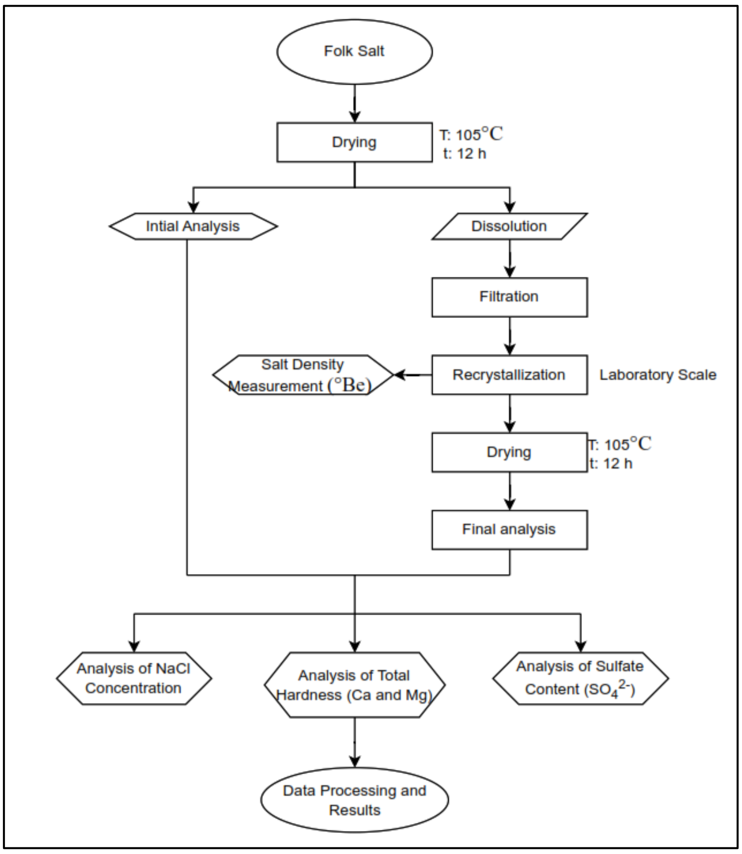

Figure 2. Research workflow diagram

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The quality analysis of the recrystallized salt, specifically the NaCl content, was performed using the argentometry method [16]. Salt product impurities were expressed by the total hardness parameter, which was analyzed using the complexometry method [17], while sulfate impurities were analyzed using UV spectrophotometry (Shimadzu) at a wavelength of 420 nm [17].

3. ANALYSIS AND RESULT

3.1. Characteristics of the Crude Salt Solution

The crude salt raw material was first dried before being processed into a salt solution. This process aimed to remove the moisture content on the surface of the crude salt that was physically adsorbed [18]. The mass of the dry crude salt was used to accurately prepare the desired concentrations of the salt solution.

The initial analysis of the NaCl content and impurities in the crude salt was conducted to establish a baseline for determining whether the quality of the recrystallized salt improved. The results of the quality analysis of the crude salt raw material for the recrystallization solution are presented in Table 1. The table shows that the crude salt raw material was not yet suitable as industrial-grade salt with respect to its NaCl content [19].

Table 1. Chemical parameters of folk salt as the raw material of recrystallized salt

Parameters	Contents
NaCl	91,00 %
Total hardness (Ca and Mg)	8,66%
Sulphate (SO_4^{2-})	395,67 ppm

Observation of the crude salt solution's characteristics involved monitoring the evaporation rate profile over the duration of the evaporation process. Additionally, salinity measurements on the Baumé scale were taken periodically from the salt solution. The objective of observing the prepared crude salt solution's characteristics was to determine the condition profile of the solution during the recrystallization process. The observed evaporation rate profile of the salt solution is shown in Figure 3.

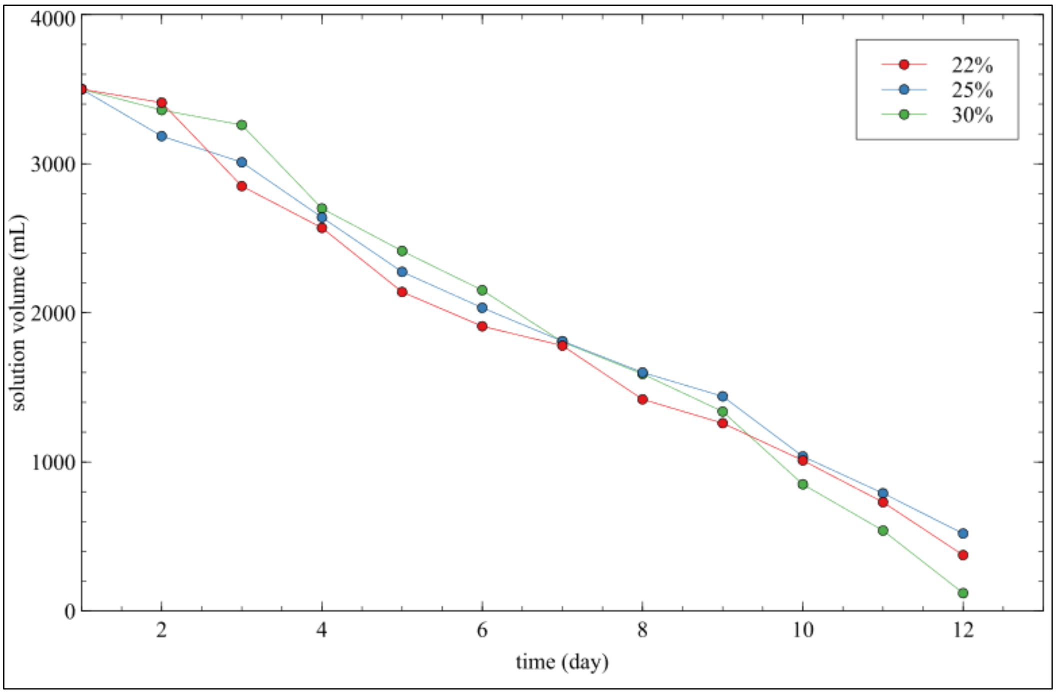

Figure 3. Evaporation rate profile in several concentration of salt solution

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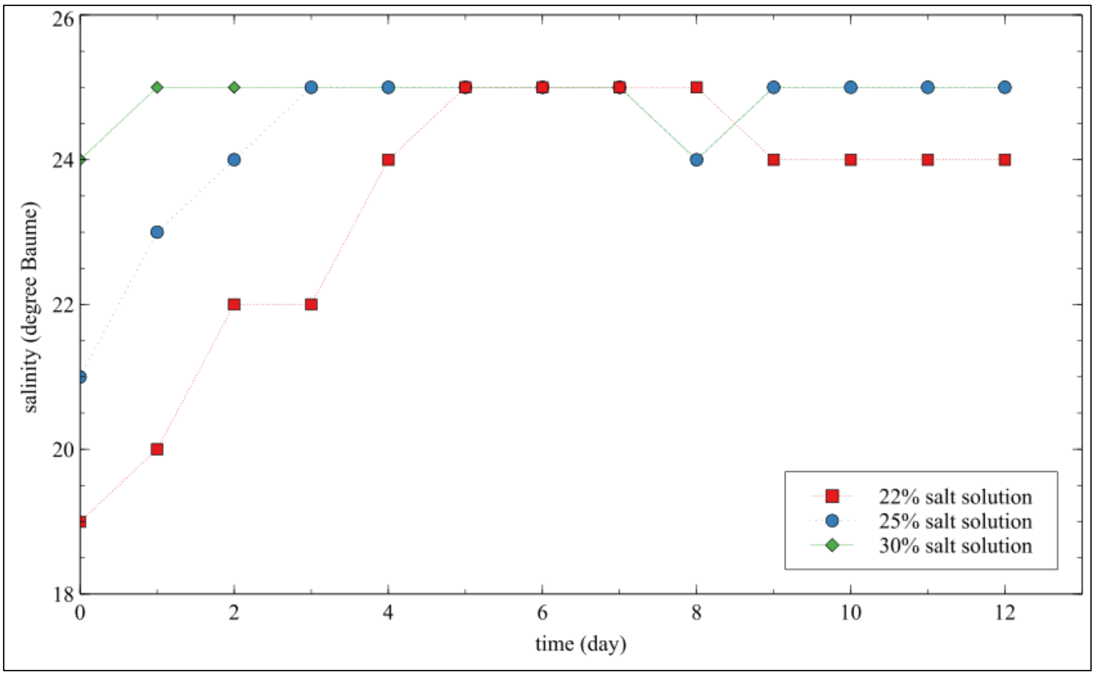

Figure 4. Salinity profile of salt solution observed in specific period

Decreasing trend phenomena in the evaporation rate of the salt solution over time is attributed to the increasing saturation of the solution. This condition hinders the formation of water vapor during the evaporation process. Furthermore, the gradual formation of salt crystals also contributes to a slower evaporation rate [20].

The measured salinity of the salt solution exhibits the trend depicted in Figure 4. These salinity values were obtained using an aerometer and are expressed on the degree Baumé (°Be) scale. The solution's salinity showed an increasing trend across all concentrations. This is attributed to the periodic evaporation of the solvent, which causes the salt solute to become more concentrated and subsequently undergo recrystallization. A stable salinity value indicates an equilibrium between the evaporated solvent and the formed salt crystals 20. The higher the initial concentration of the salt solution, the more rapidly it reaches saturation and tends to form salt crystals.

3.2. Characteristics of the Recrystallized Salt Product

For all solution concentrations tested (22%, 25%, and 30%), salt crystals formed when the solution's salinity reached 24-25 °Be. This data can serve as a benchmark for both pilot-scale and large-scale salt ponds to determine the point at which the salt is ready to crystallize. The quality analysis of the recrystallized salt, including its NaCl content and impurities, is presented in Figure 5.

The NaCl content values indicate that recrystallization plays a significant role in increasing the NaCl content while indirectly reducing the concentration of impurities from the crude salt. The graphical trend reveals that the quality of the harvested salt fluctuated, which was attributed to inconsistent temperature, solvent volume, and harvesting times. This led to instability in the NaCl content of the recrystallized product.

The trend also shows that in each final harvest, a decrease in NaCl concentration occurred alongside an increase in impurity concentration. The NaCl content decreased significantly on the final day of product collection due to high concentrations of hardness-causing ions, namely Ca and Mg. These ions in the solution can co-precipitate with the solid recrystallized salt, thus causing a reduction in the product's final NaCl concentration. As a result, the salt formed during the final harvest had a higher hardness value than the salt collected on previous days [21].

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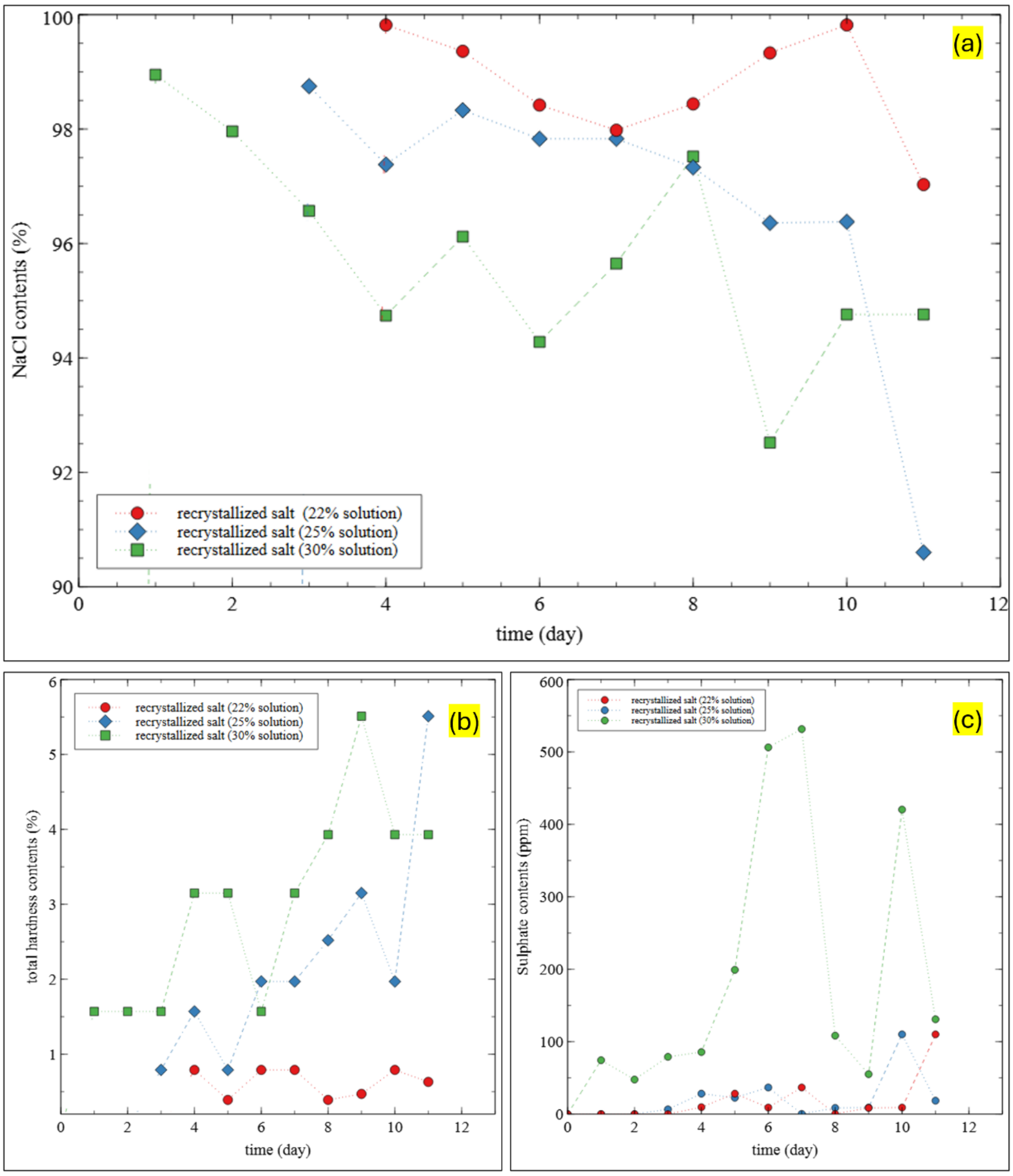

Figure 5. Profile of (a) NaCl content, (b) total hardness, and (c) sulfate in the recrystallized salt product at various initial solution concentrations

Drying the salt product at 105°C will evaporate any present water was performed to ensure the accurate determination of both NaCl and impurity content. The results obtained from the recrystallization of the 22% and 25% concentration solutions meet the requirements of the Indonesian National Standard (SNI) for industrial salt, which specifies an NaCl content greater than 97%. Based on the obtained NaCl and impurity levels, the optimal condition was achieved with the 22% solution concentration on the ninth day after it was placed in the evaporation pan. This was determined by the recrystallized salt product exhibiting an NaCl content of 99.33%, a total hardness of 0.47%, and a sulfate content of 8.56 ppm.
The 25% and 30% salt solutions exhibited relatively higher hardness values compared to the 22% concentration due to the co-precipitation of hardness-causing components with the salt, which crystallized under conditions of rapid saturation.
These optimal results can be applied on a larger pilot-plant scale. However, the salt produced from the 30% concentration solution still yielded an NaCl concentration below the required standard. This is attributed to the solution exceeding its saturation point during the initial preparation, as indicated by the presence of an incompletely dissolved precipitate. The saturation point is defined as the point at which a solvent can no longer dissolve any more solute because the maximum amount has been reached at a specific temperature. When a solution's concentration exceeds this point, the excess solute remains in solid form and does not participate in the dissolution process.

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4. CONCLUSION

Quality of folk salt can be improved to meet industrial-grade standards through an evaporation and recrystallization method, without the addition of chemical precipitating agents, on a laboratory scale using a recrystallization table simulator. The recrystallized salt with the best quality parameters was produced from a 22% salt solution and was obtained on the fifth day of the evaporation and recrystallization process.

The best recrystallized salt exhibited an NaCl content of 99.33%, a total hardness of 0.47%, and a sulfate content of 8.56 ppm, making it potentially suitable as a raw material for food-grade industrial salt, fish preservation, general industry, and iodized salt [19]. Future research could involve similar observations on a larger scale, such as a pilot or field scale. Furthermore, the total hardness analysis could be refined to specifically distinguish between Ca and Mg content.

REFERENCES

[1] M. F. Arianto, "Potensi Wilayah Pesisir di Negara Indonesia," *J. Geogr.*, vol. 20, no. 20, pp. 1–7, 2020.

[2] Kementerian Kelautan dan Perikanan, "Siaran Pers Kementerian Kelautan dan Perikanan Nomor: SP.431/SJ.5/XI2024," Jakarta, 2024. [Online]. Available: https://kkp.go.id/news/news-detail/kkp-antisipasi-perubahan-tata-kelola-impor-garam-D1z6.html.

[3] N. Taufiq, S. Putra, and R. Hartati, "Produksi Garam Dan Bittern Di Tambak Garam," *J. Kelaut. Trop.*, vol. 19, no. 1, pp. 43–47, 2016.

[4] R. S. Palin, Sulistiono, and Majariana Krisanti, "Kabupaten Jeneponto Water and Salt Conditions At Salt Pond in Bangkala District , Jeneponto Regency," *J. Teknol. Perikan. dan Kelaut.*, vol. 13, no. 1, pp. 79–88, 2022.

[5] L. Daris, A. Suryahman, and A. N. A. Massiseng, "Studi Pemetaan Sebaran Dan Luasan Usaha Tambak Garam Rakyat Di Wilayah Pesisir Kabupaten Jeneponto Provinsi Sulawesi Selatan," *J. Perikan. Unram*, vol. 13, no. 2, pp. 436–446, 2023, doi: 10.29303/jp.v13i2.541.

[6] B. Bahagia and A. Afrizal, "Analisis Kualitas Air Laut Sebagai Bahan Baku Garam di Provinsi Aceh," *Serambi Eng.*, vol. IV, no. 2, pp. 542–548, 2019.

[7] B. K. Mu'min, A. G. D. Kartika, and M. Efendy, "Parameter Lingkungan, Kadar Air dan NaCl Bunga Garam (Fleur De Sel)," *J. Mar. Res.*, vol. 10, no. 4, pp. 570–580, 2021, doi: 10.14710/jmr.v10i4.32290.

[8] M. I. Purwati, A. Gustomi, and O. Supratman, "Analisis Kualitas NaCl dan Keadaan Garam yang Dihasilkan dari Perairan Bangka Selatan," *J. Trop. Mar. Sci.*, vol. 3, no. 2, pp. 53–64, 2020, doi: 10.33019/jour.trop.mar.sci.v3i2.1783.

[9] S. A. Pebrianti, S. H. Kusumah, and N. Yunita, "Identifikasi Permasalahan Kualitas Garam Industri di PT Niaga Garam Cemerlang Menggunakan Check Sheet, Pareto Chart dan Fishbone Analysis," *J. Ilmu Tek.*, vol. 2, no. 3, pp. 1–23, 2021.

[10] Y. U. Hoiriyah, "Peningkatan Kualitas Produksi Garam Menggunakan Teknologi Geomembran," *J. Stud. Manaj. dan Bisnis*, vol. 6, no. 2, pp. 71–76, 2019, doi: 10.21107/jsmb.v6i2.6684.

[11] Indriyanti, Kasmawati, and G. Farida, "Analisis Potensi Kapasitas Produksi Garam Berdasarkan Rasio Komponen Tambak Garam Di Kecamatan Arungkeke Kabupaten Jeneponto," *Nusant. J. Ilmu Pengetah. Sos.*, vol. 8, no. 7, pp. 1838–1850, 2021.

[12] A. Fitrayawati, Y. Rahmawati, N. Amin, and S. Nurkhamidah, "Pra Desain Pabrik Pembuatan Garam Industri Soda Kaustik dari Garam Rakyat," *J. Tek. ITS*, vol. 10, no. 2, 2021.

[13] K. Akbar, M. Zamhari, E. Dewi, and J. T. Kimia, "Pemurnian Garam Rakyat Menjadi NaCL Standar Industri Purification Of Folk Salt Into Industrial Standard NaCl," *J. Tek. Sains*, vol. 08, no. 02, pp. 126–134, 2023.

[14] D. Prihatmoko, A. Mustofa, A. Faidlon, and Z. Arifin, "Rancang Bangun Sistem Kontrol Dan Monitor Produksi Garam Menggunakan Internet of Things," *J. Disprotek*, vol. 15, no. 1, pp. 65–72, 2024, doi: 10.34001/jdpt.v15i1.5895.

[15] R. Hartati, W. Widianingsih, B. W. RTD, M. B. Puspa, and E. Supriyo, "Analisa Air Tambak Desa Kaliwlingi sebagai Bahan Baku Produksi Garam Konsumsi," *J. Mar. Res.*, vol. 11, no. 4, Nov. 2022, doi: 10.14710/jmr.v11i4.35353.

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E-ISSN : 2621-3966

[16] Badan Standardisasi Nasional, "SNI 8207:2016 Garam industri aneka pangan." Badan Standardisasi Nasional, Jakarta, pp. 1–19, 2016, [Online]. Available: www.bsn.go.id.
[17] C. S. Kamilah and N. S. Octaviani, "Optimasi Penurunan Kadar Ca, Mg dan SO42- pada Garam Rakyat," Politeknik Negeri Bandung, 2024.
[18] A. Pramanik, R. P. Kalagi, V. J. Barge, and S. R. Gadre, "Adsorption of water on sodium chloride surfaces: electrostatics – guided ab initio studies," *Theor. Chem. Acc.*, vol. 114, no. 1, pp. 129–136, 2005, doi: 10.1007/s00214-005-0653-0.
[19] H. N. Firdaus, M. F. Harun, Y. Rahmawati, and S. Susianto, "Pra Desain Pabrik Garam Farmasi dari Garam Olahan dengan Kapasitas Produksi 2000 Ton/Tahun," *J. Tek. ITS*, vol. 13, no. 3, pp. 166–171, 2024.
[20] L. B. Bunio, J. Wang, R. Kannaiyan, and I. D. Gates, "Evaporation and crystallization of NaCl-water droplets suspended in air by acoustic levitation," *Chem. Eng. Sci.*, vol. 251, p. 117441, 2022, doi: https://doi.org/10.1016/j.ces.2022.117441.
[21] E. Wae, R. T. Tjahjanto, and D. Mardiana, "Study of Sodium Chloride Production Using Gradual Evaporation of Seawater from Nggolonio Sea, East Nusa Tenggara," *J. Pure Appl. Chem. Res.*, vol. 10, no. 2, pp. 123–131, 2021, doi: 10.21776/ub.jpacr.2021.010.02.483.