



ARTIKEL PENELITIAN

An extensive analysis and examination of techniques to enhance the efficiency of water extraction from wastewater generated during the recycling of nickel manganese cobalt (NMC) batteries using reverse osmosis membrane technology.

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OBJECTIVES Industrial water consumption will account for 22% of global water demand by 2030. Industry water conservation is encouraged by rapid corporate growth. Industrial resource usage and pollutant emissions can be reduced via cleaner production methods. Recycling is essential to greener production and the circular economy. Recycling is crucial to achieving the 2030 Sustainable Development Goals. The electric vehicle (EV) sector has propelled battery business growth in recent years, especially in Indonesia. The electric vehicle (EV) sector will benefit from using Nickel Manganese Cobalt (NMC) batteries. **METHODS** The study will use reverse osmosis (RO) membrane filtration to recover water from recovered NMC battery effluent. The experiment will investigate feed solution concentrations, pressures (8, 10, and 12 bar), and temperatures (30, 40, and 50°C). Two factors—permeate flux and metal ion rejection—determine reverse osmosis membrane efficiency. **RESULTS** Li and Na metal rejection was maximum at 30°C and 12 bar, with 94-

96% and 90–93% rejection rates, respectively. Under certain operating conditions, reverse osmosis membrane technology significantly reduced sodium (Na) concentration in NMC battery recycling effluent. **CONCLUSIONS** Thus, wastewater is no longer saline. Reverse osmosis water can be reused for cooling due to its Li and Na concentrations.

KEYWORDS clean water; cleaner production; circular economy; NMC battery; reverse osmosis technology

1. INTRODUCTION

Using secondary sources has become a significant trend in various research aspects, acquiring clean water and recovering valuable metals. In the context of valuable metal recovery, the utilization of secondary sources includes the retrieval of secondary source metals such as geothermal sources or batteries (Jenie et al. 2018; Maulidia et al. 2023; Petrus et al. 2022; Sujoto et al. 2021). Obtaining clean water entails cleaning and repurposing wastewater or effluent for more environmentally sustainable objectives. This practice not only aids in alleviating the burden on natural freshwater supplies but also plays a role in diminishing liquid waste and mitigating environmental consequences.

The rise in water demand can be attributed to factors such as population expansion, economic advancement, and shifts in consumer behaviours. The need for water on a global scale has experienced a significant increase of 600% throughout the last century. This number amounts to an annual increase rate of 1.8%. Based on current data, the yearly growth rate is estimated to be 1%, although it is essential to note that this figure may be overly optimistic (Figure 1). By

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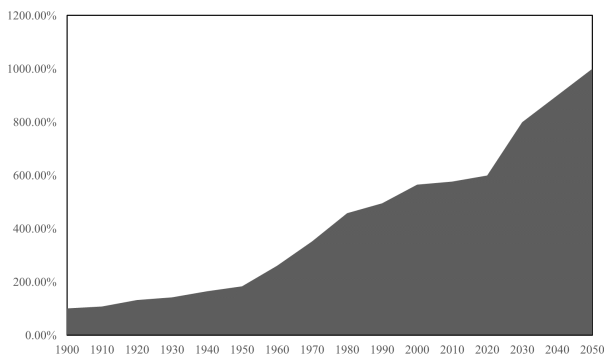


FIGURE 1. Long-term trends in global freshwater utilization (Ritchie and Roser 2017).

TABLE 1. Li and Na ion concentrations in the feed solution.

Feed solution	Li (ppm)	Na (ppm)
1	91,22	2.929
2	73,77	2.445
3	46,53	1.960

2030, it is predicted that there will be an increase in industrial water demand from 800 billion m^3 in 2009 to 1500 m^3 . The industrial sector takes a high portion, reaching 16% of global water demand, and is expected to increase to 22% in 2030 (Vajnhandl and Valh 2014). The increasing water demand in various sectors will impact the water supply. Groundwater is the second largest freshwater resource, fulfilling one-third of global drinking water needs. Climate change has reduced groundwater availability due to rising temperatures and changes in rainfall. Sea level rise also contributes to the decline of groundwater quality (Shirazi and Kargari 2015; Swain et al. 2022). The global water demand is projected to experience substantial growth across all three sectors - industry, residential, and agriculture - during the next two decades. The growth rate in industrial and domestic demand is projected to exceed that of the agricultural market; nonetheless, it is essential to note that agriculture will continue to maintain its status as the largest sector in terms of demand. The increase in non-agricultural demand is projected to surpass the growth rate in the agricultural market.

Declining global water availability and quality encourages preventive actions to avert global water scarcity. One of the objectives outlined in the Sustainable Development Goals (SDGs) for 2030 is to improve water usage efficiency in many sectors, explicitly concerning clean water and adequate sanitation. These goals also aim to ensure the sustainable utilization and provision of freshwater resources to combat water scarcity and significantly decrease the number of individuals affected. In line with implementing the Sustainable Development Goals 2030 program, it is necessary to conserve water supply, especially for industry. Applying a cleaner production concept becomes essential as a water conservation action in line with SDGs (Giannetti et al. 2020).

The cleaner production concept is a strategy to prevent and reduce risks to people and the environment, which is applied to all product life cycles. This strategy aims to avoid large consumption of natural resources and increase the effectiveness of processes to minimize the emissions produced. Recycling is an application of cleaner production that takes a vital role in the circular economy concept. Water recovery from wastewater is one of the alternatives for cleaner production related to water scarcity (de Oliveira Neto et al. 2021).

Meanwhile, greenhouse gas emissions prevention encourages the development of electric vehicles (EVs) as an alternative low-carbon emission technology. This action will significantly increase the need for batteries as one of its components (Veza et al. 2022). Nickel Manganese Cobalt (NMC) batteries are secondary lithium-ion (Li-ion) batteries that are utilized in EVs (Zhang et al. 2018). The NMC battery component consists of a cathode in the form of $LiNi_{0,33}Co_{0,33}Mn_{0,33}O_2$. These metals are non-renewable resources, so the amount is limited and requires a high cost for mining (Primo et al. 2021). Therefore, applying a circular economy in the battery industry is necessary. However, the wastewater generated from recycling is a problem and

requires conservation actions. The objective is to extract water from the wastewater generated during the recycling process of NMC batteries, aligning with the Sustainable Development Goals (SDGs) set for 2030. This initiative is particularly relevant due to the rapid growth of the NMC battery sector.

NMC battery recycled wastewater needs to go through a

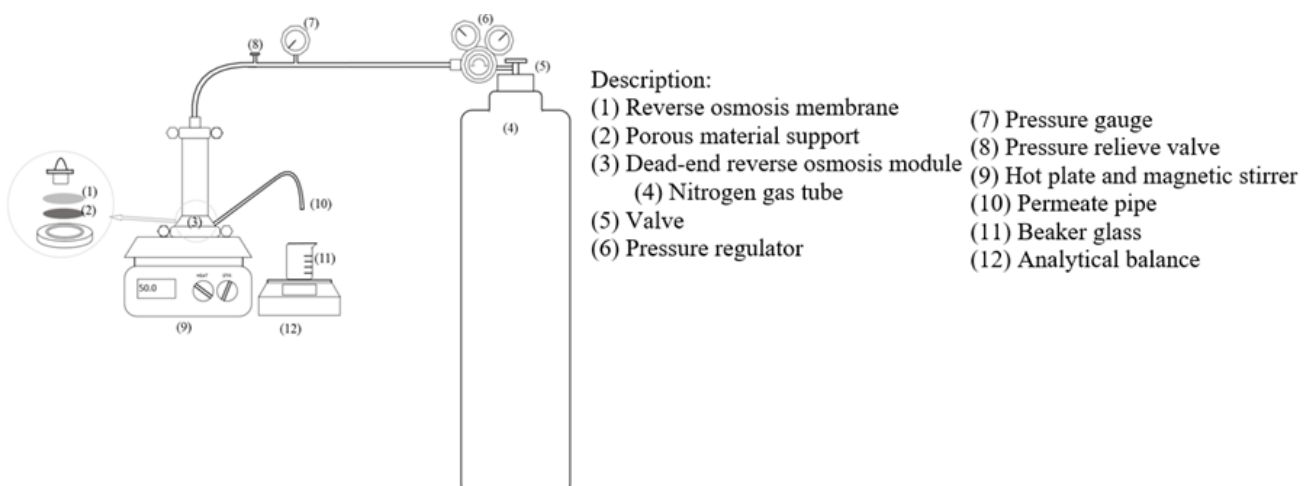


FIGURE 2. Batch reverse osmosis equipment.

treatment process before it can be reused, especially for total dissolved solids (TDS) values and metal levels such as lithium (Li) and sodium (Na). The TDS content in the treated water must be reduced to 100-500 ppm (Merriam et al. 2022). The environmentally safe level of treated water lithium metal is 25 ppm, while sodium has no specific specifications. However, the sodium content must be less than 750 pm when the treated water is reused as process water. Sodium in the treated water will reduce heat transfer performance and cause fouling and corrosion (Kuloğlu et al. 2022).

Reverse osmosis is a solvent transfer phenomenon from a higher-concentration solution to a lower-concentration solution through a semipermeable layer. This phenomenon occurs when the hydrostatic pressure applied to the system exceeds the osmosis pressure. Reverse osmosis separates water from dissolved solids in water purification applications. This process will produce a permeate containing lower dissolved solids and a retentate containing higher ones (Ahuchaogu et al. 2018). A reverse osmosis membrane is a semipermeable layer consisting of a thin polymer layer and porous support. Reverse osmosis membranes have 0.1-1 nm pores and are used in desalination applications (Yang et al. 2019). Reverse osmosis membrane filtration, possibly to recover water from NMC battery recycled wastewater to obtain reusable treated water. The performance of the reverse osmosis membrane is determined by the permeate flux and the percentage of metal ion rejection, which is influenced by operation conditions such as pressure, temperature, and feed concentration (Gedam 2012; Shigidi et al. 2021).

2. RESEARCH METHODOLOGY

2.1 Materials

The present study utilizes the wastewater generated from the recycling of NMC batteries at the Ceramics and Composites Laboratory, a Department of Chemical Engineering component at Universitas Gadjah Mada. The device employed in this study is the SG-RO3-4040 reverse osmosis membrane manufactured by RisingSun Membrane Technology (Beijing) Co., Ltd. In addition, the Energy Conservation and Pollution Prevention Laboratory, affiliated with the Department of Chemical Engineering at Universitas Gadjah Mada, generated the demineralized water.

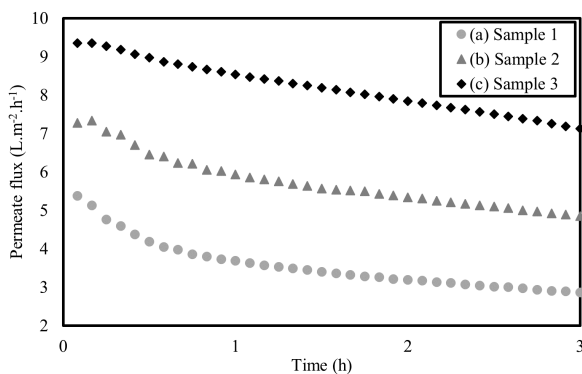


FIGURE 3. Permeate flux versus time (a) Sample 1: Feed 1, T= 30°C, P= 8 bar; (b) Sample 2: Feed 2, T= 30°C, P= 8 bar; (c) Sample 3: Feed 3, T= 30°C, P= 8 bar.

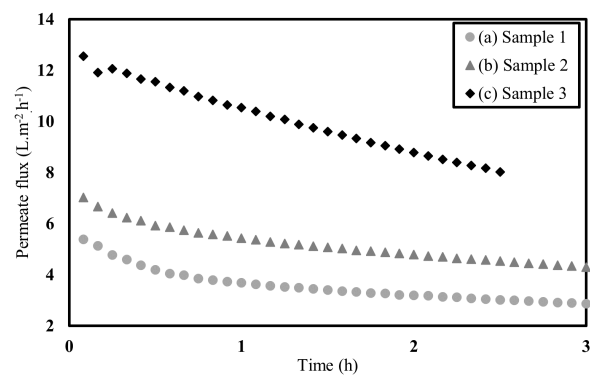


FIGURE 4. Permeate flux versus time (a) Feed 1, T= 30°C, P= 8 bar; (b) Feed 1, T= 30°C, P= 10 bar; (c) Feed 1, T= 30°C, P= 12 bar.

2.2 Feed preparation

The feed solution was prepared by diluting NMC battery recycling wastewater into three different concentration variations. Dilution is a strategic measure aimed at reducing fouling in filtration processes. By adjusting the concentration of the solution through dilution, we can prevent the accumulation of particles or deposits that may cause blockages on the filter or membrane surface. This step also helps maintain the long-term performance of the filter or membrane, making it an effective method for ensuring the sustainability of the filtration process. Samples of wastewater were collected, measuring 50 mL, 37.5 mL, and 25 mL, respectively. These samples were subsequently diluted to a final amount of 1 L. The Li and Na concentrations were assessed using the Inductively Coupled Plasma (ICP) Laboratory for Instrumental Analysis, Department of Chemical Engineering, Universitas Gadjah Mada (Perkin Elmer Optima 8300 ICP-OES system). Table 1 displays the Li and Na ion concentrations in the feed solution.

2.3 Equipment setup

The experiment was carried out using the batch reverse osmosis equipment depicted in Figure 2 over 10 hours. The membrane was incised to yield a surface area of 80 cm² and immersed in demineralized water for several minutes before its utilization. Following the membrane installation in the module, a volume of 100 mL of feed solution was introduced into the membrane module. The filtration process used three different concentration levels: pressure (8, 10, and 12 bar) and temperature (30°C, 40°C, and 50°C). The permeate intro-

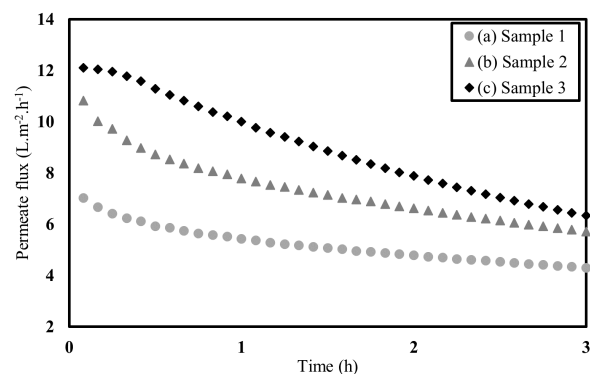


FIGURE 5. Permeate flux versus time a) Feed 1, T= 30°C, P= 10 bar; b) Feed 1, T= 40°C, P= 10 bar; c) Feed 1, T= 50°C, P= 10 bar.

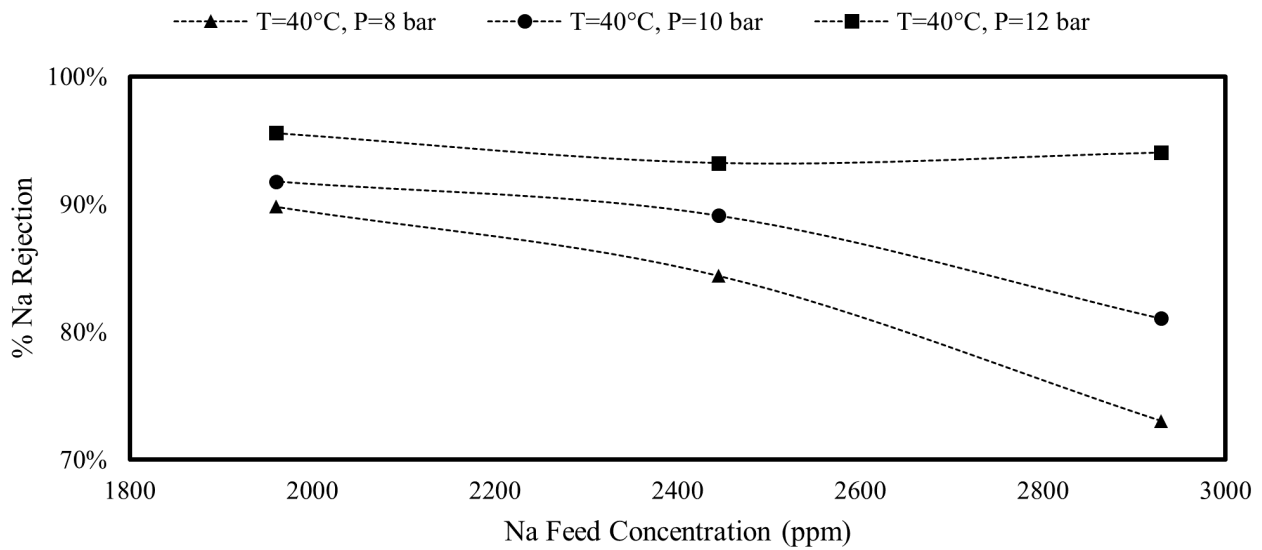
duced into the beaker was subjected to periodic weight measurements and observation at 5-minute intervals. The metal content of the permeate from different variations was analyzed using the Inductive Coupled Plasma (ICP) technique.

2.4 Theoretical model

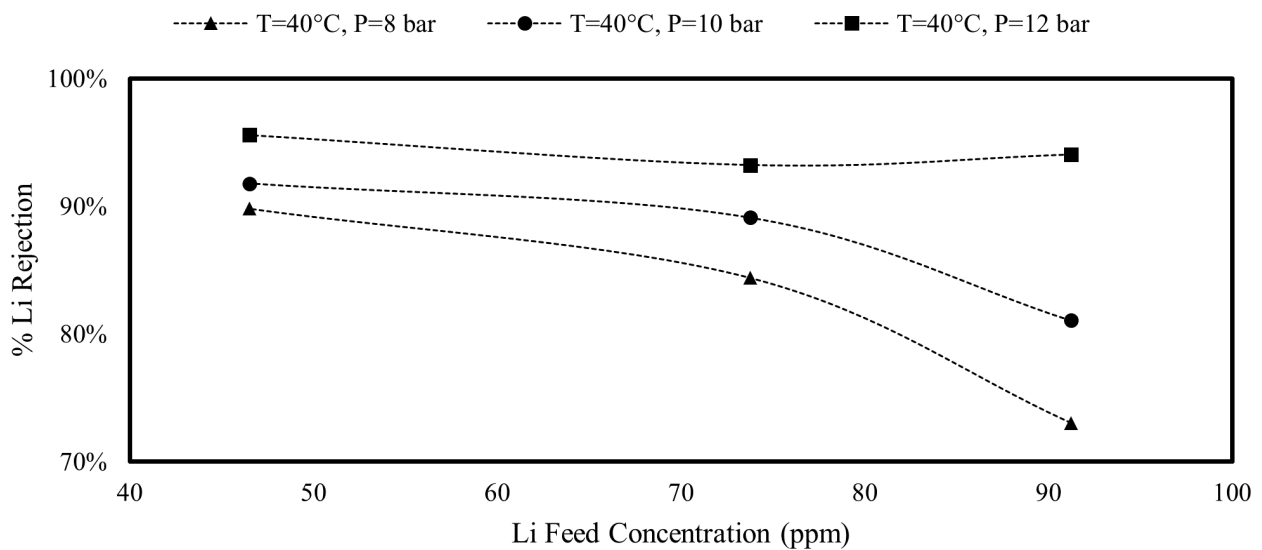
The term "permeate flux" in reverse osmosis (RO) technology pertains to the velocity at which purified water, known as permeate, traverses the RO membrane about its surface area. The standard units of measurement for this quantity are liters per square meter per hour (LMH). Reverse osmosis (RO) is a process in which water is compelled to traverse a semipermeable membrane, selectively permitting the passage of water molecules while effectively preventing the course of most dissolved salts, minerals, and other impurities. The evaluation of the performance and efficiency of a reverse osmosis (RO) system relies on the crucial metric

known as permeate flux. The system's performance is subject to multiple elements, encompassing the feedwater pressure, the reverse osmosis (RO) membrane quality, the temperature, and the attributes of the feedwater, such as its salt concentration and fouling potential.

A greater permeate flux signifies that the reverse osmosis (RO) system is generating filtered water at an accelerated pace, a characteristic that may be advantageous in some contexts. Nevertheless, the operation of the reverse osmosis (RO) system at overly elevated flux levels may result in membrane fouling and a gradual decline in system performance. Hence, it is imperative to optimize the permeate flux while simultaneously preserving the integrity of the membrane in reverse osmosis (RO) technology to provide adequate water filtration. The permeate flow refers to the quantity of permeate obtained during membrane operation, expressed as the volume per unit membrane surface area per unit of time (Sujoto



(a)



(b)

FIGURE 6. Impact of feed concentration on (a) sodium (Na) rejection and (b) lithium (Li) rejection at a fixed temperature of 40°C under diverse pressure and feed concentration conditions.

et al. 2021; Sutijan et al. 2023).

$$J_v = \frac{V}{At} \tag{1}$$

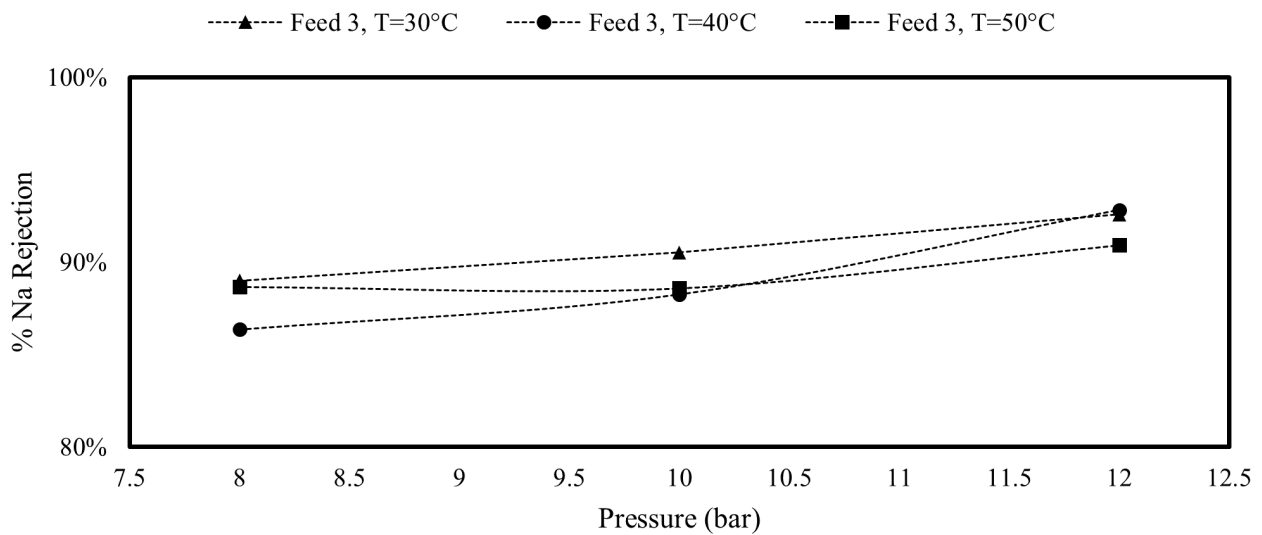
To calculate the permeate flux (J_v) in reverse osmosis technology, you need to take into account the permeate volume (V), the membrane surface area (A), and the time (T) during which the system operates (Equation 1). Permeate flux, often measured in liters per square meter per hour ($L/m^2.hr$), quantifies the rate at which purified water (permeate) flows through the membrane per unit area (Sujoto et al. 2021).

The theoretical framework presented by Hong et al. (1997) is based on the utilization of particle transport and mass conservation equations, with a particular emphasis on developing a polarisation layer on the surface of the membrane. To simplify the intricacy of the equations, Hong et al. (1997) proposed several hypotheses relevant to the present work as well. Equation 2 was derived to describe the progres-

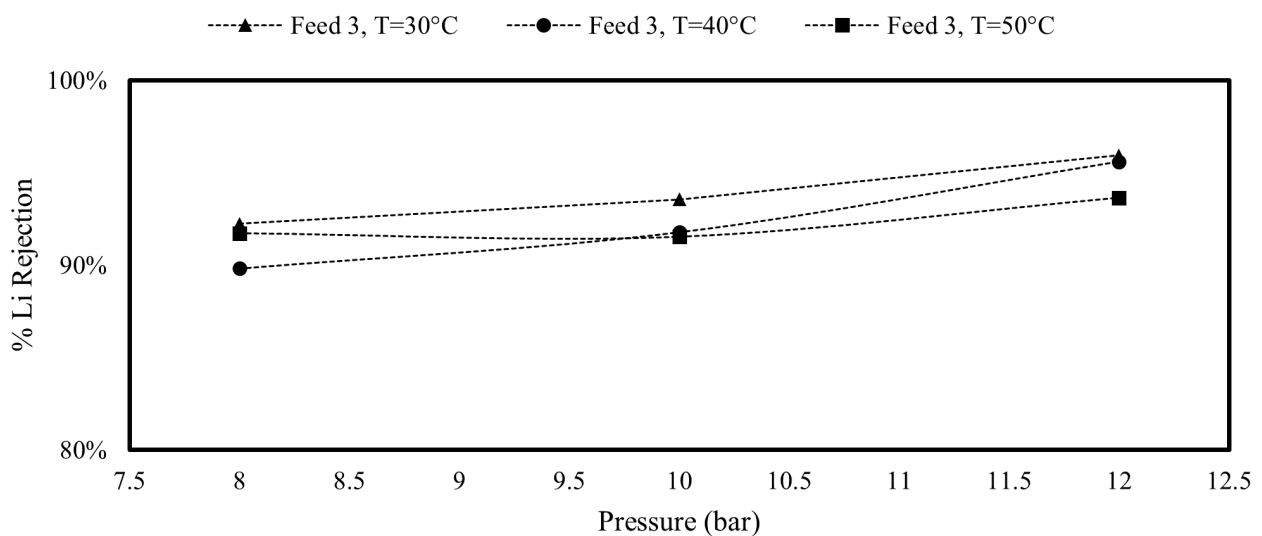
sion of the permeate flux concerning filtration time.

$$J_v = J_0 \left(\frac{3k_B T A_s (\theta_{max}) C_0 TMP}{2\pi a_p^3 D (\mu R_m)^2 \rho_s} \right) \tag{2}$$

J_0 represents the initial permeate flux in meters per second ($m \cdot s^{-1}$), k_B signifies the Boltzmann constant in joules per Kelvin ($J \cdot K^{-1}$), T denotes the temperature in Kelvin (K), $A_s(\theta_{max})$ accounts for neighbouring particle effects in the cake layer using Happel's cell model (-), C_0 stands for the feed particle concentration in kilograms per cubic meter ($kg \cdot m^{-3}$), TMP represents the transmembrane pressure in pascals (Pa), a_p signifies the particle radius in meters (m), R_m represents membrane resistance (m^{-1}), ρ_s represents particle density ($kg \cdot m^{-3}$), μ represents dynamic viscosity of the suspension ($Pa \cdot s$), D represents the diffusion coeff The equation presented below encompasses the subtle dynamics associated with the evolution of permeate flux, thereby estab-



(a)



(b)

FIGURE 7. Influence of operating pressure on (a) sodium (Na) ion rejection and (b) lithium (Li) ion rejection with feed 3 at different temperature settings.

lishing a fundamental comprehension of filtration systems reliant on membrane technology. The diffusion coefficient of silica particles is determined through the Stokes–Einstein relation (Equation 3) (Sujoto et al. 2022).

$$D = \frac{k_B T}{6\pi\mu a_p} \tag{3}$$

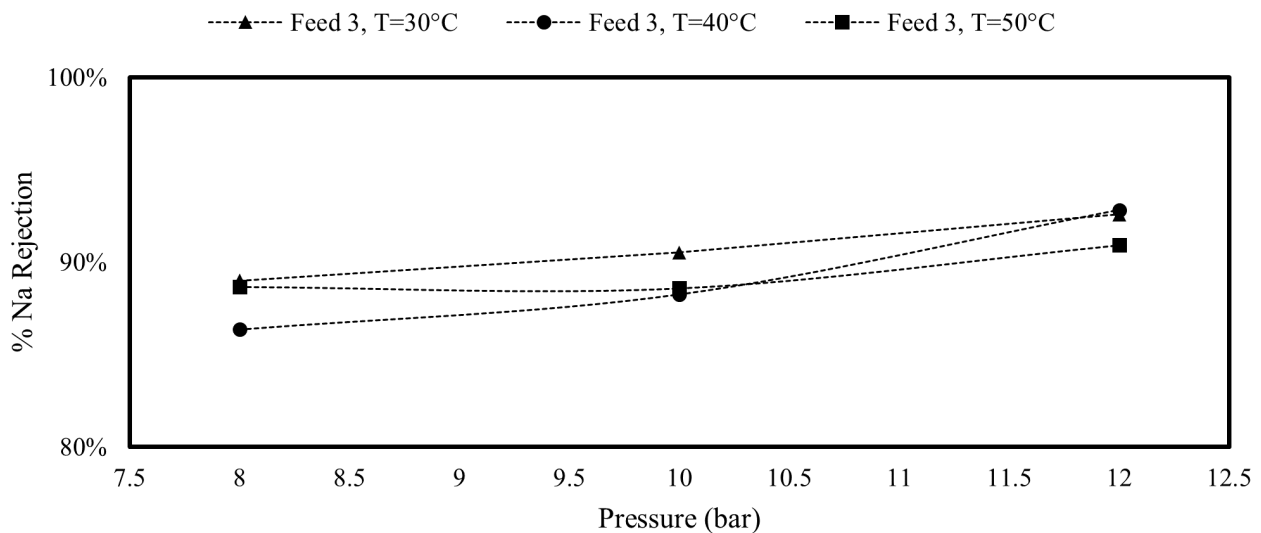
The correction function, represented as $As(\theta_{max})$, is intricately linked to the porosity of the filter cake that forms on the membrane surface. This relationship has been previously explained by Happel (1958) and Happel and Brenner (1983). In the current investigation, Equation 4 was employed to precisely determine the maximum value of As , referred to as $As(\theta_{max})$. This equation incorporates a dimensionless parameter θ , which is contingent upon the porosity n , as illustrated in Equation 5. In their study, Hong et al. (1997) assigned a value of 0.36 to the porosity, denoted as n , representing the minimal theoretical porosity of a stack of spheres.

$$As(\theta_{max}) = \frac{1 + (\frac{2}{3})\theta^5}{1 - (\frac{3}{2})\theta + (\frac{3}{2})\theta^5 - \theta^6} \tag{4}$$

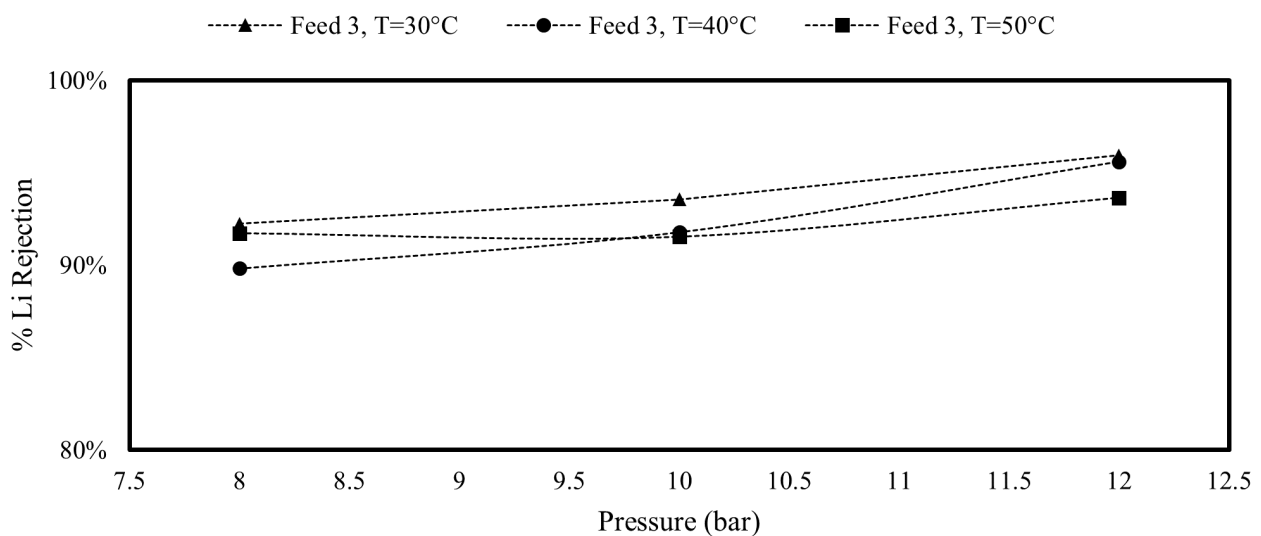
$$\theta = (1 - n)^{\frac{1}{3}} \tag{5}$$

The parameter $R(\%)$ represents the rejection percentage, quantifying the efficiency of removing specific ions or contaminants from the feedwater in membrane technology. This essential parameter gauges the membrane's effectiveness in selectively allowing certain particles or ions to pass through while blocking others, thereby determining the purification capability of the membrane system (Equation 6).

$$R(\%) = \frac{C_{feed} - C_{permeate}}{C_{feed}} \times 100\% \tag{6}$$



(a)



(b)

FIGURE 8. Influence of operating pressure on (a) sodium (Na) ion rejection and (a) lithium (Li) ion rejection with feed 3 at different pressure settings.

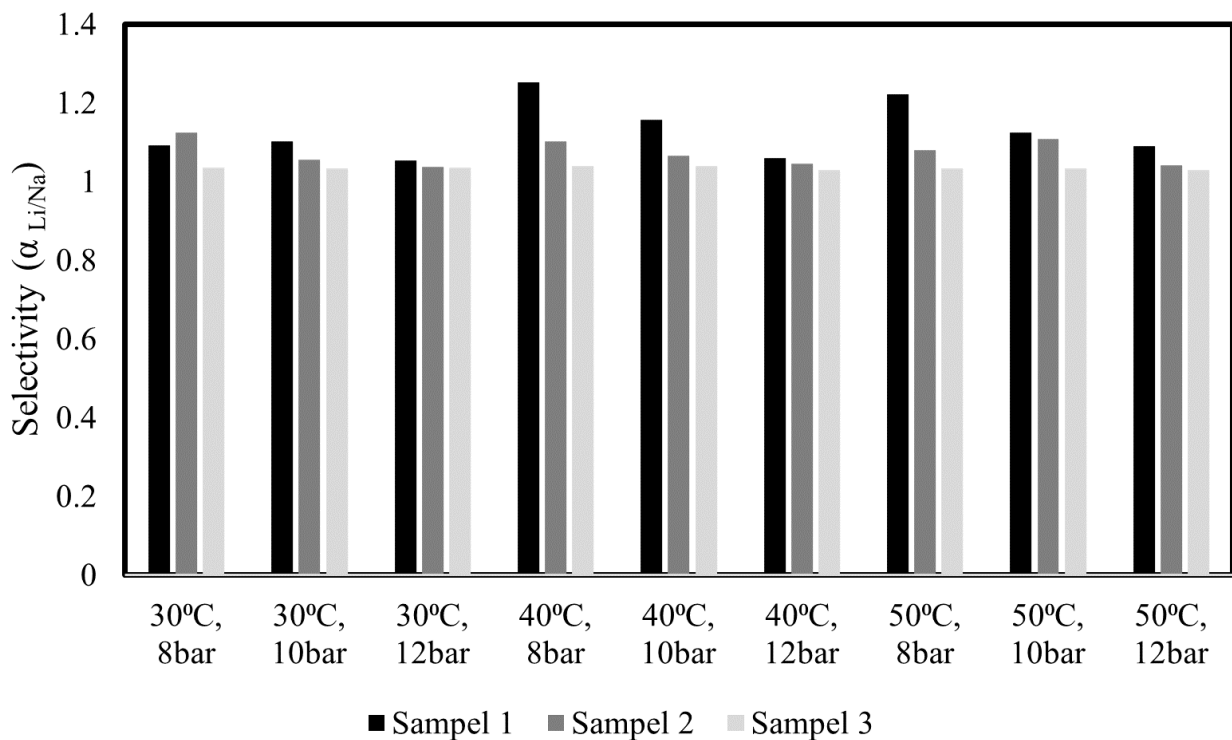


FIGURE 9. The selectivity value of Li/Na separation.

In the realm of membrane technology, the assessment of the effectiveness of the filtration process heavily relies on the concentration of ions in the feedwater, symbolized as C_{feed} . The concentration refers to the original levels of various ions or pollutants in the untreated water supply. During filtration, the membrane exhibits selectivity by permitting the passage of specific ions while keeping others. The representation of the concentration of these ions in the resulting permeate is denoted as $C_{permeate}$. This metric indicates the purified water’s quality, representing the degree to which the membrane effectively eliminates contaminants from the feedwater. The assessment of membrane efficiency and contaminant removal degree is crucial in evaluating the performance of membrane-based purification systems. This assessment may be accurately conducted by comparing the variables C_{feed} and $C_{permeate}$, making this comparison an essential metric. The selectivity (α) in the context of separating lithium ions (Li^+) and sodium ions (Na^+) refers to the membrane’s or separation technology’s ability to separate these ions from a mixture selectively. Selectivity can be calculated using the following equation 7 (Ounissi et al. 2022).

$$\alpha_{Li/Na} = \frac{\%Rejection\ of\ Li^+}{\%Rejection\ of\ Na^+} \quad (7)$$

Here, $\alpha_{Li/Na}$ represents the selectivity of lithium over sodium. This equation provides the ratio between the percentage rejection of lithium ions (%Rejection of Li^+) and the percentage rejection of sodium ions (%Rejection of Na^+). The higher the selectivity value, the better the membrane or separation technology separates lithium from sodium ions in a mixture. However, to calculate this selectivity, you need experimental data or information about the percentage rejection

of lithium ions (%Rejection of Li^+) and the percentage rejection of sodium ions (%Rejection of Na^+). These values are obtained from separation experiments conducted using a specific membrane or technology under typical operational conditions. Using this data, you can calculate the selectivity value using the abovementioned formula.

3. RESULTS AND DISCUSSION

The operating conditions heavily influence the determination of optimal settings for a process. Hence, a series of experiments were conducted to investigate the impact of different factors, including feed concentration, pressure (8, 10, and 12 bar), and temperature (30, 40, and 50°C), on the efficiency of water recovery from NMC battery recycled wastewater using reverse osmosis filtering. The aim was to determine the optimal parameters for this process.

3.1 The effect of operating conditions on membrane permeate flux

The extended duration of operation will result in a reduction in permeate flux, as illustrated in Fig. 3. Concentration polarisation, a phenomenon characterized by the buildup of ions on the surface of the reverse osmosis membrane, is responsible for this occurrence (Dévora-Isiordia et al. 2023; Sujoto et al. 2022; Sutijan et al. 2023). A longer duration of concentration polarisation was observed when utilizing a lower concentration of feed solution. Figure 3a demonstrates a significant reduction in the permeate flux rate for 3 hours. On the other hand, the reduction in permeate flux rate exhibits a higher level of stability when the concentration of the feed solution is reduced, as illustrated in Figure 3b and Figure 3c.

Concentration polarisation is a phenomenon that is also linked to an elevation in resistance to solvent flow (Borhan

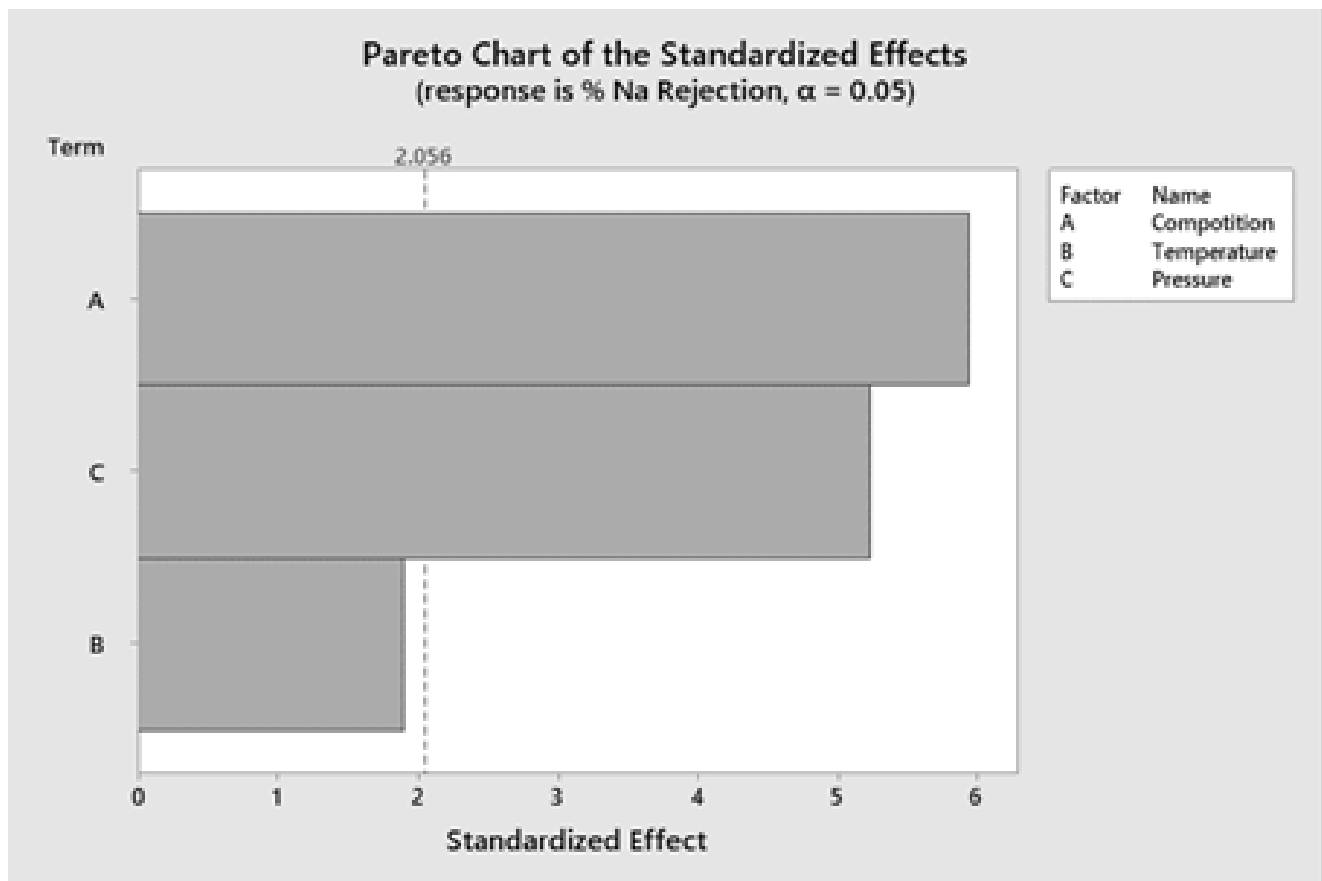


FIGURE 10. Analyzing parameter influence using the Pareto chart.

and Mat Johari 2014). This phenomenon can be attributed to the imbalance in mass transfer between dissolved components in the bulk phase and the surface of the membrane (Dévora-Isiordia et al. 2023). Concentration polarisation on the membrane presents notable obstacles in filtration and water purification procedures. Membrane fouling is observed when a concentration of solutes is close to the membrane surface, decreasing the permeate flow rate through the membrane (Sujoto et al. 2022). The accumulation of solutes has the potential to give rise to the formation of deposits or thin layers on the surface of the membrane, commonly referred to as fouling. Fouling can potentially diminish the efficiency of membranes, elevate operational pressures, and necessitate more frequent cleaning or replacement procedures (Sujoto et al. 2022). Hence, the effective management and control of concentration polarisation play a vital role in designing and operating filtration systems, aiming to achieve optimal and sustainable performance.

To get maximum efficiency of a reverse osmosis membrane, the applied pressure must surpass the osmotic pressure of the solution. The significance of pressure in facilitating solvent transfer and subsequently increasing permeate flux is emphasized in a study by Gedam (2012). As depicted in Figure 4, the influence of an augmented applied pressure on the resultant permeate flux is significantly advantageous. Increased pressure levels directly correlate with a more significant positive impact on the quantity of permeate generated. Nevertheless, it is essential to acknowledge that when the pressure increases, the decrease in permeate flux rate might become more pronounced, especially when compared to lower-pressure situations linked to concentration polarisation.

The observed phenomena can be ascribed to the increased pressure, which leads to an enhanced transfer of solvent and a simultaneous rise in the buildup of ions on the membrane's surface. The results of this study underscore the significant importance of pressure in maximizing the efficiency of reverse osmosis systems. They also draw attention to the need to carefully manage the interplay between pressure and other operational factors to attain the intended results in water purification and separation procedures.

The impact of temperature on the efficacy of filtration systems, namely in reverse osmosis, is of utmost significance. The alteration of solution viscosity is significantly influenced by temperature. There is ample evidence to support the notion that an increase in temperature leads to a decrease in the viscosity of a solution. The observed drop in viscosity significantly impacts the rate of solvent diffusion, resulting in a notable increase in permeate flux. As illustrated in Figure 6, there is a clear correlation between temperature and permeate flux. This pattern remains consistent across different feed modifications, as demonstrated in Figure 5. The results show a positive correlation between temperature elevation and an augmented permeate flux. The observed positive association can be ascribed to higher temperatures promoting increased solvent diffusion over the membrane, leading to a higher permeate flux rate.

Nevertheless, it is crucial to acknowledge that although elevated temperatures can enhance the diffusion of solvents and the flux of permeation, they can also intensify the pace at which the flux decreases, as evidenced by the findings presented in Figure 5. The observed phenomena can be ascribed to the increased concentration of ions on the surface of the

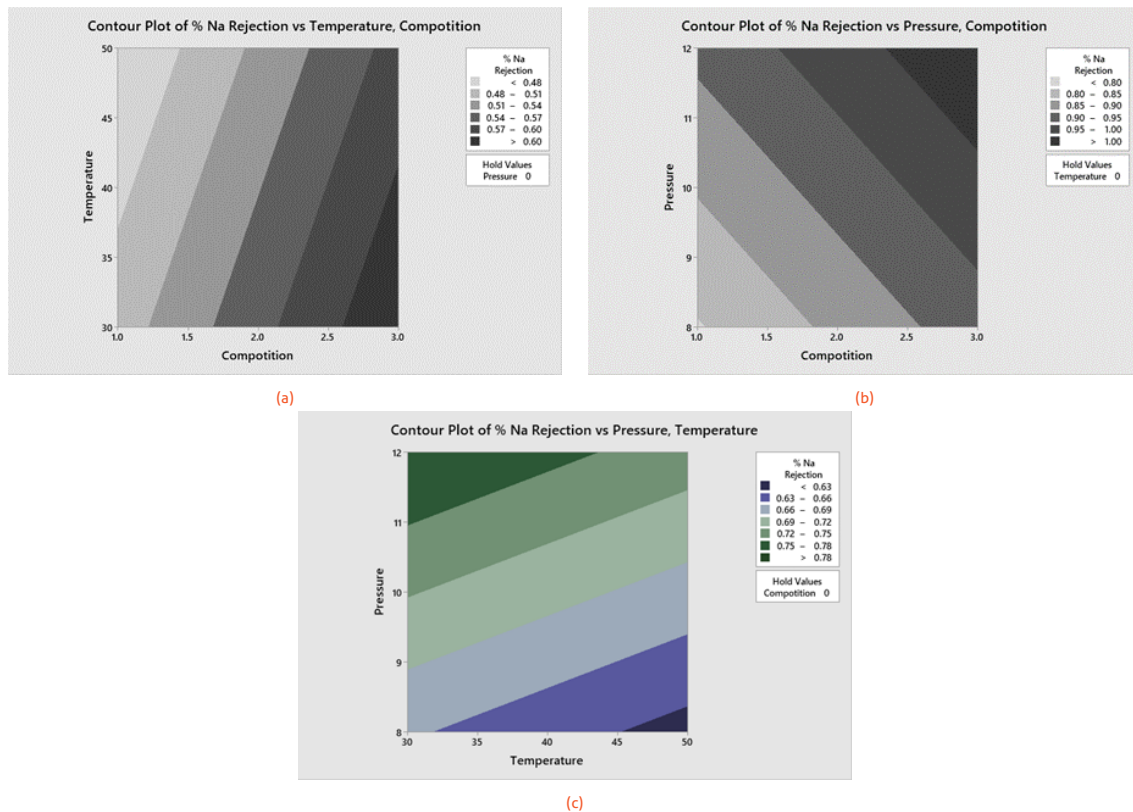


FIGURE 11. Analyzing parameter influence using the contour plot (a) temperature vs composition (b) pressure vs composition (c) pressure vs temperature.

membrane, a process that occurs more rapidly at higher temperatures. Therefore, managing temperature is crucial to optimize filtration processes and attain higher permeate flux rates. It is vital to strike a delicate equilibrium between the benefits of enhanced flux and the potential difficulties that may arise from increased ion concentration at the membrane surface. Comprehending the complicated temperature impacts is crucial for precisely adjusting and optimizing reverse osmosis systems across many applications, ranging from water purification to industrial separation processes.

3.2 Effect of operation conditions on membrane rejection

The concentration of ions in the feed solution is a critical component that plays a crucial role in influencing the outcome of reverse osmosis procedures. The concentration of the feed solution significantly influences the number of ions present in the solution. The augmentation of ion concentrations, such as sodium (Na) and lithium (Li), in the feed solution has been observed to have a paradoxical impact on rejecting these metallic elements, as demonstrated in Figure 6. This phenomenon can be elucidated by considering the concept of concentration polarisation. When the concentration of ions in the feed solution is high, concentration polarisation becomes more noticeable, increasing the resistance to the solvent flow. A high concentration of ions in a solution poses a more incredible difficulty for the solvent to pass through the membrane, primarily due to an elevated resistance to solvent flow. As a result, more ions from the solution traverse the membrane. Paradoxically, this phenomenon leads to a decrease in the membrane’s ability to reject these ions. The complex interaction of feed concentration, concentration polarisation, and rejection rates is a crucial factor to be taken into account in reverse osmosis operations. To

enhance the efficiency and selectivity of the separation process, researchers and engineers must navigate the interplay of these components carefully. It is crucial to have a comprehensive understanding of the influence of feed concentration fluctuations on rejection rates and concentration polarisation to efficiently design and operate reverse osmosis systems for various applications, including but not limited to desalination and metal recovery.

Understanding and enhancing the operations of reverse osmosis systems necessitates a comprehensive comprehension of the crucial correlation between operating pressure and the rejection of metal ions. As depicted in Figure 7, it is apparent that there is a significant enhancement in metal ion rejection as operating pressures increase. The elucidation of the mechanism underlying this phenomenon can be achieved by examining the fundamental principles of reverse osmosis. The augmentation of the operating pressure yields a dual consequence that directly influences the rejection of metal ions (Sutijan et al. 2023). First and foremost, it amplifies the propulsive effect of the solvent, commonly water molecules, across the membrane. The heightened driving force enhances the efficiency of water molecule movement across the membrane, increasing the solvent transfer rate.

Furthermore, increasing the operating pressure reduces the osmotic pressure of the solution (Sutijan et al. 2023). When the externally applied pressure surpasses the osmotic pressure of the solution, it successfully counteracts the inherent osmotic inclination for solvent molecules to move from an area of lower solute concentration to an area of higher solute concentration. The use of pressure enhances the solvent transfer rate, hence intensifying the counteractive effect. The retention of metal ions on the surface of the reverse osmosis membrane is enhanced when the operating

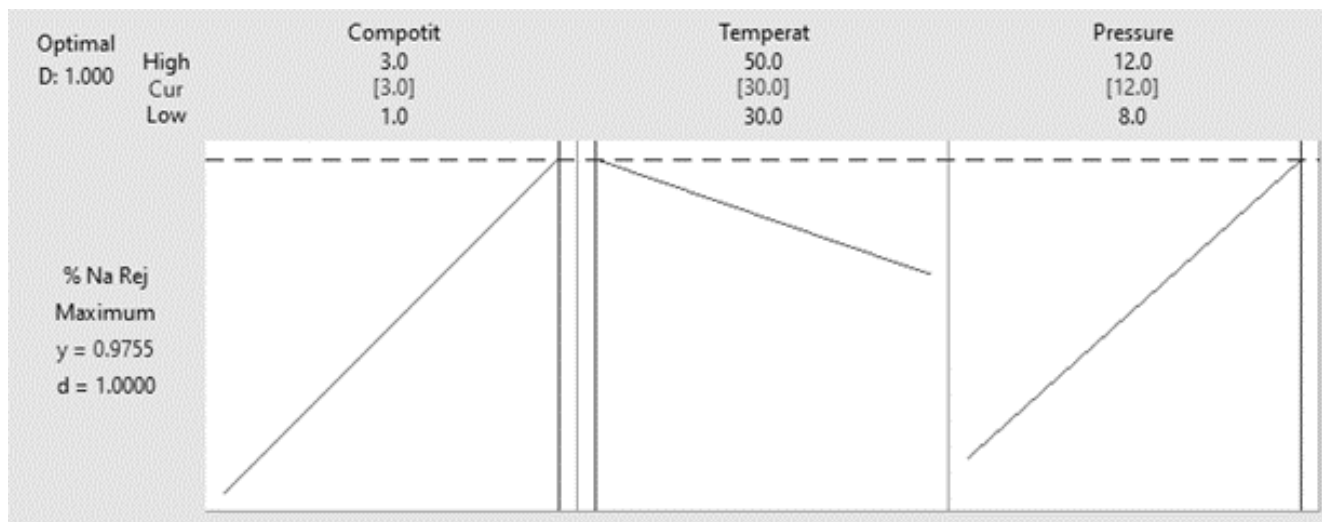


FIGURE 12. Analyzing the best conditions using optimizer in Minitab.

pressure increases, leading to a higher solvent transfer rate. The deposition of ions on the surface of the membrane results in an increased ability to reject metal ions, hence improving the separation efficiency. Comprehending the complex interplay among operating pressure, solvent transfer, and metal ion rejection is paramount in optimizing reverse osmosis systems. Engineers and researchers must meticulously evaluate and modify the operational pressure to attain the targeted degree of metal ion rejection while upholding the filtration process's overall performance and efficiency.

The correlation between temperature and the rejection of metal ions in reverse osmosis systems is a captivating phenomenon that merits additional investigation. Figure 8 also provides a clear visual representation of the negative relationship between temperature and the rejection of metal ions, thereby elucidating the complex dynamics involved in this phenomenon. This link can be understood by examining the underlying mechanisms activated by temperature fluctuations. As the temperature rises, several subsequent alterations occur within the system. Initially, the viscosity of the solution undergoes a significant decrease. The reduction in viscosity subsequently leads to an elevation in the solubility of the solute present in the feed. As the solubility of the solute increases, its ability to interact with the membrane surface is enhanced (Mustika et al. 2022; Sujoto et al. 2022). An essential outcome of these temperature-induced alterations is the augmentation of water permeability across the membrane. As the viscosity reduces, the solvent, usually water, exhibits enhanced mobility within the porous structure of the membrane, resulting in an increased rate of water permeation (Sujoto et al. 2022).

Moreover, the enhanced solubility of the solute in the feed solution increases the diffusion of the solute across the membrane. This phenomenon implies that an elevated temperature improves the mobility of solute particles across the membrane, increasing the probability of metal ions traversing through (Mustika et al. 2022). The cumulative impacts of these temperature-induced modifications ultimately lead to a decrease in the rejection rate of metal ions during the reverse osmosis process. Elevated temperatures have been observed to augment specific facets of membrane permeability. However, it impairs the system's capacity to reject

metal ions efficiently. Researchers and engineers must carefully consider temperature conditions during the design and operation of reverse osmosis systems, especially when the rejection of metal ions is a crucial performance criterion. Achieving the appropriate separation efficiency and sustaining overall system performance necessitates carefully considering and optimizing temperature, operating pressure, and other pertinent variables.

The findings consistently demonstrate a pattern in the percentages of lithium (Li) and sodium (Na) metal rejection under various experimental circumstances. The observed pattern stays consistent, exhibiting a notable resemblance in the reaction of lithium (Li) and sodium (Na) metals to changes in critical operational factors such as pressure, temperature, and feed concentration. When considering pressure fluctuations, it becomes apparent that increasing the operating pressure results in higher rejection percentages for both lithium (Li) and sodium (Na) metals. The adherence above to the fundamental principles of reverse osmosis underscores the role of elevated pressure in augmenting the rates of solvent transfer and successfully mitigating osmotic pressure, thus reinforcing the rejection of metal ions. Similarly, when temperature changes, a synchronized trend becomes apparent. There is a continuous correlation between elevated temperatures and decreased rejection rates for both lithium (Li) and sodium (Na) metals. This observation reflects the complex interaction of solute solubility, solvent permeation, and solute diffusion through the membrane, which all influence the rejection of metal ions. In addition, there is a continuous parallel relationship between the rejection percentages of Li and Na metals and the varied concentrations of the feed solution. Specifically, greater concentrations in the feed solution are associated with reduced rejection percentages. The occurrence above highlights the significance of concentration polarisation, which becomes more pronounced as solute concentrations increase, decreasing rejection efficiency. The constant patterns observed in the rejection behaviour of metals offer valuable insights for enhancing the efficiency of reverse osmosis operations, particularly in situations where the separation and recovery of precious lithium (Li) and sodium (Na) metals are involved. Comprehending how modifications in operational parameters affect rejection percent-

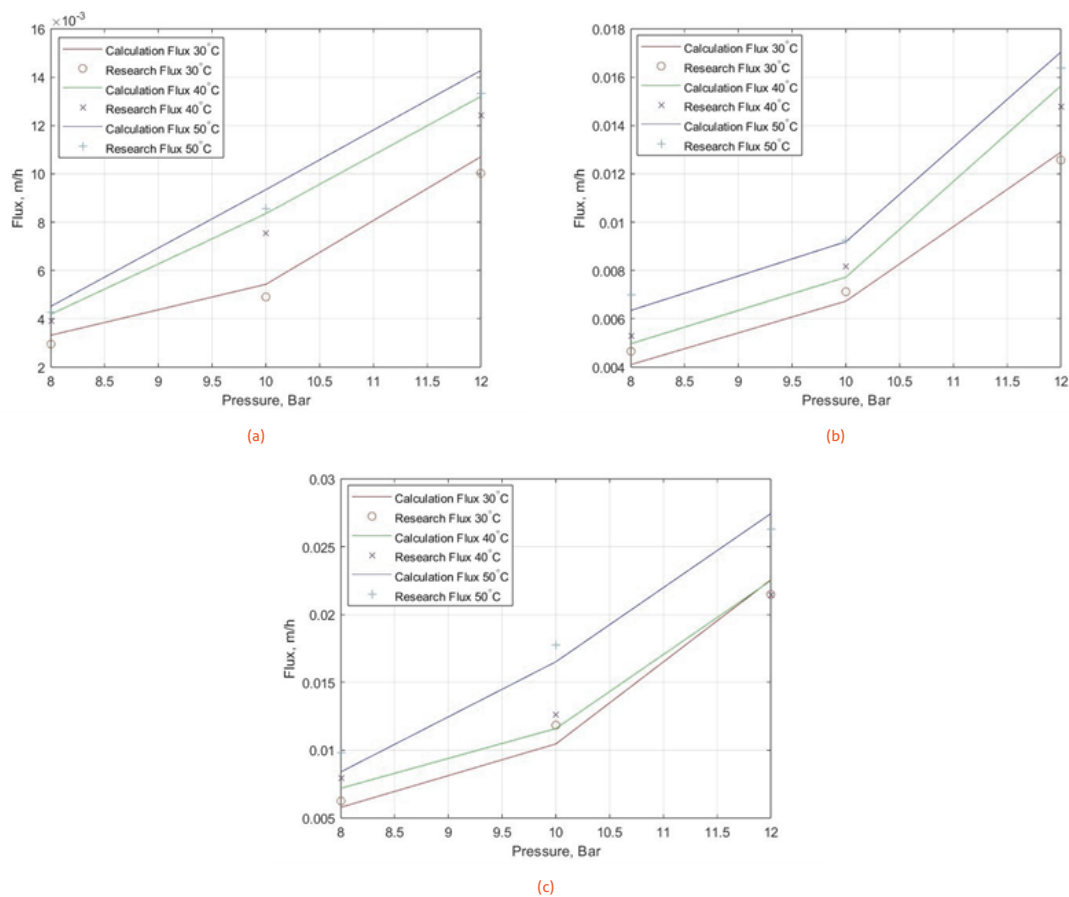


FIGURE 13. Influence of temperature and pressure on flux: model-based analysis (a) Sample 1; (b) Sample 2; (c) Sample 3.

ages allows for educated decision-making, guaranteeing the attainment of desired levels of metal ion separation while considering practical limitations and efficiency factors.

The concept of recovering freshwater from the provided data relates to the technology’s ability to separate clean water from a mixture contaminated with lithium (Li⁺) and sodium (Na⁺). In this context, the concept of selectivity is crucial. Firstly, let’s interpret the data in the context of clean water recovery. Let’s assume that the desired clean water has the lowest possible sodium concentration, meaning high selectivity for sodium is necessary to ensure the recovered water has low sodium content, as shown in Figure 9.

In Figure 9, selectivity values greater than 1 indicate that the separation technology strongly prefers lithium over sodium. In other words, the membrane or technology efficiently separates lithium from sodium. This statement means that the separated water has a low sodium content, approaching zero, which is highly desirable in clean water recovery. However, if selectivity values approach one or even fall below one, it indicates that the separation technology slightly prefers lithium and sodium. In such cases, although clean water is successfully separated, the recovered clean water might have a higher sodium content. Therefore, for clean water recovery applications, high selectivity values for sodium are crucial. Water with low sodium content is essential for human health and environmental sustainability. In order to optimize the recovery of clean water, it is imperative to identify and deploy separation methods that exhibit a high degree of selectivity towards salt. Through the comprehension and examination of selectivity values, researchers

and engineers can make informed decisions in selecting the most appropriate separation method to attain optimal quality clean water.

3.3 Assessment of best operation parameters

Evaluating the most favorable operational parameters provides significant findings regarding the maximum attainable rejection rates for the membrane filtration procedure. In industrial water treatment, it is pertinent to observe that prevailing rules frequently lack severe thresholds for lithium (Li) concentrations. Upon conducting a detailed analysis, it becomes apparent that the concentrations of Li obtained through reverse osmosis are far lower than the established threshold of 25 parts per million (ppm). This observation highlights the treated water’s environmental safety, as the Li levels stay below acceptable limits. Based on data from the United States Geological Survey (USGS), it can be determined that the standard for freshwater quality typically requires that the concentration of dissolved salts remains below the threshold of 1000 ppm (parts per million) (United States Geological Survey (USGS) 2013). Therefore, it can be stated that all the water quality outcomes generated within this research conform to the established freshwater standards. A pivotal aspect of this study lies in identifying the operational conditions that yield the lowest concentration of dissolved salts, which reflects the optimal conditions in terms of water quality.

Due to the notable disparity in sodium and lithium concentrations, the focus of the Response Surface Methodology (RSM) analysis will be meticulously directed towards sodium

ions. This strategic approach is motivated by the significantly higher prevalence of sodium within the system, requiring a detailed exploration of its intricate interactions and behaviours. By delving deep into the intricacies of sodium ions, the RSM analysis aims to unveil nuanced patterns and optimize various parameters associated with their presence. This deliberate concentration of sodium aligns with the prevailing composition dynamics. It facilitates a more nuanced understanding of the complex interplay within the system, thereby enhancing the overall precision and reliability of the analytical outcomes. Response Surface Methodology (RSM) is a statistical method to determine the ideal conditions. RSM is a methodology that enables researchers to systematically design experiments, analyze resulting data, and construct mathematical models that comprehend the intricate relationships among various factors influencing water quality. With the assistance of RSM, we can fine-tune operational conditions to meet or even surpass established freshwater standards while minimizing the concentration of dissolved salts. Thus, RSM is invaluable in conducting this research to achieve optimal outcomes in line with freshwater standards.

The findings of the Response Surface Methodology (RSM) investigation have yielded several graphical representations, the initial being the Pareto chart (Figure 10). The chart is a visual representation that illustrates the relative importance of different factors within the investigated system. The Pareto chart demonstrates that composition is the parameter with the most significant impact among those studied. This phenomenon indicates that alterations in composition have a considerable effect on the behaviour of the system. After considering the composition, it becomes evident that pressure is the subsequent influential parameter, indicating its substantial impact on the observed results.

On the other hand, it has been observed that temperature exerts a comparatively lesser influence when compared to the other variables. Although temperature does have an impact, its effect is relatively minor. In brief, the Pareto chart derived from the RSM analysis presents a distinct ranking of parameter influence, with composition being identified as the most significant factor, followed by pressure. At the same time, the temperature is found to have a relatively minor effect on the system's behaviour. This visual depiction facilitates the direction of subsequent inquiries and enhancement endeavours within the examined system.

The contour plots generated from the Response Surface Methodology (RSM) analysis provide insights into the interrelationships between the primary variables: temperature, feed composition, pressure, and the percentage of Sodium (Na) rejection. These interactions are visually shown in Figure 11. The data shown in Figure 11a demonstrates a positive correlation between lower feed concentration and lower temperature and increased Sodium (Na) rejection rates. This phenomenon implies that the simultaneous application of reduced feed concentration and lower temperature can increase Sodium (Na) removal. In contrast, it can be observed from Figure 11b that elevated pressure and reduced feed concentration result in heightened levels of Sodium (Na) rejection. The findings suggest that the effectiveness of the process in decreasing Sodium (Na) can be significantly improved while operating under high-pressure circumstances and low feed concentration.

Additionally, it can be observed from Figure 11c that decreased temperature and increased pressure lead to elevated levels of Sodium (Na) rejection. Based on this discovery, it can be inferred that manipulating temperature and pressure can effectively augment the degree of Sodium (Na) reduction in this particular procedure. Researchers have devised a mathematical expression called Equation 8 to enhance understanding of the phenomenon using the principles of Response Surface Methodology (RSM). The equation is vital in establishing the relationship between the rejection % and the many parameters being considered. Equation 8 is to establish a mathematical model that clearly understands the complex correlation between the rejection % and the specified vital parameters. This equation not only improves our comprehension of the fundamental phenomenon but also offers a mechanism for predictive modelling, optimization, and more informed decision-making within the framework of the investigated system.

$$\begin{aligned} \text{Na Rejection} = & 0.498 + 0.065.\text{Composition} \\ & - 0.0022.\text{Temperature} + 0.0291.\text{Pressure} \end{aligned} \quad (8)$$

The subsequent phase is optimization via an optimization system, aided by software tools like Minitab, based on the observations and analyses derived from Response Surface Methodology (RSM), encompassing graphs and equations, as shown in Figure 12. The optimal conditions determined in this study were found in sample condition 3, where the operating temperature and pressure were set at 30°C and 12 bar, respectively. Under these specific parameters, the system exhibited a rejection rate exceeding 97%. This finding underscores the significance of precise temperature and pressure control in reverse osmosis processes, emphasizing the need for meticulous adjustments to achieve optimal outcomes in industrial applications.

In the given scenario, Minitab is a beneficial tool for developing and executing further tests while optimizing the factors impacting the intended answer. Utilizing the optimization tools provided by Minitab makes it possible to ascertain the most favourable amalgamation of parameter values previously identified within the response surface methodology (RSM) equation. The objective is to attain the system's targeted rejection rate or outcome by minimizing or enhancing the corresponding response. The optimization process may entail employing optimization algorithms offered by Minitab, including Response Surface Methodology (RSM) experimental design approaches, operations research methodologies, or other applicable algorithms. Therefore, Minitab is crucial for planning, optimizing, and monitoring processes. It aids in attaining optimal results by utilizing the insights obtained from the previous response surface methodology (RSM) analysis.

3.4 Model Simulation

The built model underwent testing in simulated industrial environments involving three various pressure levels and three varied temperature suspensions. These conditions were utilized in dead-end tests. The examination of several factors has indicated that fluctuations in concentration, temperature, and pressure inside the reverse osmosis dead-end system substantially influence the process of freshwater re-

TABLE 2. Table of calculated parameters using the existing model.

Parameter	Sample 1	Sample 2	Sample 3
a_p [m]	252.6695	251.6945	253.1427
R_m [m^{-1}]	256.6803	246.2300	232.1899

covery. Therefore, developing a precise model that can accurately estimate water flux is crucial. During the preceding discourse, a qualitative analysis was conducted on multiple parameters to comprehend the impact of concentration, temperature, and pressure changes. Based on the findings of this qualitative research, it can be inferred that elevating pressure and operating temperature have the potential to augment freshwater flux while concurrently diminishing rejection rates. Given the disparity in particle size between sodium and lithium, the simulation model will prioritize the characterization and analysis of sodium ions. This decision stems from the distinct physical properties associated with sodium particles, necessitating a focused approach in the simulation process. By concentrating on sodium ions, the study aims to gain in-depth insights into their behaviour and interactions within the system, contributing to a comprehensive understanding of the overall process dynamics. This section delves into the study's quantitative analysis, as depicted in Figure 13, utilizing equations 2 through 5.

Figure 13 depicts the juxtaposition of the computed flux values and those derived from field observations across three distinct scenarios. The comparisons above demonstrate a noteworthy resemblance between the model's projected flux values and those directly measured in the field. The findings of this study suggest that the model utilized in the research is resilient and adept at accurately representing the events being examined. The observed similarity between the flux values obtained through calculations and those obtained through field measurements indicates a significant degree of precision in the model's ability to forecast the system's performance across different operational scenarios. The above-mentioned observation suggests that the utilized model is dependable for comprehending and predicting the system's reaction to parameter changes such as concentration, temperature, and pressure. Therefore, this discovery confirms the appropriateness of the model, instilling assurance that it functions as a reliable instrument for evaluating and strategizing reverse osmosis procedures in various scenarios. The practical implications of this alignment are significant, as it suggests that this model can be utilized as a prediction tool for optimizing operational conditions in industrial settings. This functionality facilitates enhanced operational effectiveness and more intelligent decision-making in administering exceedingly essential water resources.

Quantitative analysis plays a pivotal role in assessing the impact of the model on parameter calculation in the reverse osmosis dead-end process. This particular stage holds significant importance for parameters not directly examined in the study and cannot be ascertained through qualitative methodologies. Employing empirical data, doing qualitative assessments, and referencing known mathematical formulas makes it feasible to determine specific constants and parameters, as demonstrated in Table 2.

The parameter values for a_p (particle radius) and R_m

(membrane resistance) for three distinct samples, namely Sample 1, Sample 2, and Sample 3, are presented in Table 3. The measured values (in meters) for Sample 1, Sample 2, and Sample 3 are 252.6695, 251.6945, and 253.1427, respectively. In contrast, the R_m values (expressed in m^{-1}) corresponding to Sample 1, Sample 2, and Sample 3 are 256.6803, 246.2300, and 232.1899, respectively. The particle sizes in samples 1, 2, and 3 are nearly identical. The observation that Sample 1 has the highest R_m value, followed by Sample 2 and Sample 3, indicates that fouling phenomena tend to develop at a slower rate with lower salt concentration in the feed. This phenomenon leads to a faster flux. Examining these variations in values can yield significant insights about the properties of the membranes employed in this investigation.

4. CONCLUSIONS

The interplay of pressure, temperature, and feed concentration significantly influences the effectiveness of reverse osmosis membranes in the delicate process of water recovery from NMC battery-recycled wastewater. This research extensively examined the variables above, ultimately identifying the precise circumstances that result in the most favourable results. The comprehensive investigation of operational parameters revealed an intriguing phenomenon: when the system was operated at a temperature of 30°C and a pressure of 12 bar, the percentages of rejected Li and Na ions exhibited a significant increase, with Li metal achieving an impressive rejection rate of 94-96% and Na metal achieving a rejection rate of 90-93%. The results above indicate not only elevated rates of rejection but also the effectiveness of the reverse osmosis membrane in substantially decreasing sodium concentration in the recovered wastewater of NMC batteries. The importance of this revelation extends beyond fundamental statistical analysis. The reverse osmosis membrane has effectively increased the rejection rates, resulting in a significant transformation of the NMC battery recycled wastewater from a saline state to a condition that can no longer be categorized as saline. The transition above holds significant practical ramifications, particularly within the domain of water reuse. The permeate, characterized by reduced Li and Na, assumes significance as a valuable resource. The prospective applications of this technology encompass a wide range of industrial processes, wherein the utilization of cooling water is among numerous feasible applications. The transition from wastewater to a reusable resource highlights these discoveries significance in sustainable water management. Moreover, the research utilized sophisticated methodologies, such as Response Surface Methodology (RSM) analysis, to investigate the best settings. Condition 3 emerged as the exemplification of efficiency, showcasing an outstanding rejection rate above 97%. The thorough examination highlights the strength and reliability of the results, offering a clear comprehension of the complex mechanisms involved in the reverse osmosis procedure. Furthermore, it is essential to highlight that the simulation model utilized in this research effectively replicated the actual results seen in the real world. The unity between the simulation and reality not only serves to authenticate the precision of the model but also establishes it as a standard against which future research endeavours can be measured. The study's findings confirm the importance

of the model in predicting and elucidating the observed occurrences, establishing it as an essential instrument for investigating the complexities of mass transfer in the reverse osmosis dead-end system. The results above jointly emphasize the efficacy of the reverse osmosis technique in treating NMC battery-recycled wastewater and the prospects for additional investigations, advancements, and environmentally friendly water treatment and reuse approaches.

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