

Nutrient recovery and hydrogen production from wastewater by electrodialysis process: Optimization using response surface methodology

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ABSTRACT

The increasing demand for energy, water, and nutrient management has attained interest in sustainable wastewater treatment processes. The electrodialysis (ED) process provides sustainable water and energy management thanks to both nutrient recovery efficiency and hydrogen production. In this study, the ED process has been investigated, focusing on the impact of current density, number of ion-exchange membranes, initial conductivity, and the pH of both diluate and concentrate solutions. The individual and combined effects of these parameters on ED performance were analyzed. At the same time, nutrient recovery efficiency, hydrogen production, and energy consumption values were modeled using Response Surface Methodology (RSM), where correlation equations were created and optimum conditions were determined. When the analysis results were compared with the model results, hydrogen production was determined with 95.6 % accuracy, nutrient recovery efficiency with 98.7 % accuracy, and energy consumption with 98.8 % accuracy. It was observed that the generated correlation equations successfully predicted the system operations and performance parameters. Through RSM optimization, energy consumption was reduced by 24 % in 70 % less time. 25.8 % less energy consumption was obtained for 1 mL of hydrogen production. In this way, energy and time efficiencies were achieved for better sustainability of the wastewater treatment and desalination processes.

1. Introduction

The search for alternative resources has intensified in response to the escalating demand for energy, water, and fertilizer in agricultural food production. This has brought energy conservation to the forefront, particularly in the context of water and nutrient recovery from wastewater. Domestic wastewater contains substantial amounts of nutrients such as nitrogen and phosphorus which have potential applications as fertilizers. Consequently, recent research has focused on the recovery of these nutrients from wastewater and agricultural food waste, investigating both recovery and treatment methods [1]. Electrodialysis is a process that uses an electric field to separate ions from a solution. It is particularly effective in removing nitrates and phosphates from wastewater. The applicability of the electrodialysis system on hydrogen production efficiencies with nutrient recovery is being investigated. In the studies conducted, it has been reported that it has an energy consumption ranging from 0.4 to 25 kWh/m³ [2], and consumption can be reduced by 40 % with various strategies [3]. It has been reported that

maximum hydrogen production is obtained at values varying up to 118.8 mg H₂/h kg [4]. To prevent this variability, optimization is required in system design and operational parameters [5].

Most domestic wastewater treatment plants have been designed for carbon, nitrogen, and phosphorus removal using nitrification and denitrification, aerobic and anaerobic biological processes; however, these processes do not provide nutrient recovery. Nutrient recovery can provide a significant advantage regarding environmental sustainability and resource efficiency. Nutrient recovery from domestic wastewater often involves technologies with significant capital investment and operational expenses. Each process has its own advantages and disadvantages. Although chemical precipitation is considered an efficient method, especially when lime-based precipitation is used, the sludge volume can reach up to approximately 35 % v/v, which creates additional costs and environmental difficulties for sludge management [6–8]. This problem reveals that the chemical precipitation method is a limiting factor in terms of sustainability.

Although ion exchange processes effectively remove nitrates,

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phosphates, and other ions from wastewater, the resins require chemical regeneration, resulting in the formation of more concentrated wastewater, which increases both operational costs and environmental pollution [7]. Additionally, waste management of chemicals used in the regeneration process is a significant challenge. Membrane filtration processes such as nanofiltration (NF), reverse osmosis (RO), and forward osmosis (FO) provide high treatment efficiencies. However, these technologies generally require high energy consumption and capital costs [9,10], which makes these methods economically not applicable for large-scale applications. In addition, membrane filtration processes cause further environmental problems by generating more concentrated wastewater brine. High energy consumption and concentrated wastewater brine are the main drawbacks of the RO and NF processes [11]. Thus, new methods have been investigated to recover nutrients from wastewater and increase energy efficiency in membrane processes.

Electrodialysis (ED) is an electromembrane process that separates ions from a solution using ion-exchange membranes (IEMs) under the influence of an electrical field. ED unit typically consists of a series of parallel anion (AEM) and cation (CEM) exchange membranes arranged between the electrodes as anode and cathode. As the wastewater moves on the surface of the ion exchange membranes in the system, electric current/voltage is applied to them to ensure the separation of ions by passing through the membranes. Therefore, electrically charged ions move toward the cathode, and anions move toward the anode in the direction of the applied current. The ions in the diluate compartment, which are cations, pass from the CEMs to the concentrate compartment but cannot pass the AEM and accumulate in the concentrate. Similarly, anions pass from the diluate compartment to the concentrate compartment through AEMs but are captured by CEMs. In this way, ions accumulate in the concentrate compartment and are removed in the diluate compartment [12].

The ED system has been commonly used for desalination of brackish and saline water, resource recovery, wastewater treatment, and metal recovery [13–17]. The ED process is considered a promising technology for wastewater treatment with low energy consumption. It is effective in removing ions in addition to nitrates and phosphates from wastewater and is important in terms of achieving high treatment efficiency and raw material recovery without using chemicals, preventing the formation of secondary pollutants [18]. Besides, its high efficiency in terms of energy consumption provides sustainable wastewater management [19]. Sustainability can be further increased by the integration of renewable energy sources such as solar and wind energy. Compared to traditional methods, the ED process is used in literature for the treatment of water with medium salinity. Energy consumption ranges from 0.4 to 4 kWh/m³ and is lower than traditional methods. Therefore, it offers a more economical solution for long-term operations [2]. In addition, it increases membrane clogging caused by high pressures, negatively affecting membrane performance and increasing energy consumption [20]. Since the system operates at lower pressures in the ED process, there is less accumulation on the membrane, which extends the life of the system and reduces operating costs [21].

Previous research has focused on hydrogen production with the ED process since it can simultaneously produce hydrogen gas, achieving rates of 118.8 mg H₂/h·kg Na₂SO₄ [22]. The recovery of hydrogen gas from the system benefits in reducing the energy consumption of the ED process. In addition to hydrogen generation and treatment of various types of water, nutrient recovery using the ED process has been investigated in many studies; it has been reported that the energy consumption of the ED process ranges from 0.4 to 25 kWh/m³ [2] and it can be reduced by 40 % with various strategies [3]. The wide range of reported energy consumption rates necessitates optimization of both system design and operational parameters to ensure greater consistency and efficiency [5].

Response Surface Methodology (RSM) is a highly effective statistical method used to optimize and analyze processes involving multiple variables. It combines experimental design with regression analysis to

create models that optimize performance while representing system behaviors [23]. By utilizing these models, RSM allows researchers to see the relationships between input variables and system responses. This makes it a highly applicable approach in fields such as engineering, manufacturing, and biotechnology. The flexibility and efficiency provided by RSM, especially when dealing with complex, nonlinear systems, make it superior to classical optimization methods [24].

The ED process has recently been studied for hydrogen production in addition to desalination and ion recovery, yet studies integrating hydrogen generation, nutrient recovery, and energy efficiency assessment within a single optimized framework remain scarce. While nutrient recovery has gained increasing attention in recent years, existing studies lack a comprehensive optimization approach to maximize process efficiency. In this study, RSM was employed to determine the optimal operating conditions for hydrogen production and nutrient recovery with minimum energy consumption, filling a critical gap in the literature. The influence of key process variables, including the number of IEMs, voltage, influent pH, and initial conductivity of diluate and concentrate solutions, was systematically investigated. The statistical significance of experimental data was validated through ANOVA analysis. By generating 3D response surface models, this study not only predicts outcomes for untested parameter values but also provides an in-depth understanding of parameter interactions, which has been largely overlooked in previous works. Furthermore, energy consumption was meticulously evaluated, highlighting the sustainability and feasibility of the process. Given the novelty of the ED for hydrogen production and the absence of optimization-driven studies in this domain, this research presents a pioneering approach that bridges this critical research gap. The findings contribute to the development of energy-efficient and resource-recovering hydrogen production strategies, paving the way for more sustainable industrial applications.

2. Methodology

2.1. Electrodialysis process

The ED cell used in this study was purchased from PCCell GmbH Company, located in Germany. The anode is made of Pt/Ir-coated titanium metal, and the cathode is V4A stainless steel. Both electrodes are rectangular with dimensions of 8 cm × 8 cm. The ED system can be operated with a maximum current of 5 A and a voltage of up to 30 V and the nominal flow rate is between 4 and 8 L/h.

In this study, standard anion (PCSA), cation (PCSC), and end-cation exchange membranes (End-CEM) were obtained from PCCell GmbH. End spacers are used between electrodes and the membranes to prevent water passage, while the End-CEM, known for its durability and stability over a wide pH range, is placed next to the anode to protect against chemical byproducts such as hydrogen peroxide (H₂O₂) or chlorine gas (Cl₂) that may form at the anode during operation.

Fig. 1 shows the schematic image of the ED cell, including ion exchange membranes and spacers. Different electrical potentials (5–9 V) were applied to the ED system using a DC power supply (Array 3645 A), and average electrical energy consumptions were calculated by reading the current values every 5 min. In addition, the electrolyte, dilute, and concentrate solutions were circulated using a four-device peristaltic pump (LongerPump BT100–1 L) at a speed of 110.1 mL/m. Each experiment was conducted for 30 min, and all solutions were mixed at 300 rpm stirring speed to supply homogenous solutions in the ED cell. A multimeter continuously monitored conductivity levels (Thermo Scientific-Orion Star A322).

During the experiments, hydrogen gas was produced in the cathode chamber and collected in a graduated cylinder as shown in Fig. 2. The volume of hydrogen gas produced was measured over time. The cathode compartment outlet was recirculated to the catholyte reservoir through the tube, and the solution was pumped back into the cathode compartment as the hydrogen gas rose into the cylinder. A magnetic

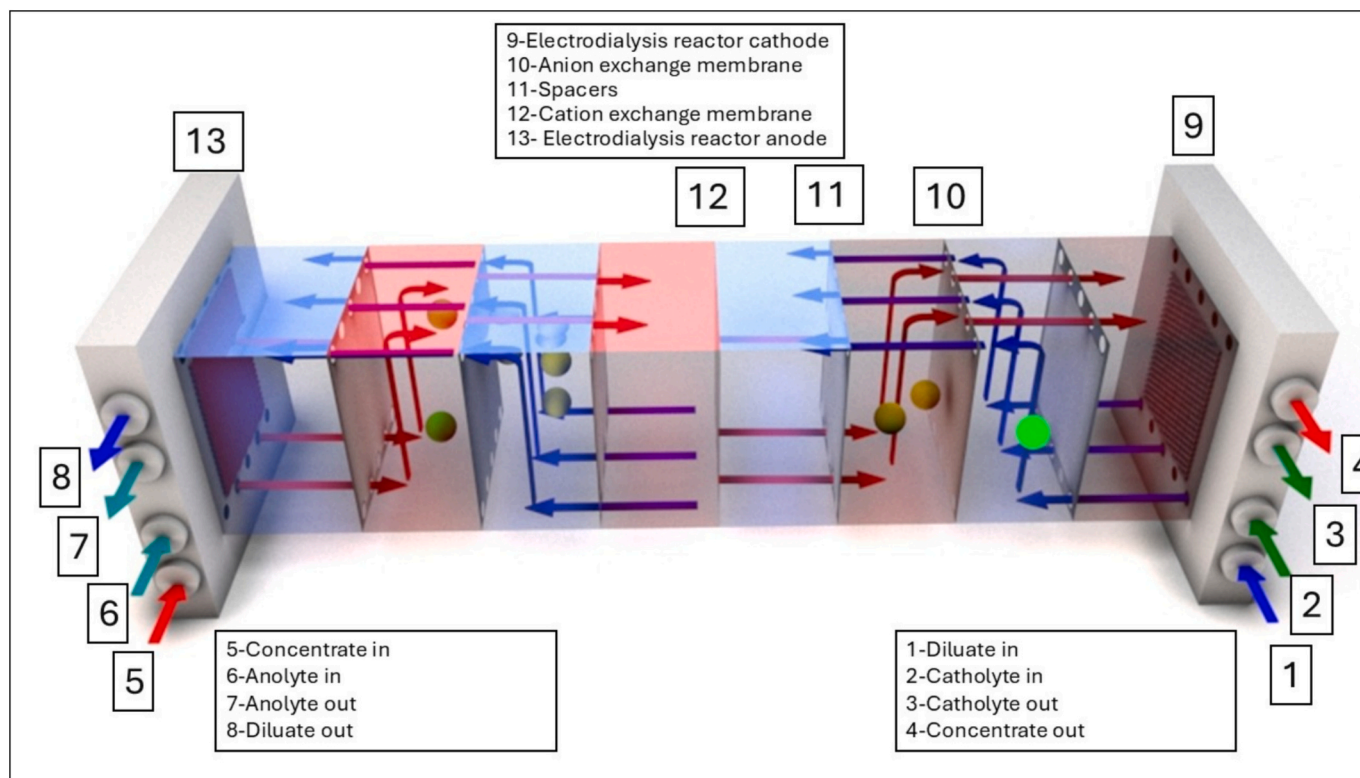


Fig. 1. Schematic representation of the ED reactor and its components (anion exchange membranes, cation exchange membranes, spacers, water flow pattern, and ion transitions in the reactor).

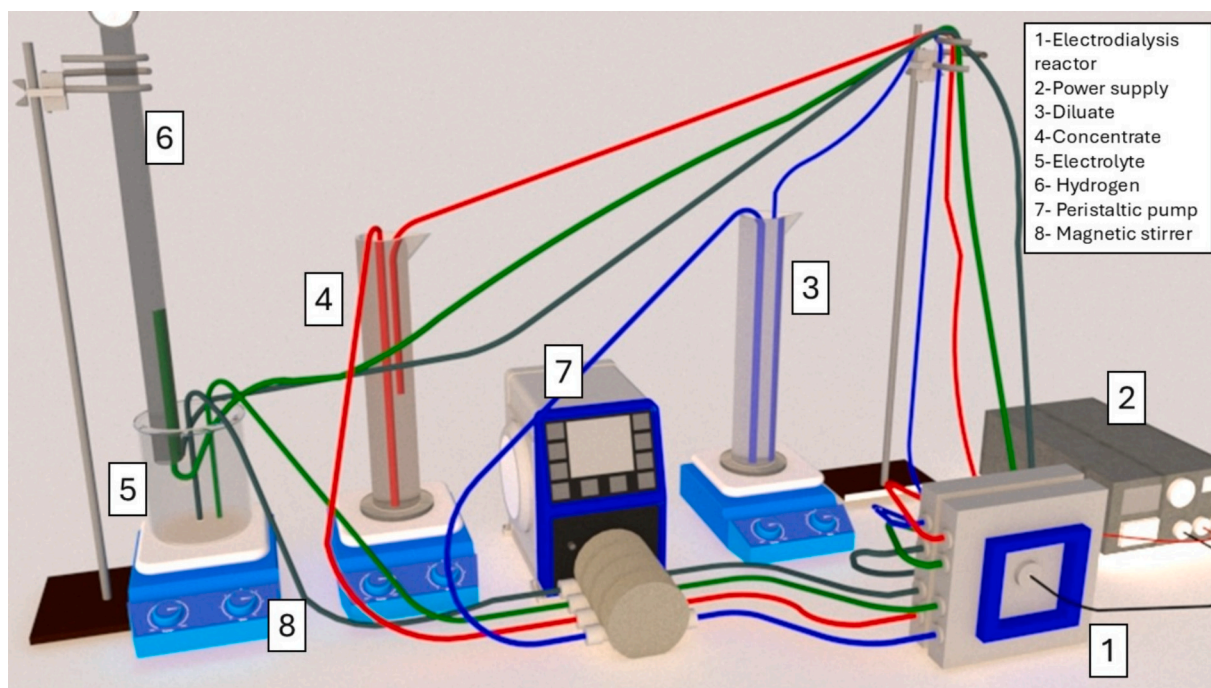


Fig. 2. Schematic representation of the system.

stirrer was used to provide homogeneous solutions in a combined configuration of the anode and cathode compartments.

2.2. Preparation of synthetic wastewater

The solutions were prepared to have the same ion concentrations and

amounts at the same ratios, simulating biologically treated municipal wastewater [25]. Seawater, which has high salinity and conductivity value in literature applications, was selected. To reach the seawater conductivity value of 54 mS/cm, 1.49 g KH_2PO_4 , 29.87 g NaNO_3 , and 10.67 g NH_4Cl were mixed thoroughly in one liter of distilled water. Synthetic wastewaters with conductivity values of 18, 27, 36, and 45

mS/cm were prepared by diluting the stock wastewater with conductivity of 54 mS/cm using distilled water.

2.3. Analytic methods

The electrical conductivity of a solution is primarily determined by the concentration and mobility of dissolved ions and is a significant indicator of ion recovery efficiency in the ED process [26]. Since the ED process removes ions by transporting them through IEMs under the influence of an electric field, a decrease in conductivity indicates successful ion recovery [27]. The conductivity recovery rate can be calculated as nutrient recovery efficiency by the following equation [28]:

$$\eta_{\text{recovery}} = \left(\frac{K_0 - K_{30}}{K_0} \right) * 100 \quad (1)$$

where η_{recovery} is nutrient recovery efficiency rate (%), K_0 : the initial conductivity of the diluate solution, K_{30} : Conductivity of diluate solution at 30 min (mS/cm). The duration was determined as 30 min to increase the industrial applicability of the system.

Energy consumption in the ED process is crucial to evaluating the efficiency of the system. The limiting factor in terms of the sustainability of water nutrient recovery systems is specific energy consumption (SEC), usually calculated in kWh/m³ [29]. The calculations were made according to the formula and used in the RSM.

$$\text{SEC} = \frac{I \cdot V_{cp} \cdot t}{V_{\text{treated}}} \quad (2)$$

where, SEC is specific energy consumption (kWh/m³), I ; is applied current (Ampere), V_{cp} ; is applied voltage (Volt), t ; is the operating time (hour), V_{treated} ; is treated volume (m³) [30].

2.4. Experimental design of RSM

Response Surface Methodology (RSM) statistically examines the impact of several independent factors on the result. Engineers often use it for process optimization, particularly in multivariate analyses. The assessment of combinations of independent variables for the research to achieve optimal process performance may be conducted using RSM. The response surface technique employs many experimental design strategies. The Central Composite Design (CCD), often used by engineers, was chosen for the assessment of numerous data within the study's framework. One benefit of CCD is its suitability for the creation of second-order models. The working concept involves modeling the impacts of nonlinear variables that interact with one another and representing these effects on the response surface [31]. The fundamental formulation:

$$Y = \beta_0 + \sum_{n=1}^k \beta_n \cdot x_n + \sum_{n=1}^k \beta_{nn} \cdot x_n^2 + \sum_{n \leq m}^k \sum_m^k \beta_{nm} \cdot x_n x_m + \dots e \quad (3)$$

In this context, 'y' signifies the expected response variable, 'n' and 'm' denote the linear and quadratic coefficients, ' β ' represents the regression coefficient, 'k' indicates the number of factors examined and optimized during the experiment, and 'e' symbolizes the random error.

There is the ability to make predictions about the response at certain factor levels thanks to the equation that is stated in terms of coded factors. High factor levels are denoted by the symbol +1 in this context, and low factor levels are denoted by the symbol -1, shown in Table 1. Because it allows for a comparison of the coefficients of the components, this coded equation is very helpful in determining the relative effect that each of the elements has [23].

Table 2 displays the statistical assessment of several model architectures (Linear, 2FI, Quadratic, Cubic) for nutrient recovery, hydrogen production, and energy consumption. F-values, p-values, and Lack of Fit p-values for each model were used to assess model fit and precision. The

Table 1

Code values of variables used in RSM.

Variables	Unit	Code	Ranges and levels				
			-2	-1	0	1	2
Number of membranes	–	A	6	8	10	12	14
Voltage	V	B	3	5	7	9	11
Initial pH	–	C	5	6	7	8	9
Initial conductivity	mS/cm	D	18	27	36	45	54
Conductivity of concentrate	mS/cm	E	1	2	3	4	5

quadratic model was selected for nutrient recovery, hydrogen production, and energy consumption due to its strong statistical validity. Specifically, the Sequential p-value (<0.0001) indicates a highly significant model, while the Lack of Fit p-value is not significant, confirming a robust fit with the experimental data. These results demonstrate that the quadratic model effectively captures the underlying relationships without the unnecessary complexity of higher-order models, such as the cubic model.

Statistical analyses were conducted using the results to identify the model in RSM, with the findings presented in Table 3. The quadratic model was chosen for the analysis due to its high R² value and satisfactory fit results, despite the presence of an aliased cubic model. The model is appropriate as the difference between the predicted R² and adjusted R² values is <0.2 for a quadratic model.

The standard deviation values are low across all three models: 0.7877 for hydrogen production, 1.01 for nutrient recovery, and 0.0138 for energy consumption. This suggests a low level of uncertainty in the model estimates. The R² values are 0.9999 for hydrogen production and energy consumption and 0.9987 for nutrient recovery efficiency. These elevated values suggest that the models nearly fully account for the data. The Adjusted R² values (0.9999 for hydrogen production and energy consumption, 0.9978 for nutrient recovery efficiency) indicate that the models are robust and that the independent variables included contribute significantly to the model's explanatory power [32].

Predicted R² values demonstrate the predictive efficacy of the models on novel data, ranging from 0.9956 to 0.9998, thereby indicating a high level of generalizability for the models. The Coefficient of Variation (C.V.%) is 0.5842 % for hydrogen production, 1.57 % for nutrient recovery efficiency, and 0.6911 % for energy consumption, indicating that the models exhibit stable and reliable performance. The "Adeq Precision" values (459.2250 for hydrogen production, 119.2950 for nutrient recovery efficiency, and energy consumption) indicate a high signal-to-noise ratio, suggesting that the model possesses strong predictive power. The statistical criteria indicate that all models demonstrate high accuracy, generalizability, and reliability [33].

3. Results and discussion

3.1. ANOVA results

The variables evaluated for process optimization included voltage, membrane number, initial conductivity, initial pH, and conductivity of concentrate, as detailed in Appendix A. During the RSM optimization process, 50 sets were analyzed, each consisting of various combinations of voltage, membrane number, initial conductivity, initial pH, and conductivity of concentrate.

The ANOVA results for nutrient recovery, hydrogen production, and energy consumption for the RSM second-order model in this study are presented in Appendix B. It provides a detailed summary of the findings from the experimental study. The evaluation focused on the independent variables A (membrane number), B (voltage), C (initial pH), D (initial conductivity), and E (conductivity of concentrate) in relation to the five performance parameters, including their interactions and second-order effects.

According to the ANOVA results, the linear, quadratic, and

Table 2
Model summary and lack of fit test results.

	Source	Sum of Squares	df	Mean Square	F-value	Sequential p-value	Lack of Fit p-value	
Nutrient recovery efficiency	Linear	4957.54	37	133.99	242.04	< 0.0001	<0.0001	
	2FI	4745.29	27	175.75	317.49	0.9984	<0.0001	
	Quadratic	25.79	22	1.17	2.12	< 0.0001	0.1558	Suggested
	Cubic	9.79	7	1.40	2.53	0.4368	0.1221	Aliased
	Pure Error	3.88	7	0.5536				
Hydrogen production	Linear	8019.62	37	216.75	202.30	< 0.0001	<0.0001	
	2FI	1926.12	27	71.34	66.58	< 0.0001	<0.0001	
	Quadratic	10.49	22	0.4770	0.4452	< 0.0001	0.9305	Suggested
	Cubic	5.60	7	0.7993	0.7460	0.9740	0.6456	Aliased
	Pure Error	7.50	7	1.07				
Energy consumption	Linear	19.44	37	0.5253	6685.84	< 0.0001	< 0.0001	
	2FI	14.75	27	0.5464	6954.06	0.4041	< 0.0001	
	Quadratic	0.0049	22	0.0002	2.86	< 0.0001	0.0789	Suggested
	Cubic	0.0004	7	0.0001	0.6509	0.0030	0.7075	Aliased
	Pure Error	0.0006	7	0.0001				

Table 3
Fitting statistics for hydrogen production, nutrient recovery efficiency, energy consumption.

	Hydrogen production	Nutrient recovery efficiency	Energy consumption
Std. Dev.	0.7877	1.01	0.0138
Mean	134.84	64.46	1.99
C.V. %	0.5842	1.57	0.6911
R ²	0.9999	0.9999	0.9999
Adjusted R ²	0.9998	0.9998	0.9999
Predicted R ²	0.9997	0.9997	0.9998
Adeq	459.2250	119.2950	119.2950
Precision			

interaction effects of all independent variables were examined for nutrient recovery, hydrogen production, and energy consumption. The statistical analysis revealed that certain interaction terms were not significant ($p > 0.05$), specifically BE for energy consumption, C and DE for hydrogen production, and C, AB, AC, BC, CE, and DE for nutrient recovery. Despite these non-significant terms, the voltage variable consistently exhibited the highest F-values, indicating its dominant influence on the system's performance. The computed F-values for voltage were 1.740E+05 for hydrogen production, 3.75E+05 for energy consumption, and 14.714 for nutrient recovery, all of which underscores its critical role in the process. Moreover, voltage was identified as the most influential parameter, as all its corresponding p-values were < 0.0001 , further validating its statistical significance. This finding is corroborated by Pareto charts, which provide a visual representation of the dominant effect of voltage compared to other process parameters. The results suggest that optimizing voltage is crucial for maximizing system performance while ensuring process efficiency. These insights contribute to a deeper understanding of the operational parameters influencing electrodialysis-based hydrogen production and resource recovery, reinforcing the necessity of targeted process optimization strategies (Appendix B) [34].

Following the completion of the study sets, the data were modeled using RSM, and a comparison was made between the actual values of the data and the anticipated values, the results are given in Fig. 3 (a-c). It is possible to assert that the values predicted by the model and the actual values of the research are identical to one another. This is because no variation was seen on the slope line in the graphs that compared the predicted values to the actual values. As an additional indicator that the R² values are quite near 1, the fact that the data are on the slope is shown. It can be seen in the graph that the red colors represent high data, while the blue hues represent low data. Graphs that display the normal plot of residuals demonstrate whether the model's prediction errors are consistent with the normal distribution. Because the deviation did not vary greatly from the 45-degree angle in any of the sets that were

used in the research, it is clear that the error is appropriate for the distribution.

In this study, the results were produced by the software Design Expert 12 according to Eq. (3) using central composite design within the scope of response surface methodology (Table 4). While creating the equations, linear quadratic and interaction coefficients of all variables were given in the equation. However, while creating the Pareto chart, the calculation was made by taking the variables that were significant in the ANOVA results. Since the effect of significant variables on the calculations was in trace amounts, they were neglected while creating the Pareto chart.

3.2. RSM results of the effect of parameters on nutrient recovery efficiency

The influence of each variable on nutrient recovery efficiency is systematically ranked, with voltage (B) emerging as the most significant positive contributor. Other notable influencing factors include initial conductivity (D), the quadratic terms for voltage (B²) and membrane number (A²), as well as the quadratic term for initial conductivity (D²). Although interaction effects, such as BD and BE, also play a role, their overall impact is comparatively lower. The presence of negative coefficients in D² and A² suggests diminishing returns or potential adverse effects as these variables increase (Fig. 4).

The interactions of various parameters and their combined effects on nutrient recovery efficiency are illustrated in the 3D graphs generated using RSM, as shown in Fig. 5. These graphs provide valuable insights into how different operational conditions influence the overall performance of the system. As shown in Fig. 5 (a-k), RSM analysis shows that a nutrient recovery efficiency of 80 % can be achieved; several parameters must be fine-tuned within specific ranges. Specifically, the number of membranes should be between 8 and 12, while the voltage must be maintained between 7 and 13 V. Additionally, the initial conductivity of the influent water should range from 18 to 42 mS/cm to ensure optimal ion recovery, and the concentrate initial conductivity should ideally be kept between 2.5 and 4 mS/cm to maintain efficient operation. The results emphasize the importance of parameter optimization to maximize efficiency, particularly the balance between membrane number, voltage, and initial conductivity levels, which collectively play a crucial role in the system's performance.

3.3. RSM results of the effect of parameters on hydrogen production

The direct effects of variables A, B, D, and E are positive, but the interactions AB, AC, AD, BC, BE, CD, and CE provide negative effects as can be seen in Fig. 6. One of the important factors in the increase in production is seen as variable B. In D and E, a positive development in production is seen, but they are not as effective as B. Alongside the

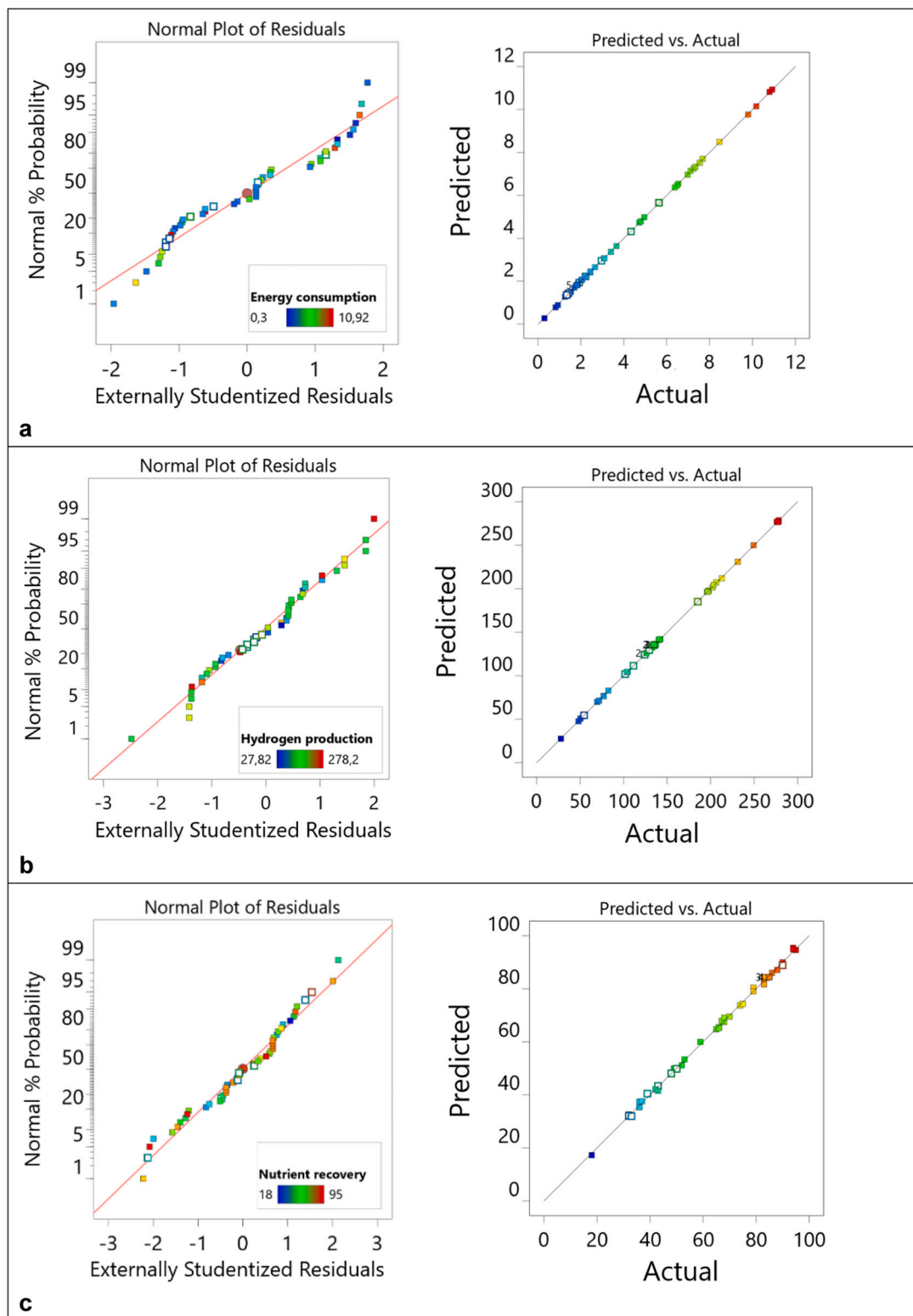


Fig. 3. Normal of residuals and predicted vs actual graphs where the data were evaluated after the response surface model, a) energy consumption, b) hydrogen production, c) nutrient recovery.

individual contributions of variables A, B, C, D, and E, the terms A^2 , B^2 , and C^2 also provide positive contributions.

The interaction of different parameters with each other and their effects on hydrogen production are given in 3D graphics created using RSM in Fig. 7. The optimum results for 150 mL hydrogen production are voltage 5–9, membrane number 8–12, and initial conductivity 18–45

mS/cm. However, no effect of conductivity of concentrate and pH was observed. The initial conductivity affects the resistance of the ED cell and, thus, influences the current passing through the system. Since hydrogen production is influenced by the current passing through the system, it can be concluded that concentrated concentration has indirect effects on hydrogen production.

Table 4

Coded equations for nutrient recovery efficiency, hydrogen production, energy consumption.

Nutrient recovery efficiency	$84.36 + 2.85A + 19.4B - 0.3C - 7.75D + 1.3E - 0.1875AB + 0.0625AC - 0.9375AD + 0.625AE + 0.125BC - 2BD + 1.0625BE + 0.375CD + 0.0625CE + 0.1875DE - 6.325A^2 - 7.075B^2 - 0.825C^2 - 5.2D^2 - 5.45E^2$
Hydrogen production	$126.69 - 29.55A + 51.95B + 0.25C + 23.9D + 1.25E - 3.1875AB - 1.375AC - 0.4375AD + 0.75AE - 1.75BC + 13.1875BD - 0.5BE - 0.375CD - 0.4375CE + 0.25DE + 7.5125A^2 + 0.7625B^2 + 1.3875C^2 - 0.3625D^2 + 0.8875E^2$
Energy consumption	$0.9077 - 0.414A + 1.332B + 0.1465C + 0.4055D - 0.078E - 0.195AB - 0.075625AC - 0.03625AD + 0.06AE + 0.00875BC + 0.261875BD + 0.001875BE + 0.10875CD + 0.07625CE - 0.106875DE + 0.360375A^2 + 0.474125B^2 + 0.121625C^2 + 0.277875D^2 + 0.119125E^2$

Overall, the RSM analysis underscores the importance of optimizing key parameters such as voltage, membrane number, and initial conductivity to maximize hydrogen production. By fine-tuning these variables within the identified ranges, the system can be operated efficiently to achieve the desired hydrogen production, while minimizing the influence of less critical parameters such as conductivity of concentrate and pH.

3.4. RSM results of the effect of parameters on energy consumption

As shown in the Pareto Chart for energy consumption in Fig. 8, variables A, B, C, D, and E directly influence energy usage; nonetheless, A has a negative impact. Variables B, C, D, and E contribute positively, but A has a negative influence. Interactions AB, AC, AD, BE, and DE have negative impacts, while interactions AE, BC, BD, CD, and CE provide positive contributions. Terms A^2 , B^2 , C^2 , D^2 , and E^2 provide positive contributions, with A^2 and B^2 demonstrating a particularly strong beneficial impact. Consequently, identifying the factors and interactions that significantly impact energy use using Pareto analysis is essential. Key variables signify domains requiring optimization to enhance energy efficiency. The positive impact of B suggests that regulating this variable may decrease energy usage, but the negative effect of A implies that this variable should be restricted. Consequently, methods may be developed to save energy and attain sustainability objectives.

The interaction of different parameters with each other and their effects on hydrogen production are given in 3D graphics created using RSM in Fig. 9. For lower energy consumption, the voltage is between 3 and 5, the number of membranes is 9–13, and the initial conductivity is 20–42 mS/cm. No effect of pH and conductivity of concentrate on energy consumption was observed. However, it can be said that the optimum pH is between 5 and 8. Additionally, the conductivity of concentrate appears to operate optimally in the range of 1.5 to 4.5 mS/

cm, suggesting that while these parameters may not directly affect energy consumption, they may contribute to the overall stability and efficiency of the hydrogen production process.

3.5. Effect of pH

The role of initial pH (C) in the ED process is primarily regulatory, influencing membrane selectivity, ion transport mechanisms, and electrochemical stability rather than serving as a major determinant of energy consumption, hydrogen production, or nutrient recovery efficiency [35]. Compared to voltage (B), membrane number (A), initial conductivity (D), and conductivity of concentrate (E), the direct impact of pH on overall system performance is relatively minor. However, its proper regulation is essential for maintaining membrane integrity and optimizing ion selectivity, making it a key factor for long-term operational stability rather than an immediate driver of efficiency.

During the ED process, pH changes occur within the system due to ion transport and electrolysis of water, which affects wastewater quality and, thus, membrane performance [36]. pH affects the permeation of organic pollutants through ion exchange membranes and the efficiency of salt removal [37].

In a domestic wastewater treatment plant using the ED process, pH optimization and voltage adjustment directly affect salt removal performance and nitrate recovery efficiency. The study determined the optimum pH as 7 [38]. A different study found that pH significantly affects arsenic removal but does not affect nitrate and fluoride removal. It was determined that it affects the removal efficiencies of various pollutants in wastewater in ED processes [39]. Understanding the interactions of pH with different variables in the process is essential to optimize system design, increase contaminant removal, and improve energy efficiencies and overall performance [38].

According to the model results, the interaction of pH (C) with other parameters and its effect on the results are given graphically in Figs. 5, 7, 9-e, h, i. In experimental studies, it was determined that hydrogen production increased by 19 %, energy consumption by 9 %, and nutrient recovery efficiency by 12 % from pH 5 to 9. Although there was an 80 % increase in pH, small effects were obtained in the results. These results are consistent with the 3D graphics produced by the model (Figs. 5–7–9) and the effect ratios given in the equations in Table 4.

For energy consumption, pH has a limited direct influence, but its interaction with other variables affects membrane resistance and ion transport efficiency. The interaction between pH (C) and membrane number (A) follows a U-shaped trend, where energy consumption initially decreases with an increasing number of membranes due to improved ion transfer efficiency, but stabilizes beyond a threshold as resistive losses become dominant. Similarly, the interaction with voltage (B) suggests that higher voltage amplifies energy consumption,

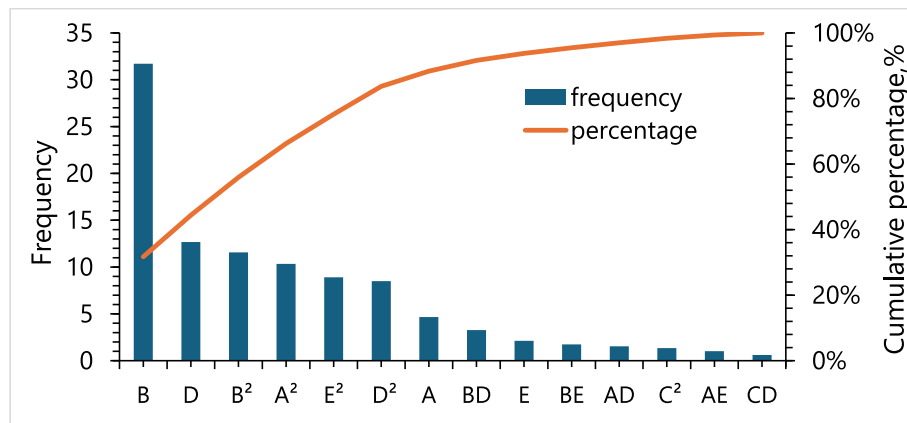


Fig. 4. Pareto Chart for Nutrient recovery Efficiency: Model Terms, Quadratic Model, and Coefficient of Determination (R^2).

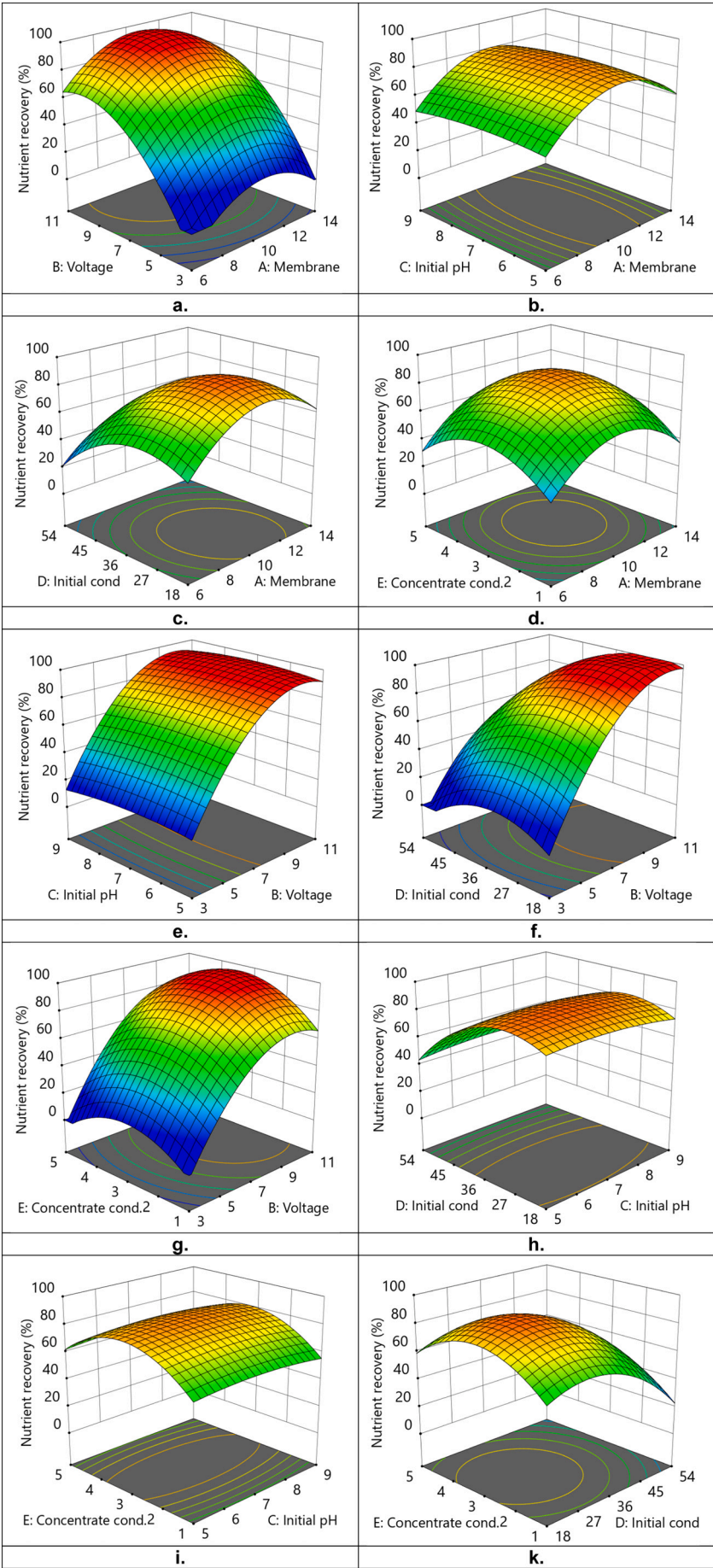


Fig. 5. The effect of voltage and membrane number(a), effect of initial pH and number of membranes (b), effect of initial conductivity and membrane number (c), effect of conductivity of concentrate and membrane number (d), effect of pH and voltage (e), effect of initial conductivity and voltage (f), effect of conductivity of concentrate and voltage (g), effect of Initial conductivity and pH (h), effect of conductivity of concentrate and pH (i), effect of conductivity of concentrate and initial conductivity (k) on nutrient recovery efficiency (%).

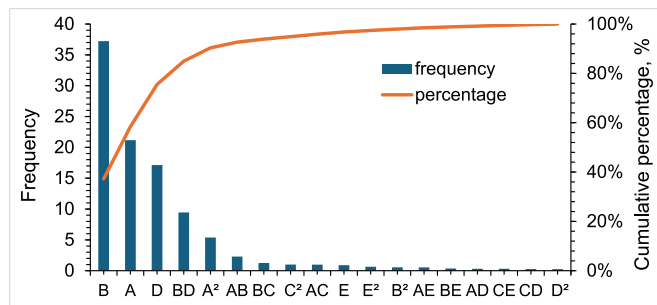


Fig. 6. Pareto Chart for hydrogen production: Model Terms, Quadratic Model and Coefficient of Determination (R^2).

regardless of pH variations, confirming that voltage control is far more influential in determining energy efficiency. The interaction with initial conductivity (D) demonstrates that higher conductivity reduces the influence of pH on energy demand, as increased ion mobility minimizes pH-induced resistance fluctuations. Lastly, the interaction with the conductivity of concentrate (E) reveals that pH effects on energy demand are minimal at high conductivity of concentrate levels, whereas at low conductivity of concentrate, pH variations may slightly influence system resistance.

For hydrogen production, pH plays a secondary role, primarily affecting membrane stability and electrochemical performance rather than directly influencing hydrogen yield. The interaction with membrane number (A) shows that increasing membrane count has a greater impact on hydrogen production than pH itself, reinforcing that membrane configuration dictates current efficiency rather than pH fluctuations. Similarly, the interaction with voltage (B) confirms that voltage remains the dominant driver of electrolysis, with pH variations introducing only minor changes. The interaction with initial conductivity (D) highlights that while extreme pH values may slightly influence ion solubility and membrane charge, their effect on hydrogen yield remains negligible. Lastly, the interaction with the conductivity of concentrate (E) indicates that pH adjustments influence ion selectivity but have no significant direct impact on hydrogen generation, reinforcing that voltage and conductivity remain the primary variables controlling electrochemical hydrogen production.

For nutrient recovery, pH modulates membrane charge interactions and ion solubility, indirectly affecting separation efficiency. Thus, it is not a major determinant of energy consumption or nutrient recovery efficiency [40]. The interaction with membrane number (A) follows a moderately increasing trend, where higher pH values enhance membrane charge stability, improving selectivity, though the nutrient recovery remains primarily dictated by membrane count. Similarly, the interaction with voltage (B) confirms that voltage is the dominant force driving ion transport, with pH variations playing a stabilizing role rather than a direct determinant of nutrient recovery efficiency. The interaction with initial conductivity (D) follows a mild increasing pattern, where higher conductivity improves nutrient recovery at elevated pH levels due to enhanced ion solubility, though excessive conductivity may lead to competition effects, reducing selectivity. Lastly, the interaction with the conductivity of concentrate (E) shows that higher pH values may slightly enhance nutrient migration at specific conductivity of concentrate levels, but this effect remains secondary to voltage and conductivity influences.

In summary, pH primarily serves as a stabilizing factor in the ED process, influencing membrane selectivity, ion solubility, and

electrochemical stability rather than acting as a direct driver of energy consumption, hydrogen production, or nutrient recovery. Although moderate pH adjustments can improve membrane lifespan and optimize ion transport, the dominant factors governing ED performance remain voltage and conductivity. Therefore, pH regulation should focus on preserving membrane integrity and maintaining stable ion transport rather than being relied upon as a primary optimization parameter for enhancing system efficiency.

3.6. Effect of membrane number

Membrane number (A) is a crucial parameter in the ED process, with its impact extending across energy consumption, hydrogen production, and nutrient recovery. Acting as both an enabler of ion transport and a source of increased system resistance, the number of membranes must be carefully optimized in relation to voltage (B), initial conductivity (D), conductivity of concentrate (E), and initial pH (C) [40]. While a well-balanced membrane configuration enhances separation efficiency and reduces energy losses, an excessive number of membranes can introduce additional resistance, limiting the system's overall effectiveness.

The number of membranes used in an ED cell influences the system's resistance and increases the surface area where ion transfer occurs. Increasing the number of membranes would increase the ion transport rate; however, after a point, the increased resistance due to the membranes would decrease current transfer. Thus, using an optimum number of membranes in an ED stack is crucial.

In the ED process, high voltage and increased surface area due to increased membrane number are the factors that directly increase nutrient recovery rates [41]. Increases in these parameters increase ion transfer rates, providing more efficient separation and recovery. An increase in the number of cell pairs can also be supported by increasing the flow rate and the applied voltage. In the literature, optimizing the number of membranes, voltage, and flow rate dramatically increases the efficiency of heavy metal removal processes in electrocoagulation and electrochemical methods. Increasing the number of cells contributes to the attraction of metal ions to the surface and the acceleration of the process, thus reducing energy consumption [42].

The selective removal of divalent ions was especially found to be improved by decreasing cell length, applying potential, and feeding water ionic strength. These findings highlight the importance of understanding the interactions of factors affecting ED efficiency and optimizing membrane properties and operational parameters for the specified objectives [43].

This study shows the effect of the number of membranes (A) on nutrient recovery efficiency. As the number of membranes increases, the current flow decreases due to the increase in resistance and distance (Fig. 5a). Therefore, the amount of hydrogen production decreases (Fig. 7a). More current needs to be given to the system to produce the same amount of hydrogen, which increases energy consumption. In the experimental studies, it was determined that when the number of membranes was increased from 5 to 15, hydrogen production decreased by 63 %, energy consumption decreased by 60 %, and nutrient recovery efficiency increased by 24 %. In the graphs produced with the model, it is seen that increasing the number of membranes increases nutrient recovery efficiency and decreases energy consumption (Fig. 9a). This is because the larger surface area allows more ion transfer with less energy [36].

For energy consumption, membrane number affects system resistance and ion transport pathways, leading to a nonlinear relationship with voltage (B) [44]. At low membrane counts, increasing voltage

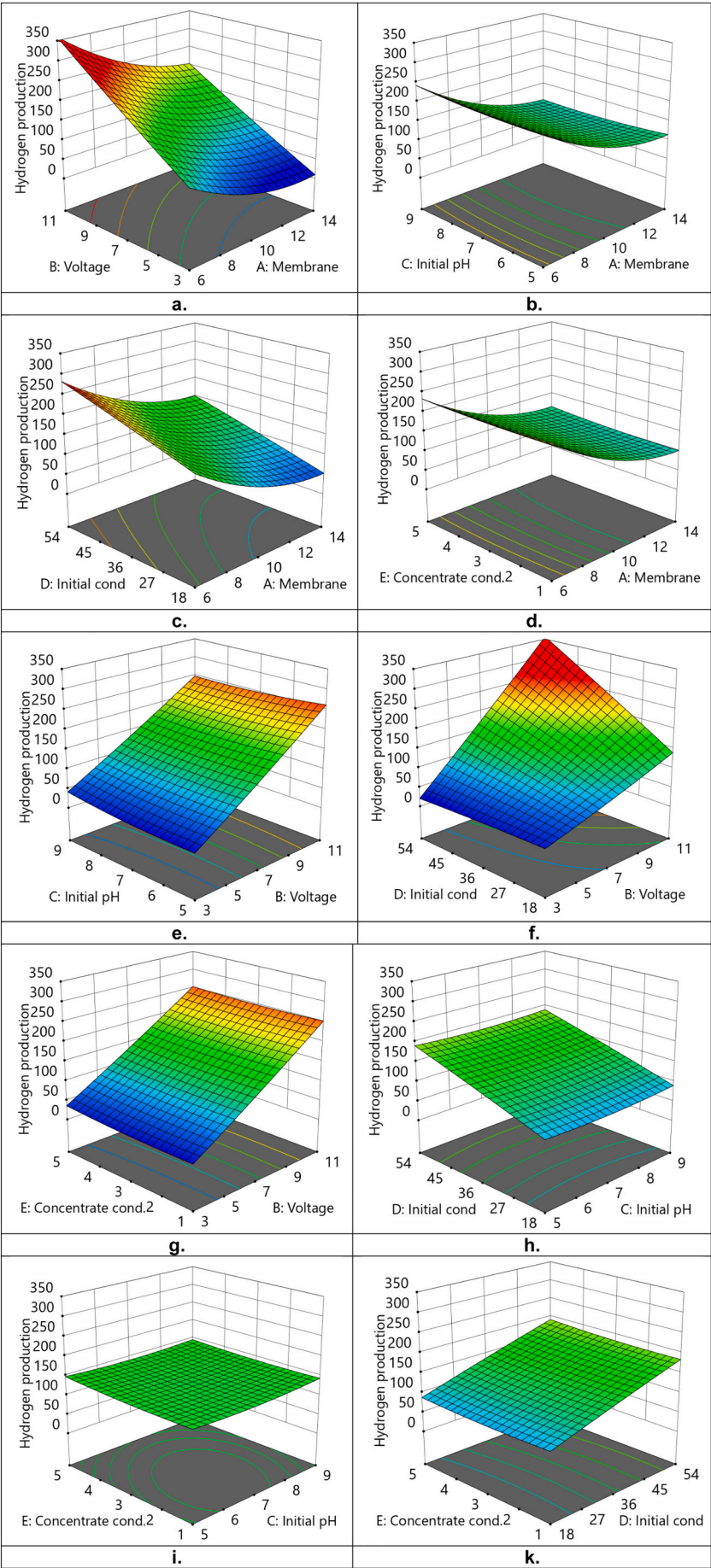


Fig. 7. The effect of voltage and membrane number(a), effect of initial pH and number of membranes (b), effect of initial conductivity and membrane number (c), effect of conductivity of concentrate and membrane number (d), effect of pH and voltage (e), effect of initial conductivity and voltage (f), effect of conductivity of concentrate and voltage (g), effect of Initial conductivity and pH (h), effect of conductivity of concentrate and pH (i), effect of conductivity of concentrated initial conductivity (k) on hydrogen production (mL).

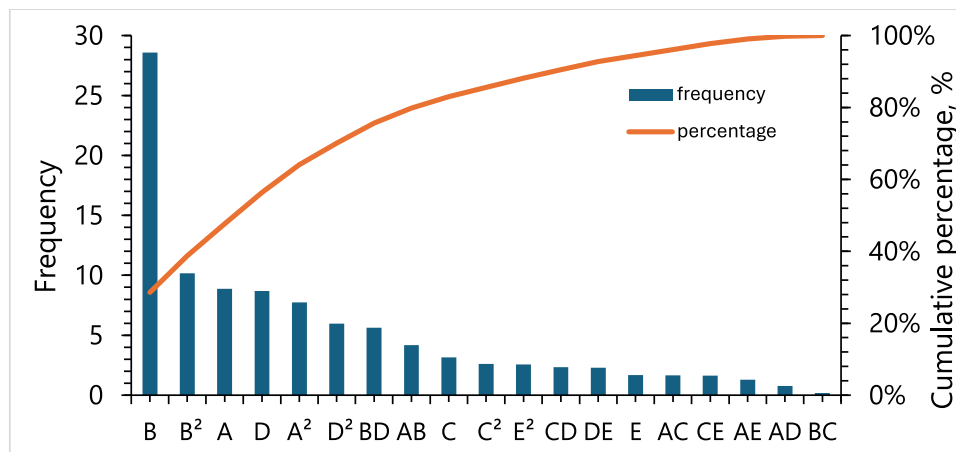


Fig. 8. Pareto Chart for energy consumption: Model Terms, Quadratic Model and Coefficient of Determination (R^2).

raises energy consumption sharply due to limited ion migration capacity. However, at higher membrane numbers, energy demand stabilizes as resistance is reduced, optimizing charge transfer efficiency. Beyond an optimal point, excessive membrane count introduces additional resistance, counteracting the benefits of higher voltage. Similarly, the interaction with initial conductivity (D) shows that higher conductivity reduces resistance and energy demand, but excessive conductivity may cause competitive ion interactions, affecting transport efficiency. The interaction with the conductivity of concentrate (E) reveals that low conductivity of concentrate increases resistance, requiring higher energy input to sustain ion migration. Conversely, high conductivity of concentrate can lead to ion crowding, reducing selectivity and increasing system inefficiencies. Lastly, pH (C) has only a minor impact on energy demand, primarily influencing membrane stability rather than direct energy efficiency.

For hydrogen production, membrane numbers strongly affect current distribution and charge transfer efficiency. The interaction with voltage (B) shows that while increasing voltage enhances hydrogen yield, an excessive number of membranes increases system resistance [44], limiting current efficiency and reducing hydrogen production. At lower membrane counts, higher current flow promotes better hydrogen evolution, while at higher membrane counts, excessive resistance leads to diminishing returns. Similarly, the interaction with initial conductivity (D) demonstrates that higher initial conductivity enhances ion mobility and hydrogen generation, but as membrane count increases, the added resistance from multiple layers counteracts this effect, reducing overall production efficiency. The interaction with the conductivity of concentrate (E) shows minimal direct impact on hydrogen yield, suggesting that voltage and ion transport efficiency are the dominant factors. Finally, pH (C) variations do not significantly alter hydrogen production, confirming that membrane resistance and voltage regulation are the primary determinants of system efficiency.

For nutrient recovery, membrane number directly affects ion separation efficiency, but its effectiveness depends on interactions with voltage, conductivity, and ion competition. The interaction with voltage (B) exhibits a parabolic trend, where nutrient recovery increases with voltage but declines at excessively high membrane numbers due to increased system resistance and reduced current efficiency. Similarly, the interaction with initial conductivity (D) follows a U-shaped response, where moderate initial conductivity yields the highest

nutrient recovery, while very low or very high conductivity levels decrease efficiency due to ion transport limitations or competitive interactions. The interaction with the conductivity of concentrate (E) also follows a symmetric parabolic trend, where both very low and very high conductivity of concentrate values negatively affect nutrient separation. At low conductivity of concentrate, limited ion mobility restricts separation, whereas high conductivity of concentrate introduces co-ion interference, reducing selectivity. Lastly, the interaction with pH (C) indicates only a minor effect, with higher pH values slightly improving nutrient recovery due to membrane charge stabilization, but this remains secondary compared to voltage and conductivity effects.

Ultimately, membrane number is a critical factor in ED performance, influencing energy demand, hydrogen production, and nutrient recovery efficiency. While an increase in membrane count initially enhances ion transport and separation, an excessive number of membranes results in higher system resistance, which consequently reduces charge transfer efficiency and ion migration rates [45]. This diminishing return necessitates a multi-variable optimization strategy, where membrane configuration is carefully balanced with voltage and conductivity to maximize ion transport efficiency while minimizing resistive losses and unnecessary energy consumption.

3.7. Effect of voltage

Voltage (B) serves as the fundamental driving force in electrodialysis, dictating the efficiency of ion migration, energy consumption, and electrochemical performance [46]. However, its effectiveness is highly dependent on other operational parameters, including membrane configuration, conductivity levels, and system resistance. While increased voltage typically enhances ion transport and hydrogen production, excessive application without proper optimization can lead to elevated energy demand and reduced efficiency due to heightened ohmic losses and resistance effects. Thus, strategic voltage regulation is essential for maximizing ED system performance.

The current density is an important parameter that directly affects system performance in ED. High current densities can generally increase the removal efficiency of pollutants, and pollutant removal efficiencies of up to 98 % have been observed at high currents [47]. However, higher current density may not always yield positive results. Higher current densities accelerate the passage of monovalent ions, slow down the

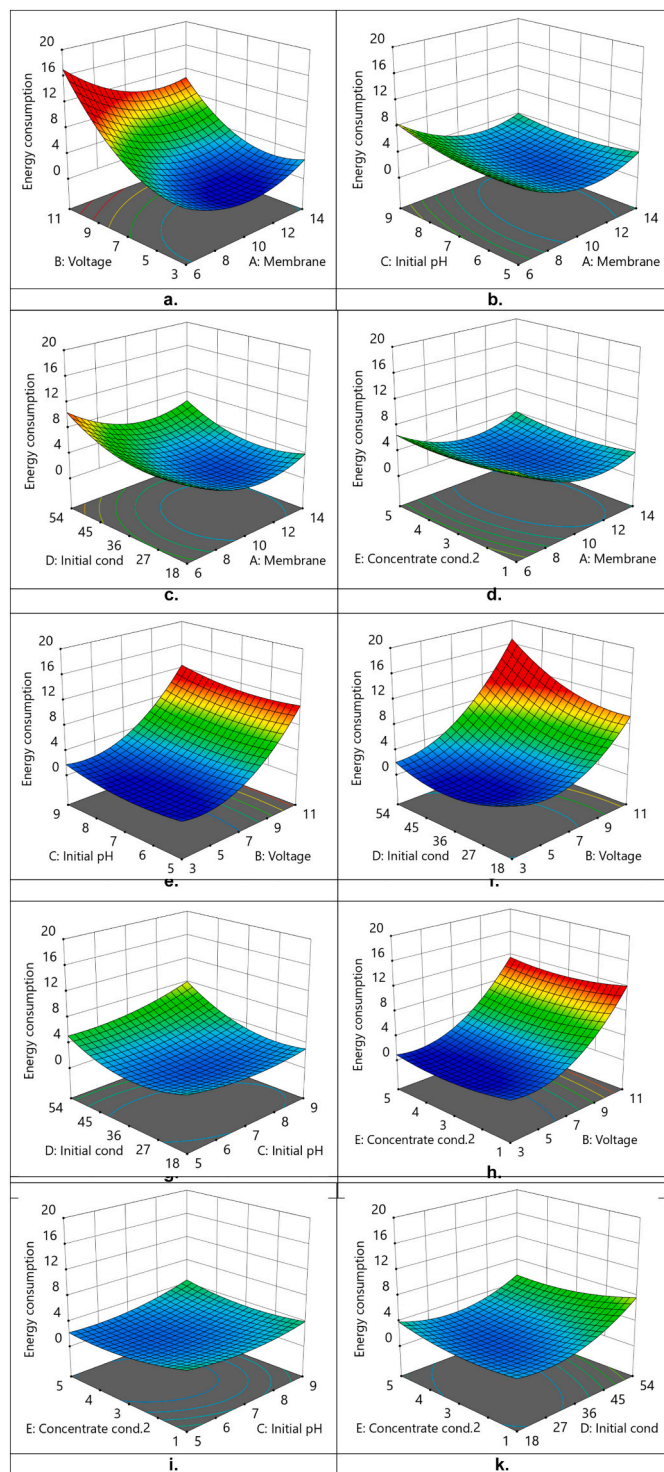


Fig. 9. It shows the effect of voltage and membrane number(a), effect of initial pH and number of membranes (b), effect of initial conductivity and membrane number (c), effect of conductivity of concentrate and membrane number (d), effect of pH and voltage (e), effect of initial conductivity and voltage (f), effect of conductivity of concentrate and voltage (g), effect of Initial conductivity and pH (h), effect of conductivity of concentrate and pH (i), effect of conductivity of concentrated initial conductivity (k) on energy consumption (kWh/m^3).

passage of divalent ions, and may reduce the selectivity of membranes [48]. This has a negative effect on nutrient recovery efficiencies.

In addition, dynamically changing the current density achieved high efficiency, especially in ammonium recovery, compared to constant

current density, and at the same time reduced energy consumption [49]. These findings highlight the need to optimize the system according to the targeted performance in ED processes and the importance of maintaining the balance between current density and ion selectivity. It shows that in the ED process, current density is critical to increase energy efficiency and achieve optimum performance.

As a result of the study, it was observed that as the current density (B) increased, hydrogen production and nutrient recovery efficiency increased, and energy consumption also increased (Fig. 5a-7a-9a). In order to improve nutrient recovery efficiency and hydrogen production while reducing energy consumption, the effects of different parameters within the system should be investigated. In experimental studies, it was observed that when the current density increased from 3 to 9, hydrogen production increased by 487 %, nutrient recovery efficiency increased by 335 %, and energy consumption increased by 1412 %. It is seen that the experimental results obtained are compatible with the model results.

For energy consumption, voltage directly determines ion mobility and transport efficiency, but its impact varies across different conditions [45]. The interaction between voltage (B) and membrane number (A) shows a nonlinear relationship, where higher voltage leads to steep energy consumption increases at low membrane counts due to limited ion pathways and high system resistance. However, at higher membrane numbers, energy consumption stabilizes as enhanced ion mobility compensates for resistance effects, improving efficiency. Beyond a certain membrane count, further voltage increases yield diminishing returns as resistance buildup restricts current flow. Similarly, the interaction between voltage (B) and initial conductivity (D) demonstrates that higher conductivity lowers system resistance, enabling more efficient ion transport and reducing energy losses at high voltage levels. Conversely, at low conductivity, applying high voltage leads to a sharp rise in energy demand, as fewer mobile ions result in greater resistance. The interaction with the conductivity of concentrate (E) follows a similar trend, where low conductivity of concentrate requires increased voltage to maintain separation efficiency, but excessive voltage application in low-conductivity environments leads to unnecessary energy expenditure. Lastly, pH (C) variations exhibit only a minor effect on energy consumption, primarily influencing membrane selectivity and long-term system stability rather than immediate energy demand.

For hydrogen production, voltage remains the dominant factor, directly driving electrolysis reactions and current flow. The interaction between voltage (B) and membrane number (A) reveals that while increasing voltage enhances hydrogen production, additional membranes introduce electrical resistance, reducing current efficiency despite higher voltage application. At lower membrane counts, hydrogen yield benefits from improved current flow, while at higher membrane counts, resistance buildup limits production efficiency. Similarly, the interaction between voltage (B) and initial conductivity (D) exhibits a strong positive correlation, where higher initial conductivity enhances charge mobility, reducing system resistance and enabling more effective electrolysis. However, at very high conductivity levels, ion competition effects may reduce membrane selectivity, limiting hydrogen generation efficiency. The interaction with conductivity of concentrate (E) indicates that voltage is the primary determinant of hydrogen production, with conductivity of concentrate exerting only a minor effect by influencing ion transport selectivity rather than electrolysis efficiency. Lastly, the interaction with initial pH (C) suggests that while pH influences membrane integrity, it has no significant direct impact on hydrogen yield, confirming that voltage remains the key variable in electrochemical hydrogen generation.

For nutrient recovery, voltage plays a pivotal role in ion migration and membrane performance, with its effects varying based on membrane count, solution conductivity, and ion selectivity conditions. The interaction between voltage (B) and membrane number (A) follows a parabolic trend, where nutrient recovery initially increases with voltage but declines at excessive membrane counts due to increased system resistance and limited charge transfer efficiency. The interaction with

initial conductivity (D) highlights a strong synergistic effect, where higher conductivity enhances ion transport efficiency under elevated voltage conditions. However, beyond an optimal threshold, osmotic pressure effects and ion competition may reduce recovery selectivity. Similarly, the interaction with conductivity of concentrate (E) follows a nonlinear trend, where nutrient recovery improves with voltage but stabilizes at high concentrate conductivities, indicating that voltage remains the primary driver of nutrient separation, while conductivity of concentrate influences ion selectivity rather than overall efficiency. Lastly, the interaction with initial pH (C) shows that pH has only a secondary effect, where higher pH values may slightly improve recovery due to enhanced membrane charge interactions, but voltage optimization remains the dominant factor in achieving high nutrient separation efficiency.

From a broader perspective, voltage is the most dominant operational parameter in the ED process, directly influencing energy demand, hydrogen production, and nutrient recovery efficiency. However, its impact is not absolute and is strongly affected by membrane configuration, conductivity levels, and system resistance. While increasing voltage enhances electrochemical efficiency, excessive application can result in higher resistive losses, intensified ion competition, and performance limitations due to membrane constraints [50]. To optimize system performance while minimizing unnecessary energy expenditure, voltage must be precisely controlled and harmonized with other operational parameters, particularly membrane count and conductivity levels, ensuring a balanced approach that maximizes ion transport, electrolysis efficiency, and nutrient recovery selectivity.

3.8. Effect of initial conductivity

Initial conductivity (D) plays a pivotal role in the electrodialysis process by directly affecting ion transport rates, membrane resistance, and overall system efficiency [51]. As conductivity increases, enhanced charge mobility reduces resistance and improves separation performance. However, its influence is highly context-dependent, varying according to interactions with voltage (B), membrane number (A), conductivity of concentrate (E), and initial pH (C). Balancing these factors is critical to optimizing system performance, as excessively high conductivity can lead to ion competition effects and reduced selectivity.

ED performance is affected by several factors, including diluate conductivity, membrane properties, and operational parameters [52]. Initial salt concentrations with high conductivity significantly affect ion transport and membrane resistance in systems such as the ED. Excessively high initial concentration may lead to increased membrane resistance. In addition, this resistance on the membranes can cause the ion transfer to slow down and energy consumption to increase [53].

On the other hand, in different studies, the conductivity inside the cell increases as the initial conductivity increases, allowing the ions to pass more quickly and easily. Low intracellular resistance reduces electricity consumption and increases efficiency. This improves nutrient recovery performance, particularly in seawater desalination and high-salinity wastewater management, making systems more energy efficient [54]. Thus, it can increase nutrient recovery efficiencies by reducing energy consumption. These studies indicate the importance of understanding and optimizing the ED processes to improve efficiency and performance in different applications.

Initial conductivity (D) was determined as an important parameter affecting the ED process. As initial conductivity increases, energy consumption and hydrogen production increase (Figs. 7f, 9f), but this time the nutrient recovery efficiency decreases (Fig. 5f). In experimental studies, it was determined that when the initial conductivity was increased from 18 to 54, hydrogen production increased by 98 %, energy consumption increased by 101 %, while nutrient recovery efficiency decreased by 45 %. High initial conductivity values allow high current passage, thus high current causes high energy consumption and hydrogen production.

For energy consumption, higher initial conductivity enhances ion availability, allowing for more efficient charge transfer and reducing system resistance [51]. The interaction between initial conductivity (D) and membrane number (A) follows a nonlinear trend, where energy consumption initially decreases with increasing membranes due to enhanced ion exchange capacity. However, beyond a threshold, additional membranes introduce excessive resistance, leading to diminishing efficiency gains. Similarly, the interaction between initial conductivity (D) and voltage (B) shows that higher voltage operation is more efficient at elevated initial conductivity levels, as it minimizes ohmic losses. Conversely, at low initial conductivity, high voltage application leads to a sharp rise in energy demand, as fewer mobile ions result in higher system resistance. The interaction with the conductivity of concentrate (E) highlights that while high initial conductivity improves ion migration, excessive conductivity of concentrate may lead to ion competition, reducing selectivity and increasing energy consumption.

For hydrogen production, higher initial conductivity enhances ion mobility, improving charge transfer efficiency and increasing hydrogen yield. The interaction between initial conductivity (D) and membrane number (A) indicates that hydrogen production benefits from increased initial conductivity, particularly at lower membrane numbers. However, as membrane count rises, system resistance offsets the advantages of higher conductivity, reducing its effect. Similarly, the interaction with voltage (B) shows that hydrogen production increases significantly at higher initial conductivity and voltage levels, as reduced resistive losses facilitate more effective electrolysis. However, at very high conductivity, excessive ion competition may reduce membrane selectivity, leading to higher energy demand. The interaction with the conductivity of concentrate (E) follows a parabolic trend, where moderate initial conductivity provides the highest hydrogen production efficiency, while excessively high conductivity of concentrate may saturate ion exchange sites, reducing selectivity. Lastly, the interaction with initial pH (C) suggests that pH has a minimal impact on hydrogen production, primarily influencing membrane stability rather than electrolysis efficiency.

For nutrient recovery, initial conductivity is a key determinant in optimizing ion separation efficiency. The interaction with membrane number (A) follows a U-shaped trend, where moderate initial conductivity yields the highest recovery, while low conductivity limits ion availability and high conductivity increases ion competition, reducing selectivity. Similarly, the interaction with voltage (B) indicates that higher voltage improves nutrient recovery, particularly when initial conductivity is elevated, as it facilitates more efficient ion transport. However, excessive conductivity may introduce osmotic pressure and non-selective ion movement, reducing overall system selectivity. The interaction with conductivity of concentrate (E) follows a similar parabolic response, where both very low and very high conductivity levels negatively impact nutrient separation. At low conductivity of concentrate, limited ion exchange hinders separation, while high conductivity of concentrate leads to unwanted co-ion interference, reducing efficiency. The interaction with initial pH (C) follows a mildly increasing trend, suggesting that higher pH may slightly improve recovery efficiency by enhancing membrane charge stability and solubility, though this effect is secondary compared to conductivity and voltage influences.

Initial conductivity is a key determinant of ion transport efficiency in ED systems, playing a significant role in energy consumption, hydrogen production, and nutrient recovery. While an increase in conductivity generally facilitates improved charge mobility and reduced membrane resistance, excessively high conductivity can introduce ionic saturation, intensified competition, and selectivity reduction, ultimately affecting system efficiency [55]. To achieve optimal performance, it is crucial to adopt a multi-variable optimization approach, where initial conductivity is carefully coordinated with voltage, membrane configuration, and conductivity of concentrate to prevent energy losses, osmotic pressure imbalances, and inefficiency in ion separation while ensuring effective system operation.

3.9. Effect of conductivity of concentrate

Conductivity of concentrate (E) is a significant factor in determining the efficiency of energy consumption, hydrogen production, and nutrient recovery in the ED process. By influencing ion transport resistance, charge selectivity, and membrane efficiency, conductivity of concentrate plays a key role in overall system performance [56]. Its impact is closely linked to voltage (B), membrane number (A), initial conductivity (D), and initial pH (C), requiring careful optimization to prevent excessive ion competition and resistance buildup while ensuring stable and efficient electrodialysis operation.

The performance of the ED is affected by factors such as the initial concentration and the volume ratio between dilute and concentrate [57]. It has been determined that diluate conductivity has a more significant effect on process speed than conductivity of concentrate [58]. However, a study found that in membranes that are in contact with low-concentration liquids on one side and high-concentration liquids on the other side, the conductivity of the low-concentration liquid has a limiting effect. Low-concentration solutions increase membrane resistance, making ion transport difficult and negatively affecting system efficiency. A pair of cation and anion-selective membranes showed 10 to 14 times more resistance when in contact with 0.01 M NaCl solutions on one side and 1.0 M NaCl solutions on the other side than when both sides were in contact with 1.0 M NaCl solutions [59].

This result shows that low-concentration solutions seriously affect the conductivity performance of the membrane and increase the membrane resistance. In a different study, it was determined that when the NaCl concentration was increased from 0.1 M to 1 M, the resistance decreased by 30 % in cation-selective membranes and by 50 % in anion-selective membranes [60]. This effect, which can occur especially when working with low-concentration solutions in applications such as seawater desalination or desalination, is a critical factor that must be considered in terms of process optimization. During system design, the effects of low-concentration solutions on membrane resistance should be determined by considering their effects on the system.

In our study, another parameter, conductivity of concentrate (E), was determined, and its effects on the ED process were investigated. In experimental studies, when conductivity of concentrate was increased from 1 mS/cm to 5 mS/cm, hydrogen production increased by 9.5 %, energy consumption by 15 %, and nutrient recovery efficiency by 15 %. This is because low-concentration solutions reduce the ion-selective permeability of the membrane and create electrical resistance. Increased resistance causes ions to pass through the membrane more slowly. Nevertheless, despite the 5-fold increase in concentrated conductivity, it affects the results at low rates and is similar to the low effect rate given by the model (Figs. 4–9).

For energy consumption, increasing the conductivity of concentrate reduces system resistance, enhancing ion transport and lowering energy demand. The interaction with membrane number (A) shows that higher conductivity of concentrate moderately decreases energy consumption, particularly with higher membrane counts, where enhanced ion mobility facilitates charge transfer. However, at lower membrane numbers, the effect is weaker due to limited ion exchange surface area. Similarly, the interaction with voltage (B) exhibits a nonlinear trend, where energy consumption rises sharply at high voltages when conductivity of concentrate is low due to increased ionic resistance. In contrast, at higher conductivity of concentrate levels, system efficiency improves, reducing the need for excess voltage. The interaction with initial conductivity (D) further highlights that excessive conductivity of concentrate may lead to ion competition, increasing co-ion migration and energy demand in high-salinity environments.

For hydrogen production, the conductivity of concentrate has only a minor direct effect, with voltage and initial conductivity playing dominant roles. The interaction with membrane number (A) suggests that hydrogen yield remains stable across different conductivity of concentrate values, reinforcing that voltage-driven electrolysis is the

primary mechanism. The interaction with voltage (B) confirms that higher voltage significantly enhances hydrogen production, while variations in conductivity of concentrate introduce only slight fluctuations. Similarly, the interaction with initial conductivity (D) indicates that higher initial conductivity enhances charge transfer efficiency, but conductivity of concentrate primarily affects membrane resistance rather than hydrogen generation. Additionally, its interaction with initial pH (C) shows minimal impact, reinforcing that hydrogen production is largely unaffected by conductivity of concentrate variations.

For nutrient recovery, the conductivity of concentrate plays a more significant role in ion selectivity and separation efficiency. The interaction with membrane number (A) reveals a parabolic relationship, where both very low and very high conductivity of concentrate values reduce recovery efficiency due to increased resistance or excessive ion competition. Similarly, the interaction with voltage (B) confirms that voltage remains the primary driver of nutrient recovery, while high conductivity of concentrate can cause co-ion interference, slightly reducing efficiency. The interaction with initial conductivity (D) suggests that while higher initial conductivity improves ion mobility, excessive conductivity of concentrate may induce ion crowding, reducing selectivity. Lastly, the interaction with initial pH (C) shows only minor effects, where higher pH slightly enhances nutrient recovery due to improved ion solubility and membrane charge interactions.

Overall, conductivity of concentrate is a secondary but essential parameter in ED system optimization, primarily modulating ion transport, membrane resistance, and charge selectivity. While its impact on hydrogen production is minimal, it significantly influences energy efficiency and nutrient recovery. To maximize ED performance, conductivity of concentrate must be carefully balanced with voltage, membrane configuration, and initial conductivity to avoid excessive ion competition, resistance buildup, and selectivity losses. A multi-variable optimization approach is necessary to ensure high system efficiency while minimizing energy costs and maintaining stable long-term operation.

On a broader scale, conductivity of concentrate serves as a critical modulating factor in ED system optimization, influencing ion transport resistance, membrane efficiency, and charge selectivity [61]. Although its direct influence on hydrogen production is minimal, it has a substantial impact on energy efficiency and nutrient recovery performance. To optimize ED operations, conductivity of concentrate must be carefully adjusted alongside voltage, membrane configuration, and initial conductivity, ensuring that excessive ion competition, resistance buildup, and selectivity degradation are minimized. Employing a multi-variable optimization framework is essential for achieving high system efficiency, reduced energy consumption, and long-term operational stability.

3.10. Validation analysis of RSM optimization

In this study, desirability response surface methodology was employed to determine the optimal conditions by evaluating all independent variables based on model parameters (Fig. 10). The desirability index ranges from 0 to 1, where 1 represents the most favorable conditions. Initially, the optimization was conducted separately for energy consumption, nutrient recovery, and hydrogen production, followed by validation experiments to confirm the model's accuracy. Subsequently, a comprehensive optimization was performed, considering all three parameters simultaneously to identify a single optimal condition, which was further validated through experimental trials. To ensure reliability, each validation experiment was conducted in triplicate, enhancing the accuracy and reproducibility of the results.

This study conducted process optimization using a model-based approach, followed by verification experiments to ensure accuracy. The optimization aimed to achieve high recovery efficiency while minimizing energy consumption, and the process continued until the conductivity value reached 1 mS/cm, corresponding to approximately 97 % recovery efficiency.

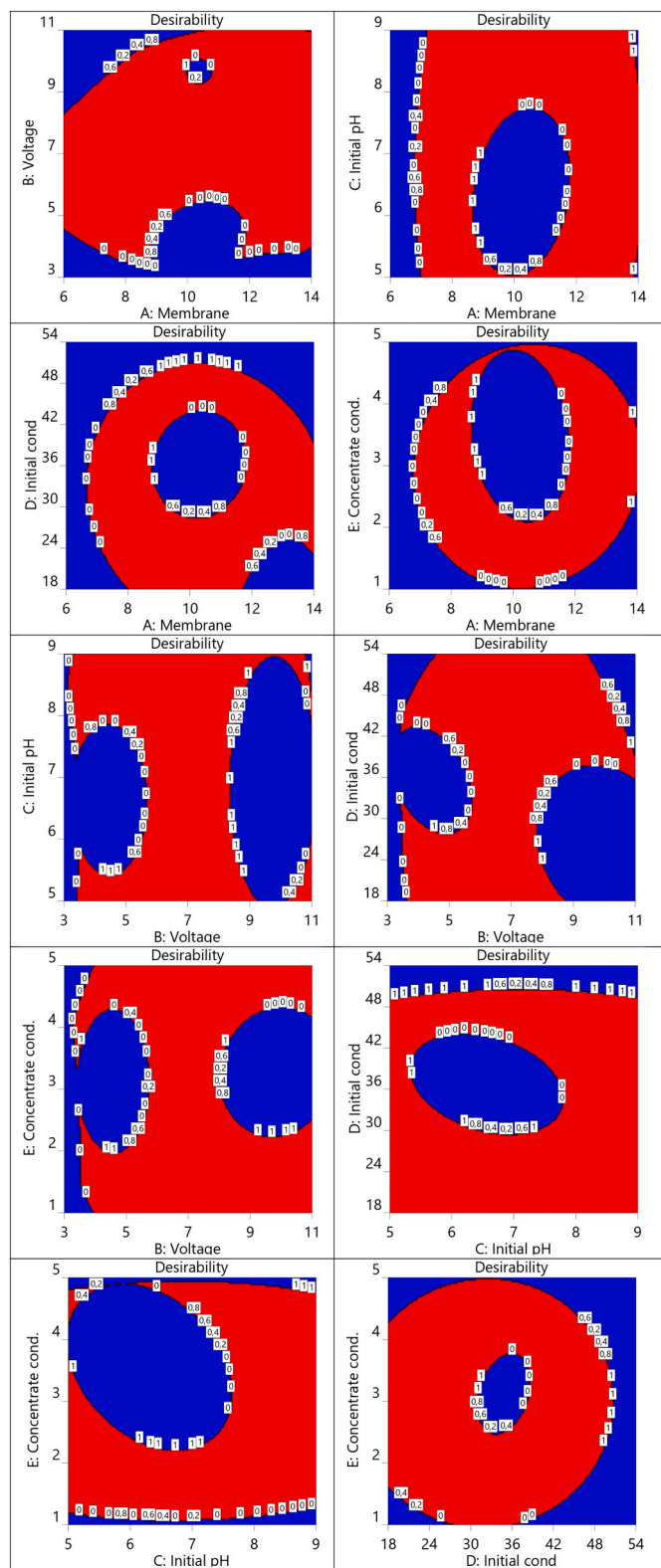


Fig. 10. Desirability responses of variables in the model.

For minimum energy consumption, the model identified the optimal parameters as A: number of membrane cells = 11, B: voltage = 5 V, C: initial pH = 7, D: initial conductivity = 38 mS/cm, and E: conductivity of concentrate = 4 mS/cm. Under these conditions, 97 % recovery efficiency was achieved with an energy consumption of 4.72 kWh/m³. However, achieving this efficiency requires an extended recovery time

of 120 min, which may present limitations for practical applications.

For maximum recovery efficiency, the model determined the optimal parameters as A: number of membrane cells = 11, B: voltage = 9 V, C: initial pH = 7, D: initial conductivity = 38 mS/cm, and E: conductivity of concentrate = 3 mS/cm. This configuration allowed 98 % recovery efficiency to be reached in only 35 min, significantly reducing the required nutrient recovery time. However, this improvement came at the cost of higher energy consumption, increasing to 6.61 kWh/m³.

To balance energy efficiency, nutrient recovery effectiveness, and hydrogen production, the model identified the overall optimum operating parameters as A: number of membrane cells = 8, B: voltage = 9 V, C: initial pH = 6, D: initial conductivity = 27 mS/cm, and E: conductivity of concentrate = 2 mS/cm. Under these conditions, 96 % recovery efficiency was achieved in 35 min with an energy consumption of 4.96 kWh/m³, demonstrating a well-optimized trade-off between performance and energy demand.

To maximize hydrogen production, the optimal parameters were determined as A: number of membrane cells = 8, B: voltage = 9 V, C: initial pH = 6, D: initial conductivity = 45 mS/cm, and E: conductivity of concentrate = 4 mS/cm. Under these conditions, 96 % recovery efficiency was achieved in 50 min, producing 472 mL of hydrogen. However, the energy consumption increased to 12.9 kWh/m³, indicating a significant trade-off between hydrogen yield and power demand. In contrast, under the optimum conditions determined by the model, 135 mL of hydrogen was produced with 4.96 kWh/m³ energy consumption, making it a more energy-efficient approach.

According to the results, maximum hydrogen production corresponded to 98.28 kJ/mL H₂, whereas the optimum operating conditions yielded 132.48 kJ/mL H₂. These findings highlight the importance of optimizing operational parameters to recovery efficiency, energy consumption, and hydrogen yield, making the process more viable for practical and industrial applications. The results further demonstrate that while higher hydrogen production can be achieved by increasing voltage and initial conductivity, energy consumption also increases significantly, necessitating a strategic trade-off in system design.

Recent studies have demonstrated the feasibility of hydrogen production through membrane-based electrochemical processes, particularly reverse electrodialysis (RED) and ED, under varying operational conditions. Ji-Hyung Han et al. (2019) explored a RED-driven water electrolysis system, highlighting that the voltage generated from the salinity gradient increases with the number of alternating cation and anion exchange membrane pairs. Their system, incorporating over 50 membrane pairs, achieved a voltage exceeding 10 V, enabling water electrolysis without overpotential minimization strategies. Notably, a hydrogen production rate of 1.1×10^{-4} mol.cm⁻².h⁻¹ was reported using neutral-pH water and artificial NaCl feed solution, demonstrating a cost-effective and eco-friendly alternative to previous, RED-based hydrogen production studies [62].

Expanding upon this, Higa et al. (2019) developed a pilot-scale RED system, utilizing the salinity gradient energy (SGE) between synthetic seawater (SW) and sewage-treated water (STW). Their system, featuring 200 membrane pairs, generated an open-circuit voltage of 28.6 V, with a maximum power output of 15.3 W at 1.4 A. Under a constant current of 1.5 A, the system achieved a hydrogen production rate of 0.90 L/h, exhibiting nearly 100 % current efficiency over 1100 h of stable operation. However, despite its efficiency, no nutrient recovery strategies were implemented, as treated wastewater was directly utilized after filtration [63].

Beyond RED, ED has also been investigated as a hydrogen production method, particularly in terms of energy efficiency and salt removal performance. da Costa Fonseca et al. (2024) examined hydrogen generation via conventional ED, revealing that NaCl concentration in the feed and electrolyte had minimal impact on H₂ and O₂ generation, with hydrogen production primarily governed by current density and membrane ion transport resistance. The study established that energy consumption is directly proportional to applied current and inversely

proportional to salt concentration, with 35 g/L NaCl yielding the lowest energy demand. Hydrogen production ranged from 0.04 to 0.22 m³/h. m², with specific hydrogen generation reaching 21.6 mL/h·cm² electrode in a bench-scale ED system. These findings provide valuable insights into optimizing ED for hydrogen production, reinforcing the significance of operating conditions and membrane characteristics in enhancing system efficiency [64].

Further advancing this approach, Alshebli (2023) investigated hydrogen recovery during Na₂SO₄ desalination via ED, demonstrating that hydrogen collection does not compromise desalination efficiency. The system exhibited a 95.2 % conductivity removal ratio, with additional improvements observed through ion-exchange resin integration, yielding a maximum hydrogen production rate of 76.8 mg H₂/h·kg Na₂SO₄ at pH 8. Moreover, it was shown that conventional ED can simultaneously desalinate saline water and produce hydrogen, with hydrogen generation reaching 118.8 mg H₂/h·kg Na₂SO₄. While energy consumption increased from 9.9 to 10.5 kWh/m³ to 12.4 kWh/m³ due to ion-exchange resin addition, this also correlated with higher hydrogen yields. The study further reported maximum energy and exergy efficiencies of 25.29 % and 28.78 %, highlighting the potential of ED for integrated desalination and hydrogen production applications [65].

Another key aspect of our study is nutrient recovery, which has been extensively investigated in the literature. Numerous studies have explored ED as an energy-efficient and sustainable approach for nutrient recovery from wastewater, positioning it as a viable alternative to conventional treatment methods. This process enables the selective recovery of essential nutrients, including nitrogen, phosphorus, and potassium, from diverse wastewater streams such as municipal effluents, urine, and digestate [66]. The performance of ED can be significantly enhanced by optimizing operational parameters, including applied voltage, membrane surface area, and the number of cell pairs [67].

Recent advancements in ED technology, particularly the development of selective ion-exchange membranes, have facilitated the simultaneous fractionation of various nutrient ions, thereby enabling the production of high-value recovered products [68]. Moreover, multi-stage ED configurations have demonstrated improved nutrient recovery efficiencies and concentration factors while ensuring that the treated effluent meets stringent water quality standards [67]. Nevertheless, challenges such as membrane fouling and energy consumption remain critical barriers to large-scale implementation, necessitating further research to enhance the long-term feasibility and operational efficiency of ED in nutrient recovery applications [66,67]. ED for nutrient recovery varies significantly depending on operational parameters such as the number of cell pairs, processing time, and wastewater characteristics. Previous studies have reported recovery efficiencies ranging from moderate to high, with longer operation times and larger membrane areas generally leading to improved performance. For instance, studies on swine manure and anaerobic digester supernatant demonstrated

moderate recovery rates over extended durations, while synthetic wastewater and aqueous solutions showed a broader range of efficiencies depending on system configurations (Table 5) [65–67].

In comparison, the findings of this study indicate that high nutrient recovery efficiencies (96–98 %) were achieved within relatively short operation times (35–120 min) and with fewer cell pairs (9–12). This contrasts with some earlier studies that required significantly longer operation durations or larger membrane areas to achieve similar or lower recovery rates. The results suggest that the optimized batch ED system used in this study is highly effective for nutrient recovery, demonstrating competitive performance even with a reduced number of cell pairs and shorter treatment durations. These findings highlight the potential of carefully controlled batch ED processes to enhance nutrient recovery efficiency while maintaining operational feasibility.

4. Conclusions

An experimental set was determined and analyzed according to model data for optimum results. Several analyses were performed, and the optimal set of values was determined as voltage 9 V, membrane number 12, initial conductivity 27 mS/cm, conductivity of concentrate 4 mS/cm, and initial pH 6, with repeated analyses, and the average results were obtained for 30 min. For the model experimental set, hydrogen production, nutrient recovery efficiency, and energy consumption results were 120.25 mL, 4.72 kWh/m³, and 89.98 %, respectively. The analyses were found to be 126 mL, 4.78 kWh/m³, and 91.20 %, respectively. When the analysis results were compared with the model results, the error margins were calculated as 4.5 % for hydrogen production, 1.3 % for nutrient recovery efficiency, and 1.2 % for energy consumption. As a result of the model study, the highest error margin was found in hydrogen production, which was below 5 %. It is thought that the inability to adjust conductivity and pH values precisely also affects these error margins. With the model optimization, energy consumption was reduced by 24 %, and the same efficiency was achieved in a 70 % shorter time. 25.8 % less energy consumption was achieved for 1 mL of hydrogen production for hydrogen production. These results show that the model is compatible with ED. The effect rates of the parameters according to the purpose of ED usage were revealed analytically. A comprehensive study on the optimization of 5 different parameters giving 3 different results with RSM was presented, and the gap in the literature was filled. Future studies can focus on the effects of other parameters, especially the effects of time and ion types with larger data points.

CRediT authorship contribution statement

Sinan Ates: Writing – original draft, Methodology, Investigation, Formal analysis. **Asifa Anwar:** Validation, Formal analysis. **Ayşe Elif**

Table 5
Comparison of nutrient recovery efficiency in electrodialysis systems under various wastewater conditions [67].

Wastewater	Number of cell pairs	Operation conditions	Time (min.)	Nutrient recovery efficiency, %	Effective area per membrane (cm ²)	Ref
Swine manure	3	10 batch mode,	600	65.38	100	[69]
Domestic anaerobic digester supernatant	30	Flow rate of 1250 mL/min (75 L/h)	4320	88.40	72,000	[70]
Aqueous solutions	110	30 L	40	50	390	[71]
Synthetic wastewater	110	19 L/h; 10 batch system	250	85	16	[49]
Synthetic wastewater	12	11 batch system, 0.5 L, 4–8 L/h	120	98		This study ¹
Synthetic wastewater	12	11 batch system, 0.5 L, 4–8 L/h	35	96	64	This study ²
Synthetic wastewater	9	10 batch system, 0.5 L, 4–8 L/h	50	97		This study ³

¹ Minimum energy consumption.

² Maximum recovery efficiency.

³ Maximum hydrogen production.

Ates: Software, Methodology. **Burak Yuzer:** Writing – review & editing. **Yusuf Bicer:** Writing – review & editing, Supervision.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.jwpe.2025.107390>.

Data availability

Data will be made available on request.

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