

Article

Analysis of the Electrochemical and Conductive Properties of Mixed Metal Complexes using Electrical Measurement Techniques

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Abstract: This study systematically investigates the physicochemical and conductive properties of aqueous and immobilized potassium hydroxide (KOH) electrolytes, providing critical data for the optimization of high-performance alkaline electrochemical devices, the ionic conductivity of KOH solutions at varying concentrations (35, 45, and 55 wt.%) was meticulously characterized across a broad operational temperature range (25–220°C), to maintain the liquid phase at temperatures exceeding the normal boiling point, all measurements were conducted under high pressure (up to 30 bar), a necessity dictated by the material's phase diagram, the experimental setup utilized custom-designed cells for both bulk aqueous solutions and electrolytes immobilized within porous pellets, with impedance spectroscopy employed as the primary analytical technique.

The results reveal a complex, non-linear relationship between conductivity, temperature, and concentration. For aqueous solutions, conductivity initially increases with temperature, driven by enhanced ionic mobility and reduced solvent viscosity, before reaching a distinct peak, the 35% KOH solution exhibited a peak conductivity of 2.8 S·cm⁻¹ at 206°C. Notably, a critical performance crossover was identified: while the 35% solution displayed superior conductivity at ambient temperature (25°C), the 45% solution demonstrated significantly higher conductivity at elevated temperatures (≥ 200°C), in contrast, the effective conductivity of immobilized electrolytes was, as expected, attenuated by the tortuous pathways and inherent porosity of the pellet matrix, the highest immobilized conductivity of 0.84 S·cm⁻¹ was achieved with the 45% solution at 200°C. Crucially, porosimetry analysis revealed that the pellet pores were only partially saturated (74–83% filled), indicating that these measured values represent a conservative baseline with substantial potential for enhancement through improved wetting protocols, the temperature-dependent behavior for all systems was accurately modeled using cubic regression equations, which showed excellent fidelity with both experimental data and established literature values, within a total estimated measurement uncertainty of ±10%, ultimately, this work provides a fundamental framework and predictive models for selecting the optimal electrolyte concentration based on specific operating temperatures, it quantifies the performance trade-offs between aqueous and immobilized systems and identifies key material engineering strategies such as reducing pellet thickness and ensuring complete pore saturation for minimizing ohmic losses and advancing the next generation of alkaline electrolyzers and fuel cells.

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1. Introduction

Alkaline water electrolysis (AWE) represents a mature and scalable technology for green hydrogen production. However, its energy conversion efficiency remains constrained by several factors, chief among them being the **ohmic resistance** of the

electrolyte [1]. Concentrated aqueous solutions of potassium hydroxide (KOH), typically in the range of 25–35 wt%, are the electrolytes of choice for AWE systems, primarily due to their superior ionic conductivity (σ) and chemical stability under highly caustic conditions, the performance of the electrochemical cell is critically dependent on this transport property, as the internal resistance directly contributes to the **ohmic overpotential (iR drop)**, thereby dictating the overall energy consumption of the system [2].

The ionic conductivity of KOH solutions is a complex function of both concentration (c) and temperature (T), under conventional operating conditions ($T < 100$ °C), conductivity maxima are well-documented to occur at concentrations around 30 wt% [3]. However, the current trend towards operating electrolyzers at elevated temperatures (>100 °C) and pressures to enhance reaction kinetics and minimize electrode overpotentials reveals a significant **knowledge gap** in the understanding of electrolyte behavior. Experimental data in this high-temperature regime are scarce, largely due to methodological challenges, these include increased vapor pressure, accelerated corrosion of cell components, and, most notably, the confounding effect of **gas bubble evolution** on the electrodes, the formation and detachment of H_2 and O_2 bubbles introduce transient, non-uniform insulating layers, which increase the apparent resistance by occluding active electrode surface area and disrupting ionic pathways [4].

Overcoming these challenges necessitates the application of advanced characterization techniques, the **Van der Pauw method**, a four-probe measurement technique, is ideally suited for these conditions, it circumvents the limitations imposed by sample geometry and provides accurate, intrinsic measurements of resistivity (ρ) even under high-temperature and high-pressure environments [5]. Complementing this, **Electrochemical Impedance Spectroscopy (EIS)** offers a powerful means to deconvolve the various contributions to the total system impedance, by analyzing the frequency response, EIS allows for the separation of the bulk electrolyte resistance (R_s) from interfacial phenomena, such as the charge-transfer resistance (R_{ct}) at the electrodes and the double-layer capacitance (C_{dl}), providing deeper insights into the electrochemical processes at the electrode-electrolyte interface [6].

Furthermore, within an AWE cell, the electrolyte does not exist solely as a bulk phase but is also confined within a **porous diaphragm** that physically separates the gaseous products, this confinement imposes **tortuous paths** for ion transport, resulting in a lower **effective conductivity (σ_{eff})** compared to the bulk value, this effective property is intrinsically linked to the microstructural characteristics of the diaphragm, including its **porosity (ϵ)** and **tortuosity (τ)**, parameters that must be accounted for in any rigorous system analysis [7].

Therefore, this study is designed to conduct a systematic investigation into the electrical properties of concentrated KOH solutions across a temperature range extending beyond 100 °C, by integrating the Van der Pauw method and EIS, we aim to delineate the optimal concentration and temperature conditions that maximize ionic conductivity while simultaneously characterizing the impact of confinement within porous structures. A fundamental understanding of these structure-property relationships is paramount for the rational design and optimization of the next generation of high-efficiency alkaline electrolyzers.

Literature review

The pursuit of a hydrogen economy is not merely an engineering challenge but is, at its core, a complex chemical problem centered on **electrocatalysis**, water electrolysis, particularly in an alkaline medium, is a process entirely dependent on overcoming the kinetic barriers of the hydrogen evolution reaction (HER) and the oxygen evolution reaction (OER), these reactions necessitate efficient catalysts capable of lowering the activation energy, thereby reducing the **overpotential** required to drive the reaction, it is here that mixed metal complexes emerge as promising materials, not only for their effectiveness but also for the potential to tailor their electronic properties at the atomic level for optimal performance [8].

In an alkaline solution such as KOH, water splitting occurs via two half-reactions at distinct electrodes. At the cathode, the hydrogen evolution reaction (HER) proceeds as $2\text{H}_2\text{O} + 2\text{e}^- \rightarrow \text{H}_2(\text{g}) + 2\text{OH}^-$, despite its apparent simplicity, this reaction involves multiple mechanistic steps, and its efficiency depends on the ability of the catalyst surface to facilitate these steps. At the anode, the oxygen evolution reaction (OER) occurs via $4\text{OH}^- \rightarrow \text{O}_2(\text{g}) + 2\text{H}_2\text{O} + 4\text{e}^-$, the OER is the true bottleneck of water electrolysis, it is a kinetically sluggish reaction involving a four-electron transfer and the formation of an O=O bond, a process that is energetically demanding and proceeds through various reactive intermediates (e.g., OH, O, OOH) on the catalyst surface, the design of advanced catalysts aims to optimally stabilize these intermediates to lower the overpotential [9].

The role of the potassium hydroxide (KOH) electrolyte is not limited to providing ionic conductivity. Chemically, it is the source of hydroxide ions (OH^-), which are the primary reactant in the OER and a product of the HER. Consequently, the concentration and activity of OH^- ions at the electrode surface directly influence the reaction kinetics, the specific conductivity of KOH solutions has been extensively documented as a function of concentration and temperature. Studies conducted at elevated temperatures and pressures show that the mobility of ions (K^+ and OH^-) increases with heat, which reduces the solution's resistance and improves the cell's overall efficiency. Other physicochemical properties of the KOH- H_2O system have also been studied to fully understand its behavior under various operating conditions [10].

Mixed metal complexes, such as nickel-iron, cobalt-iron, or nickel-cobalt oxides and hydroxides, represent a significant advancement in electrocatalysts, especially for the OER, the key to their superior performance lies in the **synergistic effect** between the different metallic centers, this synergy manifests in several ways [11]. Firstly, the presence of a second metal, such as iron in a nickel hydroxide matrix, modifies the electronic structure of the primary metal's d-orbitals, thereby optimizing the adsorption energy of OER intermediates like OH. Secondly, it can provide multiple active sites, where one metal center might specialize in one reaction step while the other facilitates a subsequent step, such as O-O bond formation. Finally, the combination of metals can enhance the intrinsic electrical conductivity of the catalyst material itself, facilitating electron transport from the active site to the electrode, understanding and precisely characterizing these effects at the atomic level is the primary goal of this study, moving from trial-and-error to the rational design of catalysts, in line with modern trends toward advanced alkaline membranes and hybrid systems [12].

To analyze these complex materials, an integrated suite of measurement techniques is essential, before testing a material as a catalyst, its intrinsic electrical conductivity must be measured, the **Van der Pauw method** is ideal for this purpose, as it allows for the precise measurement of resistivity in irregularly shaped samples, such as a pressed powder or a thin film, a technique that has been successfully adapted for various systems including electrolytes. Furthermore, the catalytic performance is evaluated using electrochemical techniques. **Linear Sweep Voltammetry (LSV)** is used to determine the overpotential and the **Tafel slope**, which provides insight into the reaction mechanism, in addition, **Electrochemical Impedance Spectroscopy (EIS)** is employed to decouple the solution resistance from the charge transfer resistance at the electrode surface (Table 1).

Table 1. Proposed Methodology

<i>Phase</i>	<i>Objective</i>	<i>Methodology / Technique</i>	<i>Key Performance Indicators (KPIs) / Analysis</i>	<i>Supporting References</i>
1. Material Synthesis & Fabrication	To prepare a series of mixed metal complexes (e.g., Ni-Fe, Ni-Co) with varying compositions.	Co-precipitation, hydrothermal, or sol-gel methods.	Control of metal molar ratios, reaction conditions (temperature, time, pH).	(as examples of advanced material synthesis)

2. Structural & Morphological Characterization	To confirm the crystal structure, surface chemical composition, and morphology of the prepared materials.	- X-ray Diffraction (XRD)- Scanning Electron Microscopy (SEM)- Energy-Dispersive X-ray Spectroscopy (EDX)- X-ray Photoelectron Spectroscopy (XPS)	- Crystal phases and crystallite size.- Surface morphology and particle distribution.- Elemental composition of the material.- Oxidation states of metals on the surface.
3, intrinsic Electrical Property Analysis	To measure the electrical conductivity of the complexes (as pressed pellets) to determine their contribution to electron transport.	Van der Pauw Method	- Resistivity (ρ) [$\Omega \cdot \text{cm}$]- Electrical Conductivity (σ) [S/cm]
4. Electrochemical Performance Evaluation	To measure the catalytic activity of the materials towards OER and HER in a KOH solution.	- Three-electrode electrochemical cell.- Linear Sweep Voltammetry (LSV).- Electrochemical Impedance Spectroscopy (EIS).- Chronoamperometry.	- Overpotential (η) at a specific current density (e.g., 10 mA/cm ²).- Tafel slope [mV/dec].- Charge transfer resistance (R_{ct}) and solution resistance (R_s).- Long-term stability of the catalyst.
5. Electrolyte System Analysis	To characterize and confirm the properties of the KOH electrolyte used in experiments to ensure result reproducibility.	- Conductivity measurement using a conductivity meter.- Use of published data and software (e.g., FactSage).	- Specific conductivity of the solution at the experimental temperature.- Comparison of results with reference data to ensure system accuracy.

2. Materials and Methods

To conduct a precise and comprehensive analysis of the ionic conductivity of potassium hydroxide (KOH) solutions under severe operating conditions of high pressure and elevated temperature, which simulate the actual environment inside industrial alkaline electrolyzers, a specialized and sophisticated experimental system was designed. At the heart of this system is a 600 mL Parr 4760 pressure vessel, the selection of this vessel was not arbitrary but was based on its exceptional capacity to withstand demanding operational pressures and temperatures, the vessel was constructed from Inconel 600, a nickel-chromium superalloy renowned for its extraordinary mechanical strength at high temperatures and its excellent chemical resistance, particularly against stress corrosion cracking in hot, concentrated alkaline solutions a common failure mechanism for lesser materials in such environments, to ensure maximum chemical purity for the experiment, the vessel was fully lined with a thick polytetrafluoroethylene (PTFE) liner, designated as

TL1 in the schematic, this liner acts as a completely inert barrier, preventing any interaction between the highly caustic KOH solution and the metallic vessel walls, this guarantees that any observed changes in conductivity are solely attributable to the intrinsic properties of the electrolyte itself, not to contamination from leached metal ions from the vessel wall.

The system was equipped with a fully integrated suite of fixtures for precise control and monitoring of the reaction environment, the system was connected to a pressurized nitrogen source, with its flow meticulously controlled via a needle valve, the purpose of the applied pressure was not only to simulate operating conditions but, more critically, to raise the boiling point of the aqueous solution, thereby permitting stable measurements at temperatures exceeding 100°C without violent boiling. For accurate monitoring of the internal conditions, the vessel was fitted with a J-type thermocouple (designated as T1) housed within a thermowell that plunged directly into the solution, this arrangement ensures an immediate and true measurement of the electrolyte's temperature itself, rather than the temperature of the external vessel wall, eliminating any error from thermal gradients, in parallel, two pressure gauges were installed to provide both an instantaneous analog display for quick monitoring and continuous digital recording of the data, allowing for the tracking of pressure stability throughout the experiment.

The core of this experimental design lies in the use of two simultaneous measurement cells, the first cell, designated as APMC, is dedicated to measuring the conductivity of the bulk aqueous KOH solution, this cell was connected to the protective cover of the vessel using a PTFE shrink tube, ensuring it remained fixed in position and electrically isolated, this cell provides fundamental baseline data on the intrinsic ionic conductivity of the electrolyte, the second cell, positioned beneath the first, was designed to measure the conductivity of an immobilized electrolyte, simulating the state of the electrolyte within the narrow pores of diaphragms used in electrolyzers, this dual configuration allows for a direct comparison between the conductivity in its free state and the effective conductivity when it is constrained within tortuous pathways a critical distinction for understanding the ohmic drop in a real cell.

To address one of the greatest challenges in long-duration measurements at high temperatures—water evaporation from the sample an intelligent strategy was implemented. Evaporation, even in a closed system, leads to a gradual increase in the KOH concentration, resulting in inaccurate and drifting measurement results, to overcome this issue, a "sacrificial" bath of KOH solution, with a concentration slightly lower (by 2%) than the test sample, was placed at the bottom of the autoclave, this solution serves to saturate the internal vessel atmosphere with water vapor, creating a vapor pressure close to that of the test sample. Consequently, the gradient in the chemical potential of water between the sample and its surrounding atmosphere is minimized, effectively halting net evaporation from the measurement cell, this meticulous technique ensures the stability of the electrolyte concentration throughout the experiment, enabling the acquisition of accurate and reliable conductivity measurements, this comprehensive configuration represents a robust system that offers stringent control over experimental conditions and superior durability against chemical degradation, thereby enabling an in-depth analysis of the ionic transport properties essential for the optimization of alkaline electrolyzer design and efficiency. Figure 1 provides a detailed schematic of this experimental setup, illustrating the key components described.

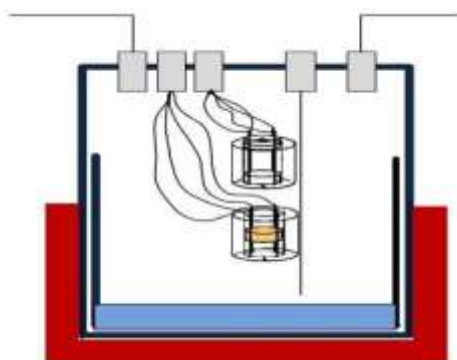


Figure 1. Schematic description of the autoclave setup.

Sample Holder and Electrode Configuration

At the heart of this experimental setup lies the sample holder, a component engineered with profound chemical and physicochemical precision, it is designed not only to withstand the hyper-aggressive chemical environment of hot, concentrated potassium hydroxide but also to optimize the accuracy of the conductivity measurements, the design leverages materials and configurations that are fundamentally chosen to enhance stability, eliminate contamination risks, and ensure consistent performance across a range of experimental conditions.

The electrodes employed in this setup are fabricated from pure platinum wires, which are subsequently coated with a layer of platinum black, this is not a cosmetic choice but a critical electrochemical one. Platinum black is a highly porous, nanocrystalline form of platinum, possessing an enormous effective surface area compared to polished platinum, this vast surface area dramatically reduces the effective current density at the electrode-electrolyte interface, thereby minimizing the phenomenon of electrode polarization. Polarization arises from the accumulation of byproducts from minor Faradaic reactions at the electrode surface, creating a counter-potential that results in erroneously high resistance readings, by using a platinized surface, the electrocatalytic activity is enhanced, facilitating these minor redox reactions and preventing charge buildup, this ensures that the measured resistance is the true bulk resistance of the electrolyte, free from polarization artifacts.

The platinum wires, 0.5 mm in diameter, are positioned with strict symmetry on opposing sides of the Teflon beaker, this geometric arrangement is a prerequisite for establishing a uniform and homogeneous electric field distribution throughout the electrolyte volume. Any asymmetry would lead to distorted field lines, creating non-uniform current pathways and introducing significant systematic measurement errors, the precise positioning ensures that every volume element of the solution between the electrodes contributes equally to the measured overall conductivity.

The selection of platinum as the core electrode material is itself critical. As a noble metal, platinum is renowned for being exceptionally chemically inert, rendering it highly resistant to corrosion even in hot, concentrated caustic solutions such as aqueous KOH. Any corrosion, however slight, would leach metallic ions into the solution, altering its ionic composition and thus its conductivity, thereby invalidating the measurement. Furthermore, platinum's high electrical conductivity ensures its own intrinsic resistance is negligible compared to that of the electrolyte, meaning the measured potential drop is almost entirely due to the solution itself.

To maintain the structural integrity and precise alignment of the electrodes, especially when subjected to thermal stress and potential mechanical agitation, they are secured by custom-designed support structures, this robust mechanical fixturing prevents any movement or bending of the platinum wires, ensuring the system's precise geometry remains constant throughout the experiment, this is an indispensable requirement for the accurate application of techniques like the Van der Pauw method, which relies critically on the fixed and known positions of the electrical contacts. Figure 2 illustrates the detailed

schematic of this Teflon-based sample holder, showcasing the precise geometric arrangement engineered specifically for conductivity measurements of aqueous KOH solutions using this advanced technique.

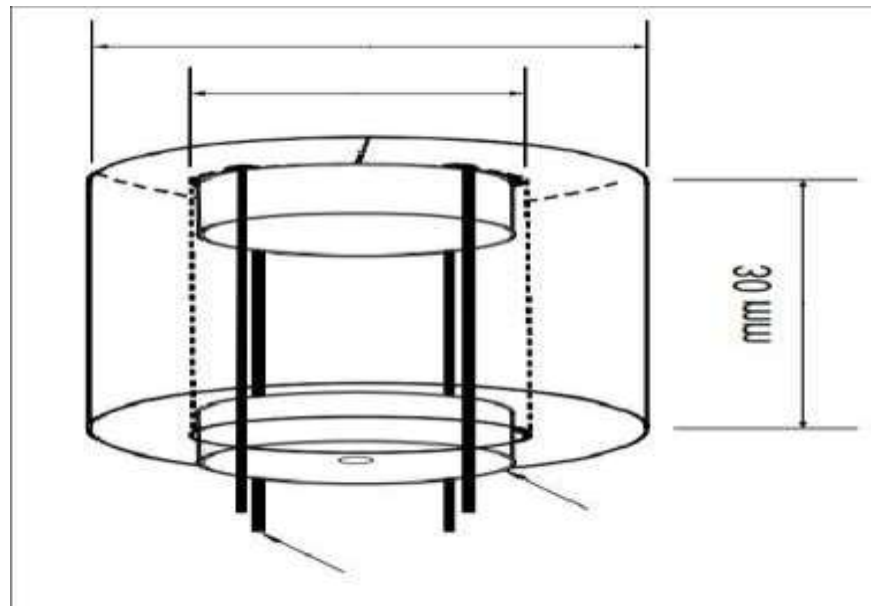


Figure 2. detailed Schematic of the Teflon-Based Sample Holder for Conductivity Measurement of Aqueous KOH Solutions Using the Van der Pauw Method

Impedance Measurement and Analysis

Impedance spectroscopy is employed to measure the conductivity of the electrolyte more precisely. Reference potentiometer Gamry 600 or 3000 is used to carry out the measurements in the "Galvanostatic Impedance Spectroscopy" mode, which involves applying a constant current (1 mA) across the electrolyte sample and the impedance, denoted as Z , is measured at different frequencies within the range of 100 kHz to 1 kHz and the impedance values are recorded between various points on the sample holder, specifically between the "working electrode", "counter electrode", reference electrode and the sense electrode for the "working electrode" and the measured impedance data is then analyzed using the following equations to calculate the conductivity of the electrolyte:

$$\sigma = \frac{\{\pi h\}}{\{\ln 2\}} \cdot \left(\frac{\{Z_{\{AB,CD\}} + Z_{\{CA,DB\}}\}}{\{2\}} \right)$$

Here:

1. (σ) is the electrical conductivity ($\frac{S}{cm}$),
2. h is the height of the sample (cm),
3. ($Z_{\{AB,CD\}}$) and ($Z_{\{CA,DB\}}$) are the measured impedances between different electrode pairs and
4. The term ($\ln(2)$) arises from the geometry of the sample holder.

Conductivity of Immobilized Electrolytes

For the conductivity measurement of frozen electrolytes, the study uses a different sample holder configuration. Figure 3 shows the sample holder design for KOH-stabilized solutions, which includes platinum wires and ceramic granules and the granule is made of perovskite powder, which is compressed under uniaxial and isostatic pressure to achieve uniformity and these granules are soaked in an aqueous solution of KOH for at least 2 h before conducting electrical conductivity measurements and the granules are fixed in place by a sturdy Teflon container and the platinum electrodes are connected to the nickel and copper wires using cold welding and this is to ensure a stable electrical connection and to reduce the effects of evaporation and condensation during measurements and entire sample holder is wrapped with Teflon tape and the setup is closed with a lid and this configuration ensures that the conductivity of the stabilized

electrolyte can be accurately measured under controlled conditions also to conductivity measurements.

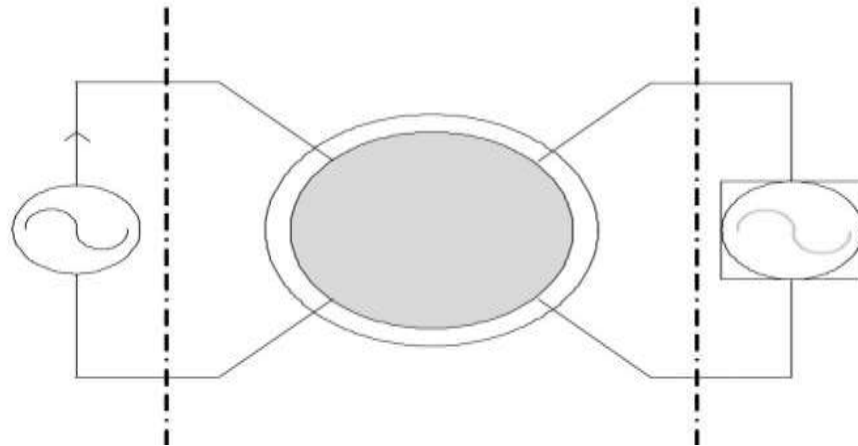


Figure 3. Potentiostat Connection to Sample Holder for Impedance Measurements

Figure 4 shows the setup for connecting a potentiostat to the specimens in order to measure the impedance of an aqueous electrolyte utilizing the "Van der Pauw technique" and the potentiostat, which is a vital instrument in electrochemical impedance spectroscopy (EIS), is used to apply a controlled current to the electrolyte sample, in this setup, the potentiostat's (WE),(CE), (RE) and (WSE) are connected to the sample holder and the purpose of the potentiostat is to generate a small AC current that will flow through the electrolyte, allowing the impedance between different electrode pairs (such as $Z_{\{AB,CD\}}$ and $Z_{\{CA,DB\}}$) to be measured and the connection between the potentiostat and the electrodes is crucial for obtaining reliable data for the conductivity of the electrolyte and the measurement of impedance at different frequencies provides detailed information about the electrolyte's ionic conductivity and other electrochemical properties and by applying an AC signal across a broad frequency range, this method minimizes the effects of electrode polarization, which would otherwise distort the measurements if a direct current (DC).

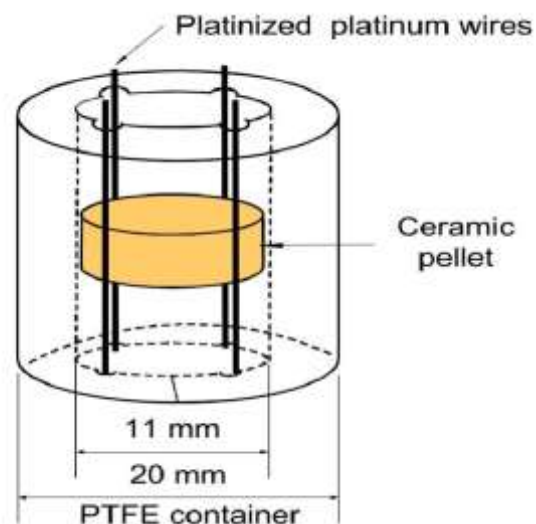


Figure 4. Setup for Measuring Conductivity of Immobilized Electrolytes

Figure 4 illustrates the sample holder designed for the conductivity assessment of immobilized electrolytes, such as those found in porous diaphragms or ceramic pellets and the holder consists of platinized platinum wires that serve as electrodes, which are

connected to the electrolyte sample and the electrodes are embedded in a ceramic pellet made from perovskite-type powder and the pellet is soaked in an aqueous.

3. Results

Phase Transition Lines of KOH

Phase transition lines for aqueous solutions of KOH are important topics in physical chemistry and materials science, as they show how the state of matter changes from a liquid to a mixture of liquid and gas under the influence of different temperatures and pressures and these lines represent the boundaries that separate the regions in which the solution can exist as a mixture of liquid and gas and the regions in which the solution remains in the liquid phase only and when studying aqueous solutions of KOH and we notice that the phase transition temperature, which is the temperature at which the solution transformation occurs from the liquid phase to a mixture of liquid and gas phase and it changes with the concentration of KOH in the solution. For pure water (0% KOH) at a "phase transition" at 100 °C temperature and 1bar pressure, which is the point at which water changes from liquid to vapor at normal atmospheric pressure also as the concentration of KOH increases, the phase transition temperature increases significantly such as when the KOH concentration reaches 50% by weight and the phase transition temperature rises to about 120°C and when the concentration reaches 80% and the temperature increases to about 196°C and this increase in the phase transition temperature with increasing KOH concentration is due to the colligative effects of the dissolved substances, increasing the KOH concentration increases the vapor pressure of the solution and thus raises the boiling point compared to pure water and this means that an aqueous solution of KOH at high concentrations becomes more resistant to evaporation and thus needs higher temperatures to turn into vapor, when working with aqueous solutions of KOH at high temperatures, such as 200°C, it becomes necessary to increase the pressure to avoid the solution turning into Vapor and ensuring that it remains in the liquid phase such as in the case of a solution containing 45% KOH by weight and it is necessary to increase the pressure to at least 10 bar to keep the solution in the liquid phase at 200 °C and in some experiments, pressures higher than 25 bar may be used and as high pressure helps maintain the liquid state of the solution and prevents boiling, which is especially important when measuring the electrical conductivity of KOH solution and the study showed that the solution pressure does not significantly affect the electrical conductivity of KOH and although high pressure raises the phase transition temperature and prevents boiling and its effect on the electrical conductivity of the solution is limited and this means that electrical conductivity depends primarily on concentration and temperature and while the effect of pressure is relatively minor in most cases [13].

Uncertainties in Conductivity Measurements

Several factors contribute to uncertainties in the conductivity measurements, including temperature differences between the measured and actual sample temperature, as well as the potential dilution of the solution due to condensation or evaporation and the temperature difference inside the autoclave was found to be a maximum of 5%, which could lead to slight measurement errors and the accuracy of the potentiostat used for impedance measurements is reported to be $\pm 1\%$, which is relatively negligible in this case [14]. A typical impedance measurement, as shown in Figure 8, exhibits a small drift (4.4%, from 0.79 Ω to 0.83 Ω over a frequency range from 100 kHz to 1 kHz), data points were generally collected at 10 kHz, where the phase angle was closest to zero, which indicates minimal capacitive or inductive contributions to the measured impedance. Considering the factors mentioned, the total uncertainty in conductivity measurements is estimated to be around $\pm 10\%$ for temperatures ranging from 25°C to 200°C [15].

Conductivity of Aqueous KOH

The results for the "aqueous conductivity" of a 35% KOH solution are shown in Figure 5, the conductivity remained relatively constant from room temperature up to 150°C, but

a slight decrease in conductivity was observed above this temperature, which has also been reported in previous studies. At 25°C, the conductivity of the 35% KOH solution was approximately $550 \text{ mS}\cdot\text{cm}^{-1}$ and it reached a peak value of $2.8 \text{ S}\cdot\text{cm}^{-1}$ at 206°C and the measured conductivity values were compared with literature data from previous study, the data for both aqueous and immobilized electrolytes were fitted using cubic regression models and the results showed that the 35% KOH solution exhibited the highest conductivity at 25°C, while the 45% solution displayed the highest conductivity at 200°C [16].

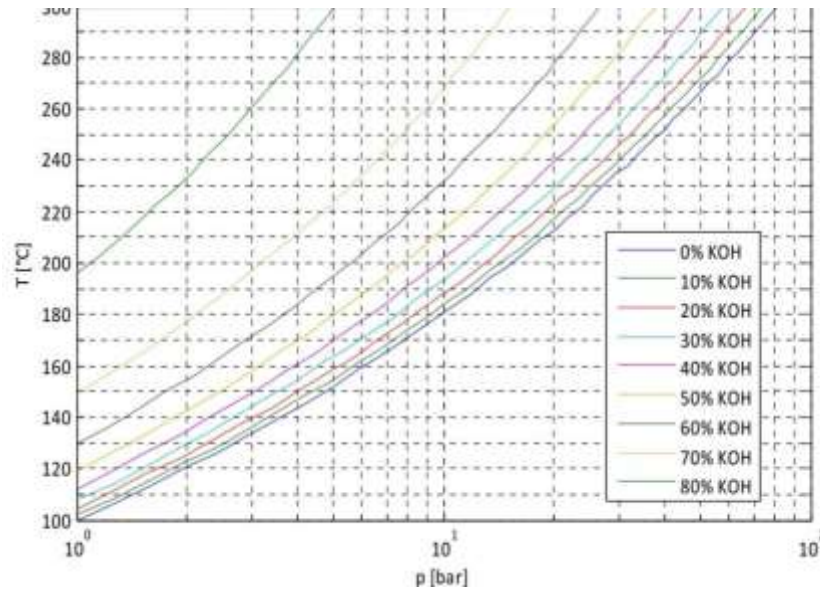


Figure 5. Phase Transition Lines for KOH Solutions.

6 shows phase transition lines for KOH solutions (0–80%) as a function of pressure and temperature, the liquid phase lies below the curves, while liquid-gas coexistence occurs above. Higher KOH concentrations require increased pressure to maintain the liquid phase at elevated temperatures, highlighting the need for pressurization during conductivity measurements near or above 200°C [17].

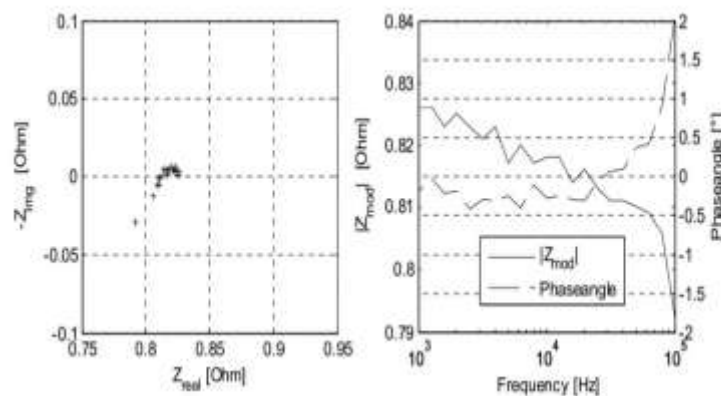


Figure 6. Nyquist and Bode Plots for Impedance Measurement.

Figure 7 shows a typical Nyquist and Bode plot for an impedance measurement (100 kHz to 1 kHz) and the Nyquist plot shows the real and imaginary components of the impedance (Z_{real} and Z_{img}) versus each other, while the Bode plot shows the magnitude of the impedance ($|Z|$) and the phase angle at different frequencies and the data shows a small impedance drift of about 4.4% over the frequency range and the phase angle close to zero at 10 kHz indicates that inductive or capacitive contributions to the impedance are negligible, making this frequency range ideal for accurate measurements of conductivity. Figure 7 displays the conductivity measurements for a 35% KOH solution as a function of temperature and the three separate measurement series (A, B, C) show similar results, with a constant conductivity slope up to 150°C and a slight decrease in

slope above this temperature. At 25°C, the conductivity is around 550 mS·cm⁻¹ and it increases to a maximum value of 2.8 S·cm⁻¹ at 206°C and the measured data (solid line) is compared with literature data from Yushkevich et al. (x symbols) [18].

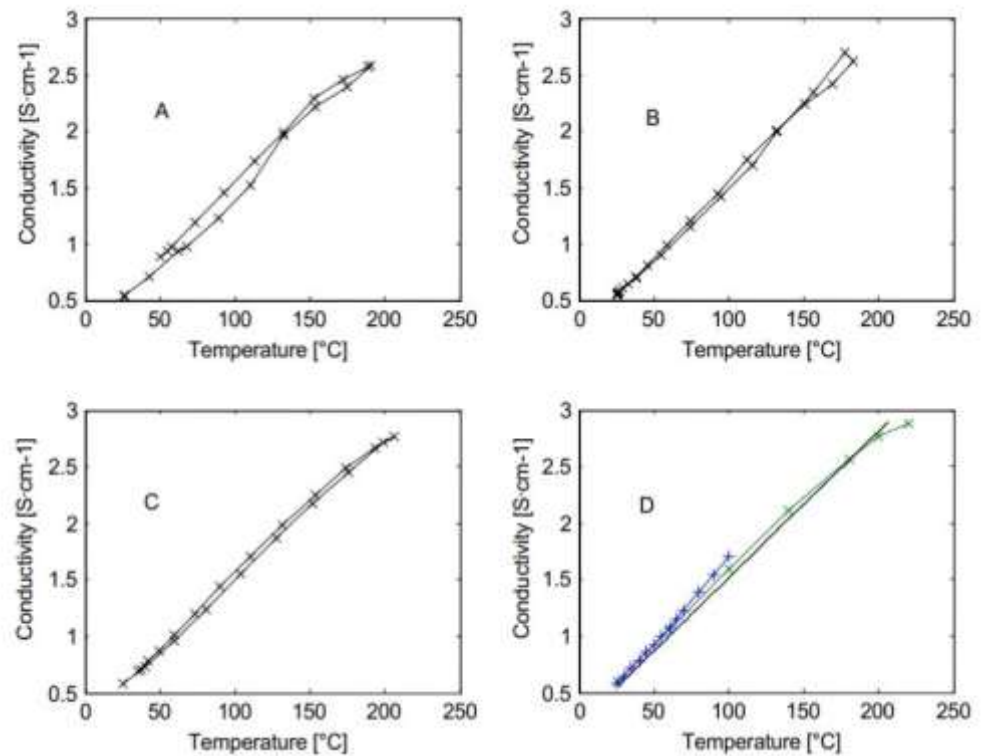


Figure 7. Conductivity of 35% KOH Solution

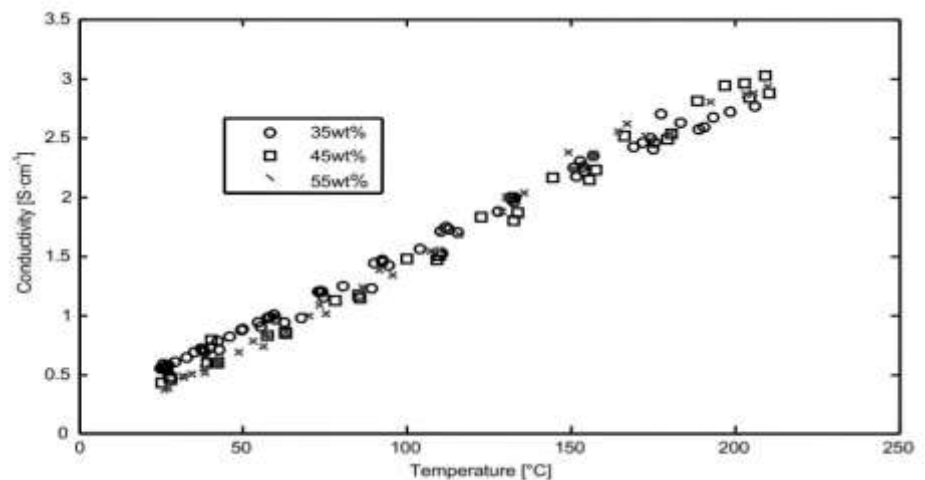


Figure 8. Conductivity of Aqueous KOH Solutions at Different Concentrations

Figure 8 shows conductivity measurements for 35%, 45%, and 55% KOH solutions, with cubic regression fits illustrating temperature-dependent behavior, the 35% solution has the highest conductivity at 25°C, while the 45% solution peaks at 200°C.

Pellet Characterization:

Porosimetry analysis of porous pellets revealed they absorb (70–85% pore volume) with KOH, leaving 15–30% unfilled. Measurements of weight, dimensions, density, and electrolyte retention were conducted. Figure 9 highlights the pellets' internal structure and its impact on electrolyte retention, while Table 1 details their physical and absorption properties (Table 2).

Table 2. Pellet Characteristics Comparison

KOH Concentration	Dry Weight [g]	Wet Weight [g]	Diameter [mm]	Height [mm]	Relative Density [%]	Specific Density of KOH at 25°C [g/cm ³]	Pores Filled [%]
35%	1.310	1.650	11.3	5.1	52	1.35	83
45%	1.360	1.670	11.2	5.4	51	1.46	80
55%	1.410	1.690	11.1	5.3	50	1.57	74

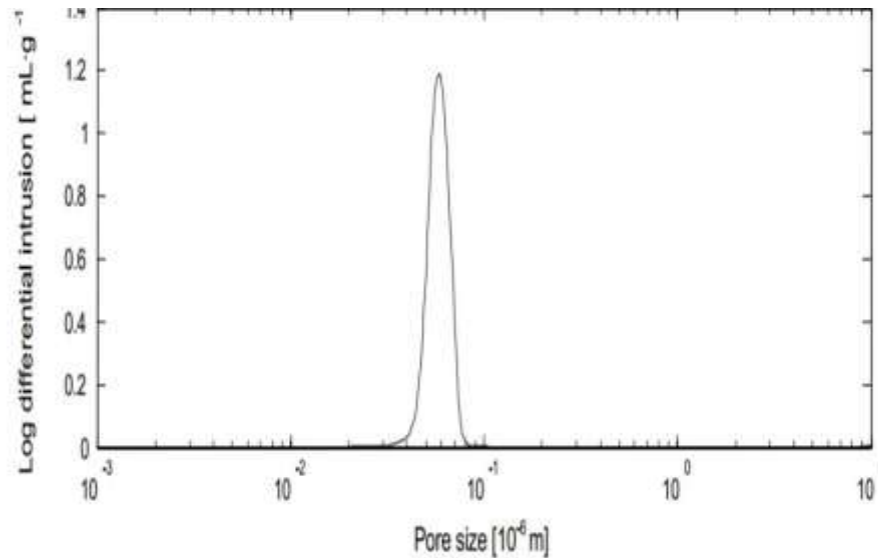


Figure 9. Porosimetry results of the pellets.

Conductivity of Immobilized Aqueous KOH

conductivity for immobilized KOH solutions in porous pellets were taken over a temperature range, with the concentration set at 35%. **Figure 10** illustrates the relationship between temperature and the measured conductivity values. At a constant pressure of 30 bar, the conductivity starts at 0.13 S·cm⁻¹ at 24°C and increases steadily to 0.68 S·cm⁻¹ at 221°C. Notably, the conductivity seems to plateau, reaching a maximum value around 220°C, indicating a temperature-dependent behavior and in **Figure 11**, the achieved data points for immobilized KOH are shown, along with cubic fitting curves for each concentration. For the 45% KOH pellets, the conductivity increases with higher temperatures, whereas the conductivity of 55% KOH pellets remains relatively constant throughout the temperature range. For the 35% KOH pellets, the conductivity exhibits a decrease in the slope above 160°C, showing that higher temperatures have a diminishing effect on the conductivity at lower concentrations.

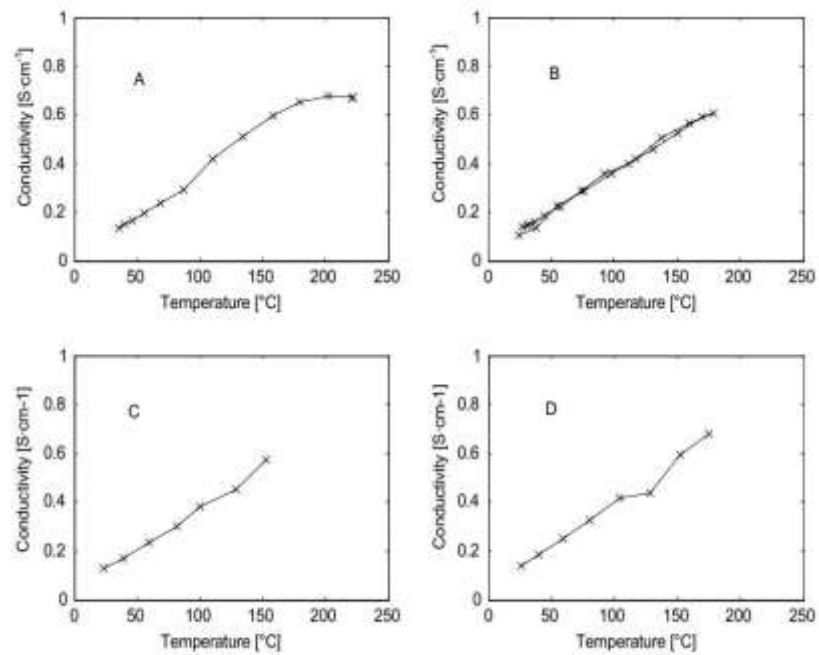


Figure 10. Measured conductivity of immobilized KOH

Conductivity analysis and regression modeling

The temperature-dependent conductivity can be calculated using a cubic equation of the form:

$$\sigma(T) = C_1 + C_2T + C_3T^2 + C_4T^3 = C1 + C2T + C3T^2 + C4T^3$$

Where:

1. $\sigma(T)$ is the conductivity at temperature T in $^{\circ}\text{C}$,
2. T is the temperature in $^{\circ}\text{C}$.

The "coefficients for cubic" fitting are listed in **Table 3**, which allows for the calculation of conductivity values over a range of temperatures for both aqueous and immobilized KOH solutions at different concentrations.

Table 3. Coefficients for Cubic Fitting

Conductivity Type	Concentration [%]	C_1	C_2	C_3	C_4
Aqueous stabilizer	35.05	-2.10E-07	6.50E-05	0.007680	0.330000
Aqueous stabilizer	35.5	-9.40E-08	2.90E-05	0.000905	0.089000
Aqueous stabilizer	43	-1.70E-07	7.10E-05	0.005980	0.271000
Aqueous stabilizer	43	7.40E-08	-2.10E-05	0.005750	-0.027500
Aqueous stabilizer	54	-3.20E-07	1.10E-04	0.005210	0.211000
Aqueous stabilizer	54	-2.30E-08	8.80E-06	0.002780	0.028500

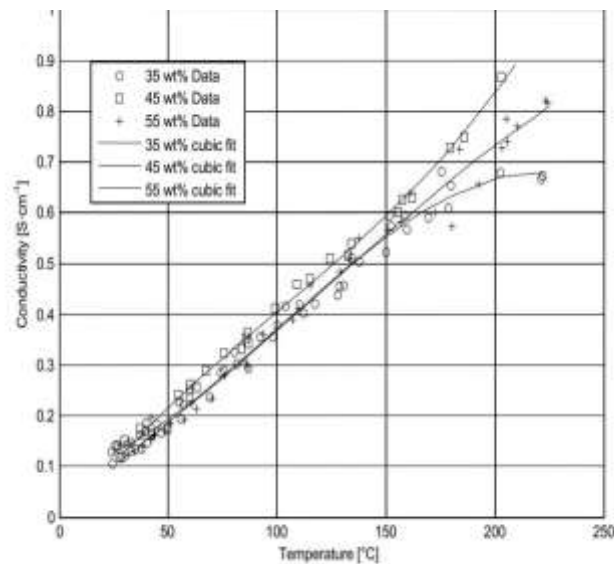


Figure 11. Conductivity measurements and cubic regression analysis of KOH at 35%, 45% and 55% across temperature ranges.

Where the measured water conductivity values for KOH solutions as shown in Figure 12 are 35% and 45% KOH solutions and in Figure 12, the correlation between the measured data and the cubic fitting curves for 35% (A) and 45% (B), is shown. And 55% (C) KOH, the cubic fits show good agreement with measured conductivity values across the temperature range, confirming the validity and reliability of the experimental approach. The highest conductivity value achieved for a 45% stabilized KOH solution was 0.84 cm^{-1} at 200°C , this result demonstrates the great potential of the stabilized KOH electrolyte in terms of conductivity, as weight measurements of the porous pellets (presented in Table 2) reveal that the pores are not filled. Completely electrolyte-based This indicates that the conductivity can be further improved through more optimized wetting procedures to ensure that unfilled pores are sufficiently saturated with Electrolyte. It is important to note that the structure used in this study can achieve lower region-specific resistance if the thickness of the pellets is reduced to $200 \mu\text{m}$ or thinner and this reduction in thickness can lead to a significant reduction in ohmic resistance electrolyte losses. In addition, the proposed structure can be operated at higher temperatures (up to 200°C or higher). Operating temperatures, such as in industrial electrolyzers or fuel cells.

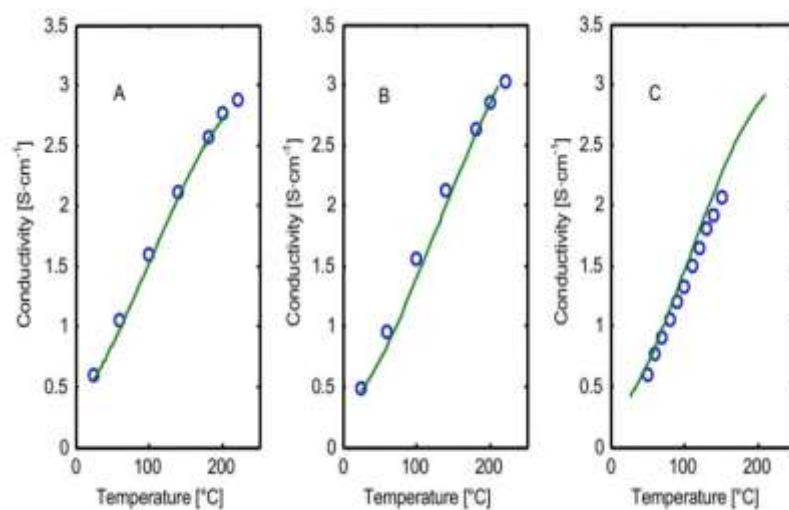


Figure 12. Comparison of Experimental and Literature Data for Aqueous KOH Conductivity at 35%, 45%, and 55%.

The relationship between water conductivity and electrostatic conductivity

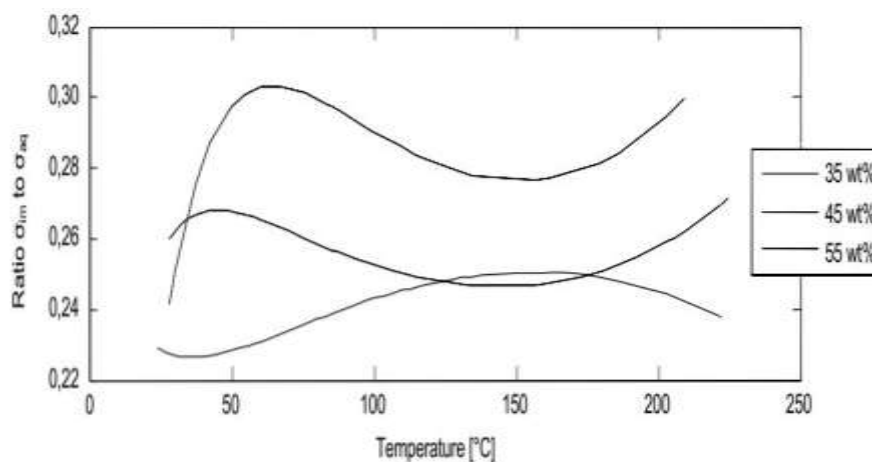


Figure 13. Ratio of the conductivity of immobilized KOH, σ_{im} , to the conductivity of aqueous KOH, σ_{aq} , for three different concentrations.

The relationship between the conductivity of stabilized KOH in the pellets and the conductivity of aqueous KOH (Figure 13).

The results obtained about phase transition lines and conductivity kinetics in KOH solutions showed the importance of the interaction between thermal and pressure factors on the state of the material and the behavior of the solution. The results confirmed that increasing KOH concentration exerts fundamental effects on the phase transition temperature and system pressure needed to maintain the liquid phase, as the boiling point of the solution increases with increasing KOH concentration. These observations are consistent with collective effects of solutes, where increasing pressure and temperature lead to enhanced stability of the liquid phase [19].

Regarding electrical conductivity, the results showed remarkable agreement with previous studies. It was observed that the conductivity increases with increasing temperature until it reaches its peak at high temperatures (206°C), which indicates improved ion movement when the thermal energy of the system increases, before the conductivity begins to decline at high concentrations due to the effects of reducing ion movement upon saturation and conductivity measurements in porous structures covered with aqueous solution of KOH showed a decrease in conductivity compared to the liquid form due to the effects of porosity and the complexity of the ionic transport structure [20]. This was confirmed by the conductivity ratio (stable compared to liquid) that showed a decrease due to the effects of closed voids and the complexity of ion movement, inferred cubic models indicated a dependence of conductivity on temperature and concentration, making it possible to improve electrical performance by reducing the thickness of porous structures or improving solution saturation. These results support their use in industrial applications such as cellular electrolysis and fuel cells [21].

4. Conclusion

This electrical conductivity of aqueous KOH solutions at 35%, 45% and 55% across a broad temperature range and the measurements were performed using the van der Pauw method, a standard technique for assessing conductivity in thin, flat samples. Among the different concentrations, the 45% KOH solution demonstrated the highest conductivity at elevated temperatures. Specifically, at 200 °C, the conductivity was calculated to be 2.9 S·cm⁻¹, based on a cubic fit applied to the experimental data, in addition to the aqueous KOH solutions, the study also examined the conductivity of KOH immobilized in a nanoporous structure, which was measured for the same three concentrations and the immobilized 45% KOH solution exhibited a conductivity of 0.84 S·cm⁻¹ at temperatures near 200 °C, which was the highest among the immobilized solutions and the conductivity

values for the immobilized 35% and 55% KOH solutions were slightly lower, at $0.67 \text{ S}\cdot\text{cm}^{-1}$ and $0.73 \text{ S}\cdot\text{cm}^{-1}$, respectively and these findings highlight that the conductivity of the immobilized KOH solution is significantly lower than the aqueous form, particularly at higher concentrations.

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