

EXPERIMENTAL ANALYSIS OF ELECTRODEIONIZATION A D.O.E APPROACH

ABHIRAM SUBRAMANIAN¹, M. SHIREESHA¹, PADAKANTI SAITEJA¹, P. SARATH¹, ARADHYULA JATIN BHANU SHANKAR¹ and V. SESA SAI KUMAR²

¹Department of Chemical Engineering, Anurag University, Ghatkesar, Medchal (Dist.), Hyderabad, Telangana, India.

²Department of Physics, Guru Nanak Institute of Technology, Ibrahimpatnam, Telangana, India.

Abstract:

Electro deionization is an ionic separation process that originally debuted 50 years ago. Initially, it was employed to separate metallic species from radioactive wastes. Electro deionization has been proved to be an environmentally beneficial method of purification, separation, and concentration. The EDI method is especially effective in terms of environmental protection, the production of ultra-pure water, and the recovery of some rare species. Weakly ionised ions, such as carbon dioxide and boron, are typically difficult to extract using membrane procedures such as reverse osmosis and electro dialysis reversal (EDR) The EDI has the benefit of continuously eliminating these germs in a highly efficient manner. The experimental investigation and mathematical modelling of an electro deionization process used for brackish water desalination in a four-compartment laboratory EDI cell, as well as the effect of varying concentrations (0.01 and 0.05 M) and cell voltage, are described in this paper (7, 14 V). The developed model includes anion exchange in the resin bed, which is controlled by the mass transport rate; ion transport in resin-free compartment solutions, membranes, and resin, which is based on the Nernst-Planck equation; and enhanced water dissociation at the anion membrane/solution interface. To compute the values, a pre-existing Simulation Model for Electro Deionization from COMSOL Multiphysics is utilised, which is then used in Design Expert 13 to perform statistical analysis of the system in order to achieve the best value and decrease the number of experiment runs.

Introduction to the topic:

Aim and objective:

Electro deionization is a relatively recent water treatment method. It deionizes brackish water using electricity and membrane technology. The primary goal of this project is to reduce the number of experimental runs and identify the most influential component.

There are many factors that affect the system, but out of all the factors, we have taken mainly 4 factors into consideration which we assumed from literature studies have a major effect on the system, namely, Voltage Concentration of feed Temperature and Flow rate. Our objective is to produce the results from COMSOL Multiphase software by using a pre-existing model of electro deionization system and computing the values within the limits deduced from the literature review. Utilizing Design Expert 13 software, analyse the experimental data acquired from COMSOL. The final consequence is that the optimum values are obtained with fewer trial runs.

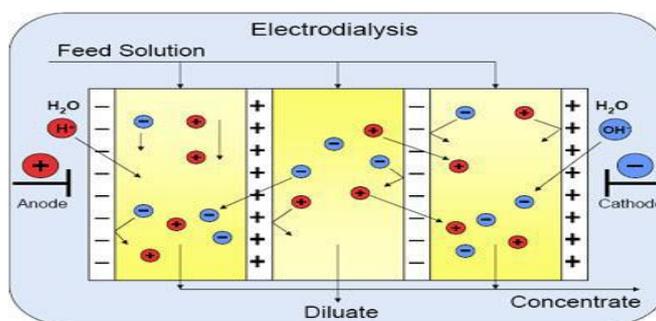
Overview of Electro Deionization:

Electro deionization was developed in the late 1950s to eliminate or reduce the concentration polarisation phenomena found in electro dialysis devices. At the International Water Conference in 1956, William Katz of Ionics provided one of the earliest descriptions of electro deionization and submitted his paper "The Present Status of Electric Membrane Demineralization." Due to the low tolerance of hardness and organics, the technology's applicability was limited. In the 1970s and 1980s, reverse osmosis was the chosen approach for ion exchange resin in high TDS waters. As RO became more popular, it was thought that EDI would be an appropriate polishing method. Chemically regenerated ion exchange systems were replaced by packaged RO and EDI systems. Companies like as Millipore, Ion Pure, and Ionics Inc. produced electro deionization devices between 1986 and 1989. The first devices were huge, expensive, and sometimes faulty. Glegg Water Conditioning launched the E-Cell brand electro deionization in 1995. Based on a modular design standard, the new technology cut costs while improving dependability. Many OEMs were also provided E-Cell, which transformed the industry. Competitors quickly followed with leak-free modular designs. Currently, numerous water treatment firms provide this technology, but it should only be used by specialists who understand the restrictions and utilise high-quality materials.

What is Electro deionization?

Electro Dialysis:

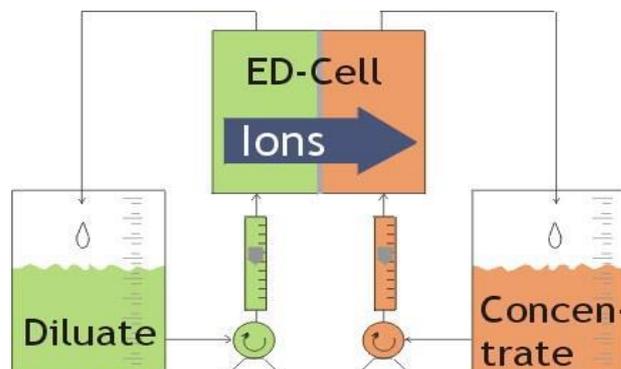
Electro dialysis (ED) is a method of transporting salt ions from one solution to another using ion-exchange membranes and an applied electric potential difference. This is done in a device known as an electro dialysis cell. The cell is divided into two compartments: a feed (dilute) compartment and a concentrate (brine) compartment, which are separated by an anion exchange membrane and a cation exchange membrane situated between two electrodes. Almost all practical electro dialysis procedures use multiple electro dialysis cells organised in a stack, with alternating anion and cation-exchange membranes creating the multiple electro dialysis cells. Distillation methods and other membrane-based processes (like as reverse osmosis (RO)) vary from electro dialysis procedures in that dissolved species are transported away from the feed stream rather than back. Because the amount of dissolved species in the feed stream is substantially smaller than that of the fluid, electro dialysis has the practical benefit of significantly better feed recovery in many applications.



Electro Dialysis Method:

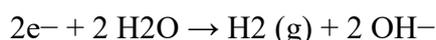
The dilute (D) input stream, brine or concentrate (C) stream, and electrode (E) stream are all permitted to pass through the proper cell compartments produced by the ion-exchange membranes in an electro dialysis stack. Negatively charged ions (e.g., chloride) in the dilute stream move toward the positively charged anode as a result of an electrical potential difference. These ions pass through the positively charged anion-exchange membrane but are stopped from migrating further toward the anode by the negatively charged cation-exchange membrane, and so remain in the C stream, which gets concentrated with the anions. The positively charged species in the D stream (for example, sodium) move toward the negatively charged cathode and pass through the negatively charged cation-exchange membrane. The positively charged anion-exchange membrane keeps these cations in the C stream and prevents them from migrating to the cathode. Electric current travels between the cathode and anode as a result of anion and cation migration. Because only an equal number of anion and cation charge equivalents are transported from the D stream to the C stream, the charge balance in each stream is maintained. The electro dialysis process produces an increase in ion concentration in the concentrate stream while depleting ions in the dilute solution input stream.

The electrode stream is the E stream, which goes past each electrode in the stack. This stream might be the same as the feed stream (e.g., sodium chloride) or it could be a distinct solution containing a different species (e.g., sodium sulphate). Anions and cations from the electrode stream may be carried into the C stream, or anions and cations from the D stream may be transported into the E stream, depending on the stack arrangement. This transport is required in each case to convey current across the stack and preserve electrically neutral stack solutions.

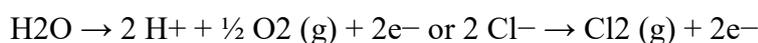


Anode and Cathode Reactions in Electro Dialysis:

Reactions take place at each electrode. At the cathode,



While at the anode,

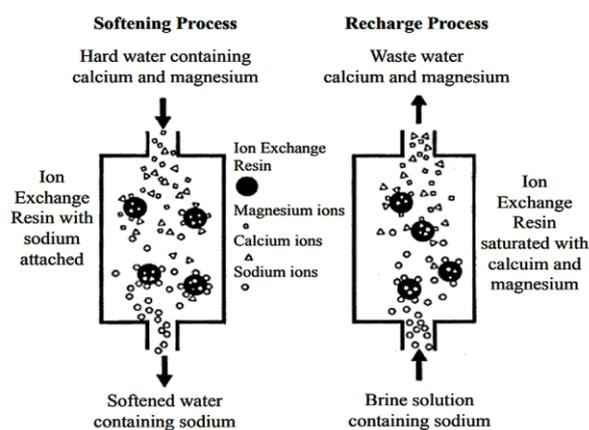


Minor amounts of hydrogen gas are produced at the cathode, while small amounts of either oxygen or chlorine gas are produced at the anode (depending on the composition of the E stream and the end ion-exchange membrane configuration). These gases are normally dissipated after each electrode compartment's E stream effluent is mixed to maintain a neutral pH and discharged or re-circulated to a separate E tank. Some (for example,) have advocated collecting hydrogen gas for use in energy generation.

Why there is a need of Ion Exchangers:

Ion exchange is the reversible exchange of one kind of ion existing in an insoluble solid with another of like charge present in a solution surrounding the solid, with the process being employed mostly for softening or demineralising water, chemical purification, and material separation.

Ion exchange resins (functionalized porous or gel polymer), zeolites, montmorillonite, clay, and soil humus are examples of common ion exchangers. Ion exchangers are classified as either **cation exchangers** (which exchange positively charged ions) or **anion exchangers** (which exchange negatively charged ions) (anions). There are also amphoteric exchangers that can exchange both cations and anions at the same time. However, the simultaneous exchange of cations and anions is frequently carried out in mixed beds that include a mixture of anion- and cation-exchange resins or by passing the solution through multiple distinct ion-exchange materials.



Ion Exchange resins:

An ion exchange resin is a polymer (plastic) that is often utilised as a tiny bead (approximately 0.5mm [500]) with a permanent, insoluble matrix and a moveable or replaceable mobile ionic component that may be swapped for other mobile ions in solution. The degree of exchange is a variable influenced by the previously described selectivity. Calcium, for example, produces a divalent ion (Ca^{+2}) that is more attractive to the exchange matrix than monovalent sodium (Na^{+1}). As a result, if a Ca^{+2} solution is poured over a bed of Na^{+1} resin, the matrix will pick up the more selective Ca^{+2} while releasing the less tightly held Na^{+1} . This is how and why softeners function.

Reaction 5: $\text{Ca}(\text{HCO}_3)_2 + \text{O-Na} \leftrightarrow \text{O-Ca} + \text{NaHCO}_3$ the softening reaction. The \leftrightarrow indicates that the reaction is reversible. **Reaction 6:** $\text{O-Ca} + \text{NaCl} \leftrightarrow \text{O-Na} + \text{CaCl}_2 + \text{NaCl}$ the regeneration reaction showing excess salt used.

Direct & indirect effect of Electro deionization:

Applications of Electro Dialysis:

In practise, electro dialysis devices may be used in either continuous or batch production operations. In a continuous process, feed is routed through a sufficient number of stacks in sequence to provide the desired product quality. The dilute and/or concentrate streams are re-circulated through the electro Dialysis systems in batch operations until the ultimate product or concentrate quality is obtained.

Electro dialysis is commonly used to deionize aqueous solutions. Desalting of sparsely conductive aqueous organic and organic solutions, on the other hand, is achievable. Electro dialysis has several applications, including:

- Large-scale desalination of brackish and saltwater, as well as salt manufacturing.
- Production of drinking water on a small and medium scale (e.g., towns & villages, construction & military camps, nitrate reduction, hotels & hospitals)
- Water re-use (for example, desalination brine treatment, industrial laundry effluent, generated water from oil/gas production, cooling tower makeup and blow down, metals industry fluids, and wash-rack water)
- Pre-demineralization (e.g., boiler makeup & pre-treatment, ultrapure water pre-treatment, process water desalination, power generation, semiconductor, chemical industry, food and beverage) (e.g., boiler makeup & pre-treatment, ultrapure water pre-treatment, process water desalination, power generation, semiconductor, chemical manufacturing, food and beverage)
- Food processing
- Agricultural water (e.g., water for greenhouses, hydroponics, irrigation, livestock)
- Glycol desalting (e.g., antifreeze / engine-coolants, capacitor electrolyte fluids, oil and gas dehydration, conditioning and processing solutions, industrial heat transfer fluids secondary coolants from heating, venting, and air conditioning (HVAC))
- Glycerine purification

Limitations:

Electro dialysis has inherent limitations in that it is best suited for eliminating low molecular weight ionic components from a feed stream. Non-charged, greater molecular weight and less mobile ionic species are often not eliminated substantially. In addition, unlike RO, electro dialysis becomes less economical when extremely low salt concentrations in the product are required, as well as with sparingly conductive feeds: current density becomes limited, and

current utilisation efficiency typically decreases as the feed salt concentration decreases, and with fewer ions in solution to carry current, both ion transport and energy efficiency suffer. As a result, to meet capacity requirements for low concentration (and sparsely conductive) input solutions, relatively wide membrane surfaces are necessary. There are innovative systems available that overcome the fundamental constraints of electro dialysis (and RO); these 11 integrated systems function synergistically, with each sub-system working in its optimal range, resulting in the lowest total operational and capital costs for a specific application.

As with RO, feed pre-treatment is required for electro dialysis systems to remove species that coat, precipitate onto, or otherwise "foul" the surface of the ion-exchange membranes. This fouling reduces the electro dialysis system's efficiency. Calcium and magnesium hardness, suspended particles, silica, and organic compounds are also of concern. Hardness may be removed by water softening; while suspended materials can be removed using micrometre or multimedia filtering. Hardness is a particular problem because scaling can accumulate on the membranes. A variety of compounds are also available to aid with scaling prevention. Furthermore, electro dialysis systems attempt to reduce scaling by frequently reversing the fluxes of dilute and concentrate as well as the polarity of the electrodes.

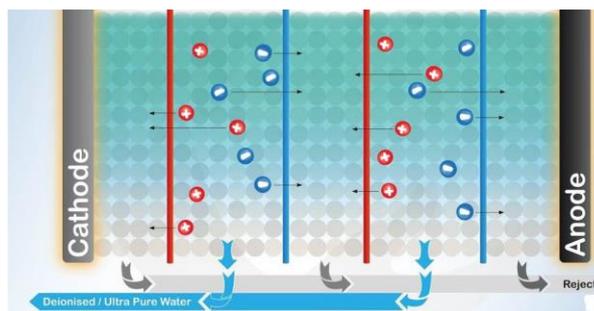
Emergence of Electro deionization:

Electro deionization is a hybrid method that combines two technologies, namely electro dialysis and ion exchange. Dynamic synergy between these two roles assists to mitigate each function's inherent weaknesses (e.g., concentration polarisation phenomena and chemical regeneration). The combination of these characteristics has resulted in a system that can handle low ionic strength solutions far more effectively than the constituent procedures alone. An EDI cell is made up of cationic and anionic membranes that are alternately positioned between the anode and cathode to create dilute and concentrated compartments.

Electro deionization (EDI) is a water treatment process that deionizes water and separates dissolved ions (impurities) from it by using electricity, ion exchange membranes, and resin. It is distinct from other water purification methods in that it is performed without the use of chemical treatments and is typically used as a polishing treatment to reverse osmosis (RO). There are other EDI machines that are known as continuous electro deionization (CEDI) since the electric current continually regenerates the resin mass. CEDI can attain very high purity, with conductivity as low as 0.1 $\mu\text{S}/\text{cm}$.

An electrode in an electrochemical cell is known as an anode or a cathode, words invented by Michael Faraday. The anode is the electrode where electrons leave the cell and oxidation takes place, whereas the cathode is the electrode where electrons enter the cell and reduction takes place. Depending on the voltage given to the cell, each electrode can become either the anode or the cathode. A bipolar electrode is one that serves as the anode of one cell and the cathode of another. Each cell is made up of an electrode and an electrolyte containing ions that are either oxidised or reduced. An electrolyte is a material that contains free ions and acts as a channel for electrical conduction. Electrolytes are also known as ionic solutions since they typically consist of ions in solution; however molten electrolytes and solid electrolytes are also

conceivable. They are frequently referred to as lytes in shortened parlance. Water is transferred between an anode (positive electrode) and a cathode (negative electrode) (negative electrode). Positive ions are directed toward the negative electrode by ion-selective membranes, whereas negative ions are directed toward the positive electrode. The end product is high quality deionized water.



Applications of Electro Deionization:

When fed with low total dissolved solids (TDS) feed (e.g., RO feed), the product can achieve extremely high purity levels (e.g., [[Purified water/Electrical Conductivity|18 mega ohms/cm], Resistivity / Conductivity Measurement of Purified Water. Ion exchange resins operate to hold ions, enabling them to pass through the ion exchange membranes. Electronics, medicines, and power generation are the primary uses of EDI technology, such as those provided by Ion Pure, E-cell, and Snow Pure.

What are the equipment and reagents required?

The electro deionization cell was constructed as shown in Fig. 2. The anode was a platinized titanium plate, and the counter-electrode was a stainless-steel plate, both had a geometric area of 103 cm² (30 cm²). With 103 cm² of exposed geometric area on each side, two Nafion cation exchange membranes and a Selemion anion exchange membrane were employed. Lewatit S-100 and M-500 mixed ion exchange resin bed volume was 18 cm³ (103 × 0.6 cm). There were also three plastic membrane spacers, four neoprene gaskets, and three Teflon gaskets utilised. Figure 3 depicts a schematic of the EDI cell and the recirculation mechanism used. To avoid direct contact with NaCl solutions, rinse solution chambers were integrated into the EDI cell at both electrodes, CS for cathode solution and AS for anode solution. This configuration is intended to avoid the production of chlorine in the anode, and hence the corrosion of the electrode. Though some chlorine ions can get through the cation exchange membrane and into the anode compartment, anode corrosion is projected to be minor due to the selected electrode material and the lack of extreme temperature, chloride concentration, and current density circumstances. The recirculation system consisted of three independent acrylic reservoirs (continuous stirring tanks, CST) with 4 L capacity, two of which were used to desalinate the NaCl solutions (one to dilute and the other to concentrate them) and the third CST was used for NaNO₃ rinse solution; then, three rotameter-type Omega FL-2024 flow meters (0 to 20 gal h⁻¹ capacity), and three Little Giant magnetic pumps at 1/30 hp. Three glass coils were employed to keep the temperature constant at 25 ± 1 °C; each reservoir

had a glass coil, which was linked in series to a circulating water bath (Brookfield, TC-502D and Julabo, Vivo RT4). A BK Precision 1672 DC power supply was utilised to provide voltage, and an electro dialysis cell system was coupled to a 34410A Agilent Digital Multi metre. An OAKTON PC 450 portable metre kit was used to measure pH and conductivity. A VARIAN type SpectrAA-200 atomic absorption spectrometer was used to determine Na⁺. Two 1.5 L solutions of 0.01 M NaCl, two 1.5 L solutions of 0.02 M NaCl, synthetic solutions that mimic brackish water, and a 1.5 L rinse solution of 0.05 M NaNO₃ were used; all were made with deionized water with a conductivity of 1 10⁻⁶ S cm⁻¹ and analytical grade chemicals. An OAKTON PC 450 portable metre kit was used to measure pH and conductivity. A VARIAN type SpectrAA-200 atomic absorption spectrometer was used to determine Na⁺. Two 1.5 L solutions of 0.01 M NaCl, two 1.5 L solutions of 0.02 M NaCl, synthetic solutions that mimic brackish water, and a 1.5 L rinse solution of 0.05 M NaNO₃ were used; all were made with deionized water with a conductivity of 1 10⁻⁶ S cm⁻¹ and analytical grade chemicals.

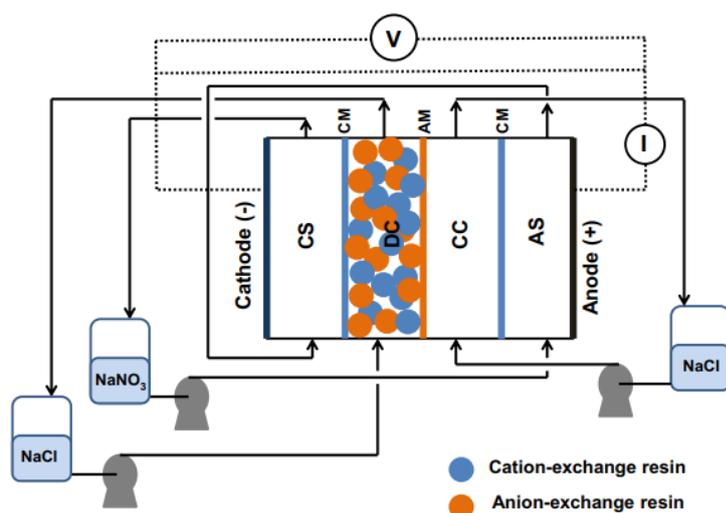


Fig 3.1: An exploded view of the electro deionization cell with a mixed bed resin

- A. Platinized titanium anode
- B. Spacer
- C. Teflon gaskets
- D. Stainless steel cathode
- E. Neoprene gaskets
- F. Plastic spacer
- G. Nafion cation exchange membrane
- H. Lewatit S-100 (cation) and M-500 (anion) mixed ion exchange resin
- I. Selemion anion exchange membrane.

Fig 3.2: Schematic of the experimental system for NaCl removal in EDI with a mixed bed resin process. The cell is made up of four compartments delimited by two exchange membranes (CM) and one anion exchange membranes (AM)

Methodology & Experimental Approach:

COMSOL Multiphysics software:

COMSOL Multiphysics is a finite element analysis, solver, and Multiphysics simulation programme that run on several platforms. It supports standard physics-based user interfaces as well as linked systems of partial differential equations (PDEs). COMSOL offers an integrated development environment (IDE) and a single workflow for electrical, mechanical, hydrodynamic, acoustical, and chemical applications.

Fig4.1 COMSOL Multiphysics

In addition to solving traditional problems using application modules, the basic Multiphysics package can solve PDEs in weak form. To control the software externally, an API for Java and Live Link for MATLAB and Autodesk Inventor are available. An Application Builder may be used to create standalone, domain-specific simulation programmes. Users have the option of using drag-and-drop tools (Form Editor) or programming (Method Editor). COMSOL Server is a separate piece of software used to handle COMSOL simulation systems in businesses. Several COMSOL modules are available, divided into the application categories of Electrical, Mechanical, Fluid, Acoustic, Chemical, Multipurpose, and Interfacing.

Multiphysics Simulation:

Multiphysics simulation (sometimes simplified to simply "Multiphysics") is described in computational modelling as the simultaneous simulation of several components of a physical system or systems. A Multiphysics simulation, for example, would be the simultaneous simulation of physical stress on an item and the temperature distribution of the object. Multiphysics simulation is connected to multiscale simulation, which is the simulation of a single process on many time or distance scales at the same time.

Multiphysics simulation, being an interdisciplinary area, may cover numerous science and engineering fields. Numerical analysis, partial differential equations, and tensor analysis are all common simulation methodologies.

- The implementation of a Multiphysics simulation follows a typical series of steps:
 - Identify the aspects of the system to be simulated, including physical processes, starting conditions, and boundary conditions.
 - Create a discrete mathematical model of the system.
 - Numerically solve the model.
 - Process the resulting data.

Experimental design layout:

Design Expert 13:



- Design of Experiment (DOE) is a systematic method to determine the relationship between factors affecting a process and the output of that process.
- In other words, it is used to find cause-and-effect relationships.
- This information is needed to manage process inputs in order to optimize the output.

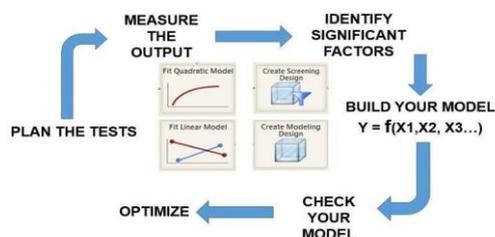
The design of experiments (DOE, DOX, or experimental design) is the planning of any activity that tries to describe and explain the variance of information under postulated conditions. The word is commonly linked with experiments in which the design includes factors that directly impact the variation, but it may also apply to the design of quasi-experiments in which natural conditions influencing the variation are chosen for observation.

In its most basic form, an experiment attempts to predict the outcome by changing the preconditions, which are represented by one or more independent variables, often known as "input variables" or "predictor variables." It is commonly assumed that a change in one or more independent variables would result in a change in one or more dependent variables, often known as "output variables" or "response variables." The experimental design may also identify control variables that must be kept constant in order to avoid outside forces from influencing the results. Not only does experimental design entail the selection of appropriate independent, dependent, and control variables, but it also entails organising the delivery of the experiment under statistically optimal circumstances given the restrictions of available resources.

There are several methods for identifying the set of design points (unique combinations of the independent variable values) to be utilised in the experiment.

The establishing of validity, reliability, and reliability are the primary considerations in experimental design. These issues, for example, can be addressed in part by carefully selecting the independent variable, decreasing the possibility of measurement error, and ensuring that the method documentation is appropriately clear. Concerns related to this include establishing adequate levels of statistical power and sensitivity.

Fig 4.3 Flow chart of Modelling and analysis in Design expert 13



- Hence, Design of Experiments (DoE) is ideally suited for such multivariable analyses: by planning one's experiments as per the principles of DoE, one can test and optimize several variables simultaneously, thus accelerating the process of discovery and optimization while saving time and precious laboratory resources.

FACTORIAL DESIGNS: (Two Factor Factorial Designs)

A two-factor factorial design is an experimental design that collects data for all potential values of the two factors of interest.

The design is a balanced two-factor factorial design if equal sample sizes are used for each of the possible factor combinations. A balanced (a b) factorial design is one that has a level of factor A, b level of factor B, and n independent replications at each of the (a b) treatment combinations. $N = abn$ is the design size.

The average change in reaction associated with a change in the level of a factor is defined as its impact. This is commonly referred to as a major impact.

If the average change in reaction across all levels of one component is not the same at all levels of the other factor, we say the factors interact.

For our experimental investigation, we employ a 24 Factorial Design.

Analysis of the Experiment:

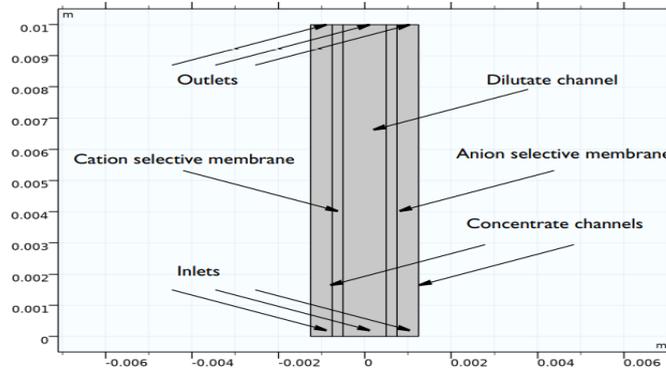
System Model:

Electro deionization is a separation process for electrolytes based on the use of electric fields and ion selective membranes. Some common applications of the electro deionization process are:

- Desalination of process streams, effluents, and drinking water
- PH regulation in order to remove acids from, for example, fruit juices and wines
- Metal electro winning of precious metals

The model geometry is based on the repetitive unit, excluding the inlet and outlet flow regions.

Fig 5.1: Model geometry



The cell contains two ion selective membrane domains: the left is mainly permeable to cations, and the right to anions. The middle domain is a free-flowing electrolyte domain, where salt is to be removed. This domain is named the dilute domain. The rightmost and leftmost domains are free flowing electrolyte domains, where the ion concentration increases during cell operation. These domains are called the concentrate domains. In the free electrolyte domains, the flow enters at the bottom inlets and exits at the top outlets at an average flow rate is $5 \text{ mm}\cdot\text{s}^{-1}$. An analytical expression (Poiseuille flow) for the fluid velocity is used in this model.

Choice of Current Distribution Interface:

In this model we make use of the Nernst-Planck equations for ion flux and charge transport by which the following equation describes the molar flux of species i (which is either Cl or Na in this model), N_i , due to diffusion, migration and convection: Dilute channel Anion selective membrane Concentrate channels Inlets Cation selective membrane Outlets

$$\mathbf{N}_i = -D_i \nabla c_i - z_i u_{\text{mob},i} F c_i \nabla \phi_l + c_i \mathbf{u}$$

The first term is the diffusion flux, D_i is the diffusion coefficient (SI unit: m^2/s). The migration term consists of the species charge number z_i , the species mobility $u_{\text{mob},i}$ (SI unit: $\text{s}\cdot\text{mol}/\text{kg}$) and the electrolyte potential (ϕ_l). In the convection term, \mathbf{u} denotes the fluid velocity vector (SI unit: m/s).

The electrolyte current density is calculated using Faraday's law by summing up the contributions from the molar fluxes, multiplied by the species charges, with the observation that the convective term vanishes due to the electro neutrality condition (see the theory for the Tertiary Current Distribution, Nernst-Planck interface):

$$\mathbf{i}_l = F \sum_{i=1}^n z_i (-D_i \nabla c_i - z_i u_{\text{m},i} F c_i \nabla \phi_l)$$

Factor	Name	Units	Type	Sub Type	Minimum	Maximum	Coded Low	Coded High	Mean	Std. Dev.
A	Voltage	V	Numeric	Continuous	7.00	14.00	-1 ↔ 7.00	+1 ↔ 14.00	10.50	3.61
B	Conc of feed	Mol/m ³	Numeric	Continuous	0.0100	0.0500	-1 ↔ 0.01	+1 ↔ 0.05	0.0300	0.0207
C	Temp	K	Numeric	Continuous	288.00	303.00	-1 ↔ 288.00	+1 ↔ 303.00	295.50	7.75
D	Flow rate	L/min	Numeric	Continuous	0.5000	1.0000	-1 ↔ 0.50	+1 ↔ 1.00	0.7500	0.2582

The conservation of current is then used to calculate the electrolyte potential.

$$\nabla \cdot \mathbf{i}_l = F \sum_{i=1}^n z_i R_i$$

Where the R_i terms are the reaction sources due the porous electrode reactions. This model uses Tertiary Current Distribution, Nernst-Planck interface when solving for the electrolyte potential in the free electrolyte and ion-selective membrane domains. In the free electrolyte domains, the only ions present are assumed to be Na^+ and Cl^- . The ion selective membranes also contain additional ions fixed in a matrix. Therefore, the fixed space charge is added while calculating the sum of charges in the electro neutrality condition: \

$$\rho_{\text{fix}} + F \sum_{i=1}^n z_i c_i = 0$$

The fixed space charge is prescribed in terms of the membrane charge concentration which is varied using an auxiliary sweep in this model.

Parameters Used: Global Defined:

Parameters used for modelling pre-exist in COMSOL

Application library under the subtheme

Electro Chemistry. The following are the parameters:

Factors Affecting the System:

There are various elements that affect the system in an electro deionization cell; however, we have focused on four factors that we believe, based on literature research, have a significant impact on the system: voltage, feed concentration, temperature, and flow rate.

FACTOR	NAME	UNITS	TYPE
A	Voltage	V	Numeric
B	Conc. of feed	Mol/m ³	Numeric
C	Temperature	K	Numeric
D	Flow rate	L/min	Numeric

Factors affecting system

We now feed these factors into Design Expert software, which employs 24 Factorial Design, and assign a range to each component, including a minimum and maximum value derived from the literature research.

Range of factors

The design Expert 13 Software then does analysis using Statistical method and then provides us with a set number of experiments to be conducted (which is less than conducting total number of experiment) to obtain the exact number of results. The table given below is the set of 16 experiments provided by design Expert software for the input given.

Name	Expression	Value	Description
W_m	0.25[mm]	2.5E-4 m	Membrane width
W_ch	1[mm]	0.001 m	Channel width
Voltage	1.5[V]	1.5 V	Total potential drop over unit cell
Flow Rate	0.5[Ltr/Min]	0.5 Ltr/Min	Channel Flow Rate
Temperature	298.15[K]	298.15 K	Temperature
L	0.01[m]	0.01 m	Cell length
D _{Na}	2.5e-9[m ² /s]	2.5E-9 m ² /s	Diffusion coefficient, Na
D _{Cl}	2e-9[m ² /s]	2E-9 m ² /s	Diffusion coefficient, Cl
Conc	0.05[mol/m ³]	0.05 mol/m ³	Inlet concentration, Cl

We then simulate all the 16 experiments with differing factor values generated by the Design expert 13 in COMSOL Multiphysics Software. COMSOL model then simulate all 16 experiments with given inputs and provide us with the result, which is the electrode potential value of the system at the end of the simulation. By using these electrode potential values obtained from COMSOL, we will then be able to calculate the pH values of the system at the end of simulation using Nernst plank Equations.

Std	Run	Factor A Voltage (V)	Factor B Conc of Feed (Mol/M ³)	Factor C Temperature (K)	Factor D Flow Rate (L/min)
6	1	14	0.01	303	0.5
5	2	7	0.01	303	0.5
8	3	14	0.05	303	0.5
2	4	14	0.01	288	0.5
9	5	7	0.01	288	1
10	6	14	0.01	288	1
3	7	7	0.05	288	0.5
11	8	7	0.05	288	1
1	9	7	0.01	288	0.5
15	10	7	0.05	303	1
12	11	14	0.05	288	1
16	12	14	0.05	303	1
7	13	7	0.05	303	0.5
13	14	7	0.01	303	1
4	15	14	0.05	288	0.5
14	16	14	0.01	303	1

$$E_{Cell} = -0.05916 * V * pH$$

RESULTS:

Std	Run	Factor A (voltage)	Factor B Conc. Of feed (mol/m ³)	Factor C	Factor D Flow rate (L/min)	E cell	pH
6	1	14	0.01	303	0.5	6.128	7.4
5	2	7	0.01	303	0.5	2.7331	6.6
8	3	14	0.05	303	0.5	6.1289	7.4
2	4	14	0.01	288	0.5	5.632032	6.8
9	5	7	0.01	288	1	2.816061	6.8
10	6	14	0.01	288	1	5.7979	7
3	7	7	0.05	288	0.5	2.40189	5.8
11	8	7	0.05	288	1	2.153424	5.2
1	9	7	0.01	288	0.5	2.857428	6.9
15	10	7	0.05	303	1	2.5675	6.2
12	11	14	0.05	288	1	6.12897	7.4
16	12	14	0.05	303	1	6.46027	7.8
7	13	7	0.05	303	0.5	2.73319	6.6
13	14	7	0.01	303	1	2.44330	5.9
4	15	14	0.05	288	0.5	5.963328	7.2
14	16	14	0.01	303	1	5.79768	7

Experimental Results Using Design Expert

Final Result with pH

Half normal plot:

In a Half normal plot by default the red “error line” will be placed such that it represents the smallest 50% of the effects. It is intended to be a visual guide to assist with selecting effects. It can be manually moved to best represent the group of smallest (on the left) effects. The red line will not move automatically until more than 50% of the effects have been selected for the model. Starting at the largest effects (on the right), select terms that fall below or to the right

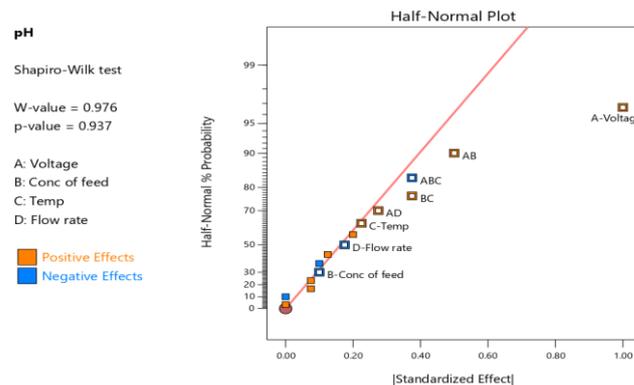
of the red line. The goal is to separate the effects into the large, likely to be repeatable effects and the small, likely to be noise effects. If no effects appear to separate from the red error line, then it is possible that no effects are significant.

Half-Normal Plot

Ctrl + right-click will show the x-axis effect values that are currently displayed.

Shift + right-click will show the y-axis percentiles that are currently displayed.

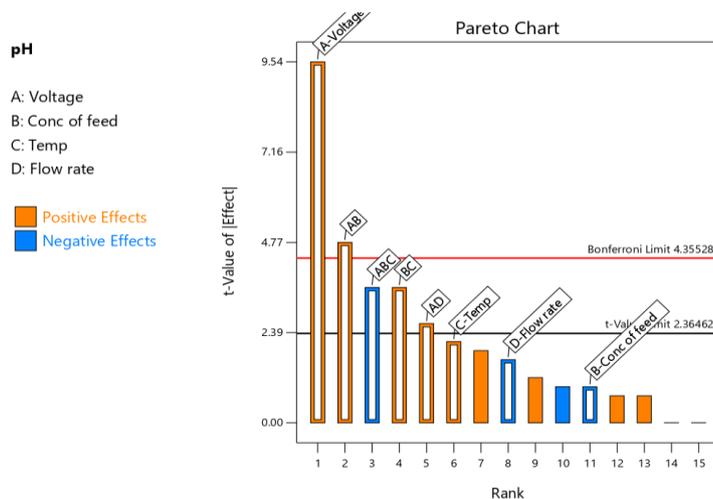
From the above obtained Half-normal plot, we can interpret that the variables which deviate more from the red line are more likely to affect the system significantly. Whereas the factors which do not deviate are less likely to affect the system. On that basis we can predict that voltage has more effect on the system, followed by the combination AB (voltage & conc. of feed). And the factor which least effect the system seems to be concentration on feed.



Factor contributions

Pareto chart:

A **Pareto chart** is a type of chart that contains both bars and a line graph, where individual values are represented in descending order by bars, and the cumulative total is represented by the line. The chart is named for the Pareto principle, which, in turn, derives its name from Vilfredo Pareto, a noted Italian economist. The left vertical axis is the frequency of occurrence, but it can alternatively represent cost or another important unit of measure. The right vertical axis is the cumulative percentage of the total number of occurrences, total cost, or total of the particular unit of measure. Because the values are in decreasing order, the cumulative function is a concave function. The Pareto chart provided below clearly depicts the effect of combination of different factors on the system. And it's clear that voltage has the most effect out of all the proposed combination of factors. Where positive effects refer to factors which enhances the ionic separation in the ionization cell, Whereas Negative effects refer to factors which decreases the ionic separation.



ANOVA for Selected Factorial Model:

Analysis of variance (ANOVA) is a collection of statistical models and their associated estimation procedures (such as the "variation" among and between groups) used to analyse the differences among means. ANOVA was developed by the statistician Ronald Fisher. ANOVA is based on the law of total variance, where the observed variance in a particular variable is partitioned into components attributable to different sources of variation. In its simplest form, ANOVA provides a statistical test of whether two or more population means are equal, and therefore generalizes the t-test beyond two means. In other words, the ANOVA is used to test the difference between t.

ANOVA for selected factorial model

Response 1: pH

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	6.79	8	0.8491	19.33	0.0004	significant
A-Voltage	4	1	4	91.06	< 0.0001	
B-Conc of feed	0.04	1	0.04	0.9106	0.3717	
C-Temp	0.2025	1	0.2025	4.61	0.0689	
D-Flow rate	0.1225	1	0.1225	2.79	0.1389	
AB	1	1	1	22.76	0.002	
AD	0.3025	1	0.3025	6.89	0.0342	
BC	0.5625	1	0.5625	12.8	0.009	
ABC	0.5625	1	0.5625	12.8	0.009	
Residual	0.3075	7	0.0439			
Cor Total	7.1	15				

Std. Dev.	0.2096	R ²	0.9567
Mean	6.75	Adjusted R ²	0.9072
C.V. %	3.11	Predicted R ²	0.7737
		Adeq Precision	15.1088

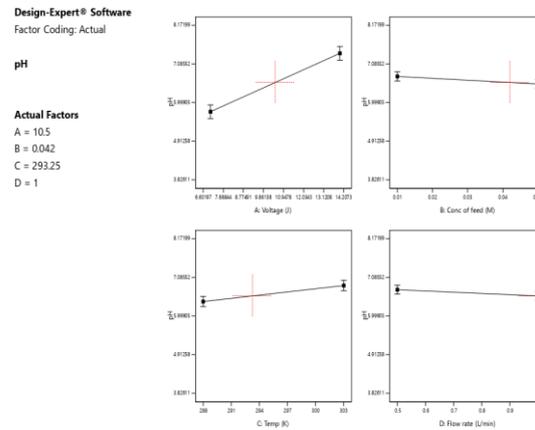
Table 6.3 Fit statistics

Factor coding is coded.

Sum of squares is Type III - Partial

Now, by performing an ANOVA analysis on the results obtained from the design of expert software, we obtain the above shown table. It shows both F-values which is used to check if the variance between the two populations is significantly different or not. In our case it tells whether, the values obtain from different tests have a significant difference.

- The Model F-value of 19.33 implies the model is significant. There is only a 0.04% chance that an F-value this large could occur due to noise.
- P-values less than 0.0500 indicate model terms are significant. In this case A, AB, AD, BC, ABC are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve your model.
- The Predicted R² of 0.7737 is in reasonable agreement with the Adjusted R² of 0.9072; i.e., the difference is less than 0.2.
- Adeq Precision measures the signal to noise ratio. A ratio greater than 4 is desirable. Your ratio of 15.109 indicates an adequate signal. This model can be used to navigate the design space.
- A standard deviation (or σ) is a measure of how dispersed the data is in relation to the mean. Low standard deviation means data are clustered around the mean, and high standard deviation indicates data are more spread out.
- The standard deviation of the data obtained from the test is observed to be 0.2096, with an observed mean of 6.75 and coefficient of variance of 3.11.



Final Model Equation in Terms of Coded Factors:

A final equation is obtained which can accurately interpret the cause and effect between different factors affecting the system and the final PH of the brackish water. This equation can be very useful for fast and accurate calculations.

$$\text{pH} = 6.75 + (0.5000)*A - (0.0500)*B + (0.1125)*C - (0.0875)*D + (0.2500)*AB + (0.1375)*AD + (0.1875)*BC - (0.1875)*ABC$$

Where, A = Voltage B = Conc. Of feed

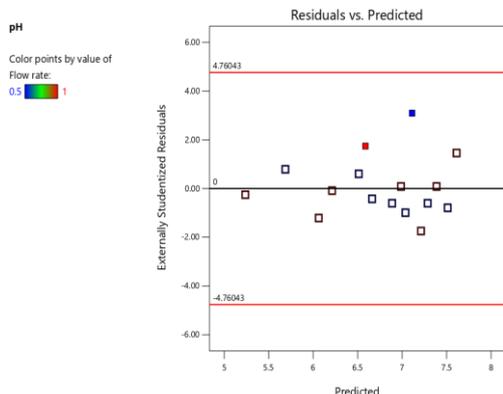
C = Temperature. D = Flow Rate

Hence using the above equation, we can find the pH value of the system for different values of A, B, C, D without simulating or conducting the experiment.

One Factor Effects:

The One Factor Effects graph shows the linear effect of changing the level of a single factor. It is constructed by predicting the responses for the low (-1) and high (+1) levels of a factor. For factorial analysis, the I-beams around the predictions are the result of least significant difference (LSD) calculations. Design expert also provides us with various one factor plots between the factors effecting the system like, pH vs Voltage, pH vs Conc. of feed, pH vs Temp, pH vs flow rate. This one factor plots can help us understand the relation between the factors of the system furthermore.

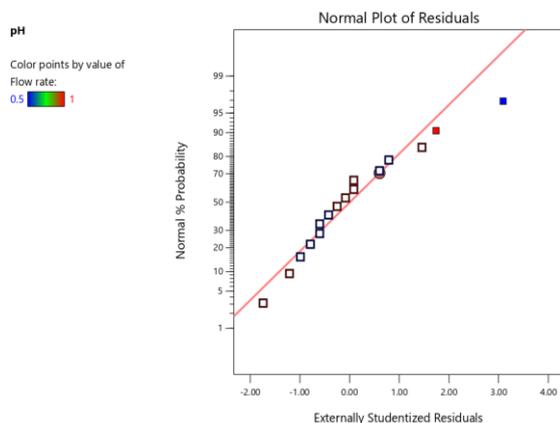
Plots:



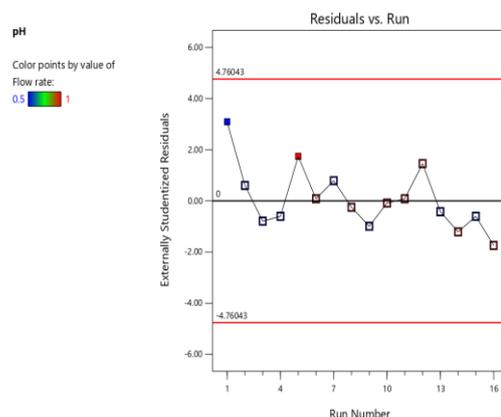
This is a plot of the residuals versus the ascending predicted response values. It is a visual check for the assumption of constant variance. The plot should be a random scatter having a consistent top to bottom range of residuals across the predictions on the X1 axis.

Residual VS Run

It is a plot of the residuals versus the experimental run order. It checks for lurking variables that may have influenced the response during the experiment. The plot should show a random scatter.

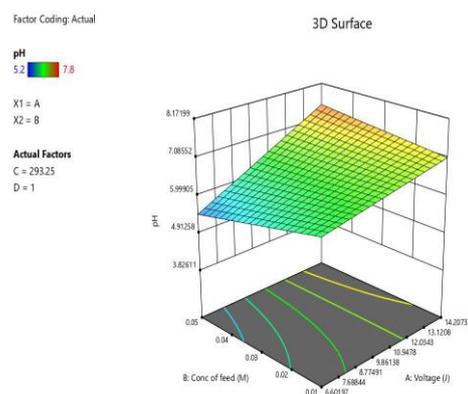


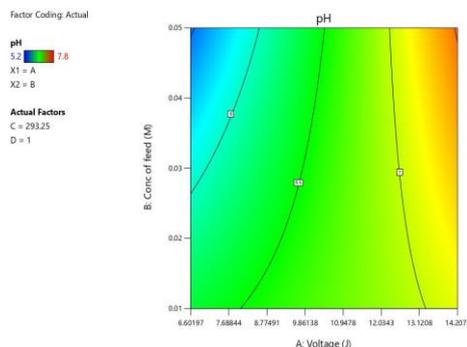
The normal probability plot indicates whether the residuals follow a normal distribution, in which case the points will follow a straight line. Expect some moderate scatter even with normal data. Look only for definite patterns like an “S-shaped” curve, which indicates that a transformation of the response may provide a better analysis.



Contour Plot

A contour plot is a graphical technique for representing a 3-dimensional surface by plotting constant z slices, called contours, on a 2-dimensional format. That is, given a value for z, lines are drawn for connecting the (x, y) coordinates where that z value occurs. The typical application of the DOE contour plot is in determining settings that will maximize (or minimize) the response variable. It can also be helpful in determining settings that result in the response variable hitting a pre-determined target value. The DOE contour plot plays a useful role in determining the settings for the next iteration of the experiment. That is, the initial experiment is typically a fractional factorial design with a fairly large number of factors. After the most important factors are determined, the DOE contour plot can be used to help define settings for a full factorial or response surface design based on a smaller number of factors.





Surface plot

Surface plots are diagrams of three-dimensional data. Rather than showing the individual data points, surface plots show a functional relationship between a designated dependent variable (Y), and two independent variables (X and Z). The plot is a companion plot to the contour plot. It is also used when you have a stored model and want to plot how the fitted response relates to two continuous variables. A surface plot displays the three-dimensional relationship in two dimensions, with the variables on the x- and y-axes, and the response variable (z) represented by a smooth surface.

Report:

Run Order	Actual Value	Predicted Value	Residual	Leverage	Internally Studentized Residuals	Externally Studentized Residuals	Cook's Distance	Influence on Fitted Value DFFITS	Standard Order
1	7.40	7.11	0.2875	0.563	2.074	3.092	0.614	3.506 ^(*)	6
2	6.60	6.51	0.0875	0.563	0.631	0.602	0.057	0.682	5
3	7.40	7.51	-0.1125	0.563	-0.812	-0.789	0.094	-0.895	8
4	6.80	6.89	-0.0875	0.563	-0.631	-0.602	0.057	-0.682	2
5	6.80	6.59	0.2125	0.563	1.533	1.741	0.336	1.974	9
6	7.00	6.99	0.0125	0.562	0.090	0.084	0.001	0.095	10
7	5.80	5.69	0.1125	0.563	0.812	0.789	0.094	0.895	3
8	5.20	5.24	-0.0375	0.563	-0.271	-0.252	0.010	-0.285	11
9	6.90	7.04	-0.1375	0.563	-0.992	-0.990	0.141	-1.123	1
10	6.20	6.21	-0.0125	0.563	-0.090	-0.084	0.001	-0.095	15
11	7.40	7.39	0.0125	0.563	0.090	0.084	0.001	0.095	12
12	7.80	7.61	0.1875	0.563	1.353	1.457	0.261	1.652	16
13	6.60	6.66	-0.0625	0.563	-0.451	-0.424	0.029	-0.480	7
14	5.90	6.06	-0.1625	0.563	-1.172	-1.211	0.196	-1.373	13
15	7.20	7.29	-0.0875	0.563	-0.631	-0.602	0.057	-0.682	4
16	7.00	7.21	-0.2125	0.563	-1.533	-1.741	0.336	-1.974	14

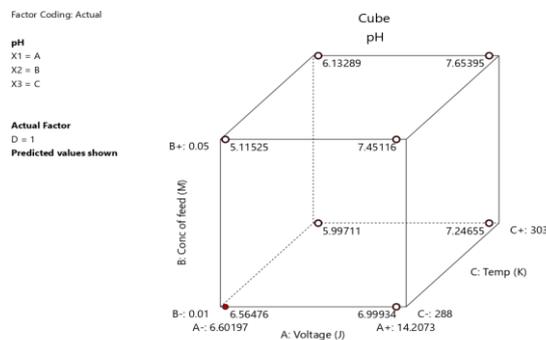
^(*) Exceeds limits.

Table 6.5 Final report table

Cube plot:

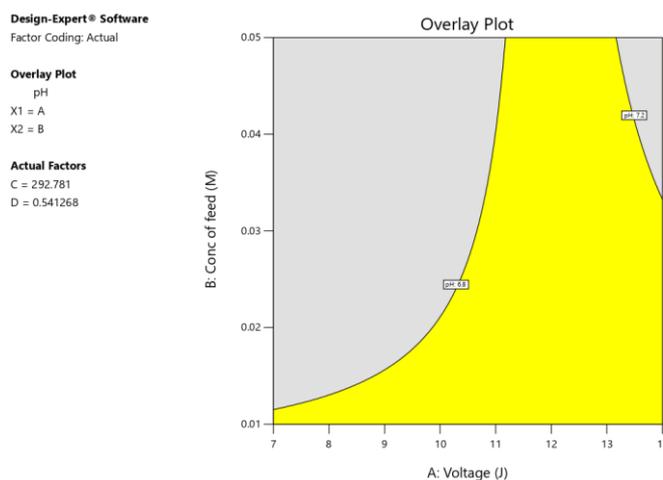
Cube plots are created for 2-level factorial designs or Plackett-Burman designs to demonstrate the correlations between two to eight components with or without a response measure. Viewing the variables without the reaction allows you to see how a design appears. When you have a two-level factorial design or a mixed design and wish to display the relationship between the variables and a response, use the Cube Plot. Cube plots can show the following:

- The combinations of factor settings and either the data mean or fitted mean for each combination.
- The combinations of factor settings without any response means.



Design Space:

The multidimensional combination and interplay of input factors (such as material qualities) and process parameters that has been shown to offer quality assurance



From the above figure, using Design Expert 13, it overlays a plot for optimal region for Electro Deionization (Yellow Region)

Point Prediction

Two-sided Confidence = 95% Population = 99%

Looking at the findings from the Design Expert 13 table above, we can plainly see that the anticipated median and mean pH values for the system are both 7.07582 and 7.07582, respectively. The table below provides the optimised condition where the supplied input parameters produce cleaner deionized solution, where pH is equal to or almost equal to 7.

Analysis	Predicted Mean	Predicted Median	Observed	Std Dev	SE Mean	95% CI low for Mean	95% CI high for Mean	95% TI low for 99% Pop	95% TI high for 99% Pop
pH	6.75	6.75		0.209591	0.0523979	6.6261	6.8739	5.6805	7.8195

Table 6.6 Point Prediction

Optimized Confirmed Location

Voltage (V)	Conc. of feed (Mol/m ³)	Temperature (K)	Flow rate(L/Min)
12.5484	0.05	292.781	0.541268

Table 6.7 Optimized Confirmed Location

Alpha	0.05
Tolerance	0.99
Interval	Two sided

Table 6.8 Parameters

Other relevant factors at this position, such as alpha and tolerance, are:

Most ionic separation occurs at this confirmed position, and the pH value at this point is 7.00, whereas the projected value is 7.07. This results in 99.2 percent accuracy.

CONCLUSIONS:

- We successfully discovered and modelled variables impacting Electro Deionisation (EDI) using COMSOL. Design Expert 13 simulation software was used to optimise crucial parameters using the Design of Experiment (D.O.E) concept.

- We decreased the number of experiment runs from 256 to 16, implying that we only had to conduct/simulate 6.25 percent of all experiments.
- We achieved a pH accuracy rate of 99.2 percent using Design Expert simulation software
- We have verified the model's accuracy and validated our Design of Experiment (D.O.E).

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