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Developing Tailored Materials for the Industrial Production of Anion Exchange Membrane Electrolyzers through a Statistical Approach

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With the aim of better identifying possible solutions to the actual critical issues regarding the uncertain performance of anion exchange membrane (AEM) electrolyzers, analytical mathematical models related to their materials' selection are developed through multivariate analysis. An objective evaluation and selection of such solutions are herein proposed through a statistical approach, based on multivariate data analysis (ANOVA), involving data from literature based on academic and commercial datasets. First, a classification of the materials of interest is made; thereafter, three electrolyzer properties are evaluated as responses (cost, current density, and time stability). In the results, it is demonstrated that each property considered is promoted by a different material's combination. For example, the highest current density (>1123 mA cm^{-2}) is achieved by employing KOH as electrolyte, whereas the lowest assembly cost (≈2 \$ Kg^{-1}) is related to platinum-based cathodes coupled with membranes at a higher loading thickness (130 µm). The selection of materials in this way will aim to gather the most suitable ones based on objective criteria, which will further drive the development of electrolyzers that are increasingly efficient and sustainable.

1. Introduction

Hydrogen-related technologies are recognized as one of the most promising solutions to overcome the continuous increase of the energy demand, but more research is needed for their mass application.[1] In this context, new materials research and development play a main role as the currently most established materials have several critical issues. Among those are the use of relatively expensive but high efficiency materials, such as platinum in proton exchange membrane electrolyzers and copper in anion exchange membrane (AEM), or metals that are poorly durable but inexpensive, such as nickel and derivatives for AEM electrolyzers.[2] Regarding materials and taking into account AEM's principal limitations which are membrane durability and hydrogen production cost, some intermediate configurations can be considered, such as the design of composite or multilayer. However, the benefit/cost ratio must be carefully considered since, for example, end-of-life disposal costs also must be taken into account, and this may be greater as the material is composed of several phases. Membrane durability is one of the major challenges as the present membrane's durability is around 30 000 h (with a target of 1 000 000 h) due to the polymer degradation from the membrane backbone chain (chemically unstable). Regarding the hydrogen production cost, this value is actually around 1279 \$ kW/H2^{-1} (2020), with a targeted cost ≤300 \$ kW/H2^{-1} (2050). Recent research has made significant strides in the design of AEM electrolyzers, particularly in the development of efficient membrane electrode assemblies.[3] These advancements have led to the successful production of green hydrogen, with the potential for commercial viability.[4] However, challenges remain in achieving high stability and performance, particularly in the oxygen evolution reaction and the long-term durability of the catalysts.[5]

In these terms, the selection of materials for these technologies is crucial, with a need for more sustainable and efficient options.[6,7] This has led to the development of analytical mathematical models for material selection, which consider factors such as environmental and economic sustainability, production efficiency, and industrial scale-up.[7] The use of renewable energy sources in electrolysis for hydrogen production has also been explored, with a focus on different processes and their prospects to overcome material concerns.[8] Zhang et al. demonstrated the potential of Mo_2C/N-doped graphene nanocomposite as a catalyst in a microbial fuel-cells-ammonia electrolytic-cell-coupled system.[9] Palhares et al. developed a low-cost electrolyzer for hydrogen production, combining alkaline water electrolysis and solar energy use.[10]

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A range of membrane classifications for AEMs have been proposed. Hagesteijn et al. categorized AEM head groups into nitrogen-based, nitrogen-free, and metal cations, with the latter showing promise due to their stability.[11] Li et al. introduced a highly durable long side-chain polybenzimidazole AEM with high conductivity and chemical stability.[12] Han et al. presented elastic long-chain multi-cation cross-linked AEMs, which are both flexible and highly conductive.[13] Various synthesis strategies for AEMs have been explored, including those derived from Nafion precursors, prepared by grafting technologies, based on functionalized commercial polymers, and containing heterogeneous compositions.[14] The most recent research has also focused on the development of efficient and cost-effective membrane materials for AEM electrolysis in hydrogen production. Zakaria et al. and Vincent et al. both highlighted the potential of AEM electrolysis, with the latter specifically noting the use of non-noble catalysts to reduce capital costs.[4,15] Vincent et al. further explored the use of an ultrathin AEM, which offers improved hydroxide ion transfer and stability.[4] These studies collectively underscore the importance of membrane materials in AEM systems.

Regarding cathode and anode materials, Manolova et al. explored the use of various substrates and coatings, finding that rougher alloy structures led to better electrochemical properties.[16] Kuleshov et al. reported on the development of high-performance composite cathodes, which significantly reduced power consumption for hydrogen production.[17] Nam et al. proposed Li-compound anodes as a promising class for high-performance Li-ion battery anodes, with specific examples of LiSn and Li_5SnP_3 nanocomposites.[18] Glucina et al. investigated a binary Cu–Al alloy as an inert anode for aluminum electrowinning, noting not only its satisfactory performance but also substantial corrosion.[19] Wang et al. introduced a novel electrode for value-generating anode reactions in water electrolyzers, capable of sustaining high current densities for the oxidation of organic molecules.[20]

The classification of electrolytes in electrolyzers is a complex and multifaceted topic. Khokhlov et al. provided a comprehensive study of molten salt electrolytes, categorizing them based on their physical and electrochemical properties.[21] Verma et al. further expanded on this, discussing the various types of electrolytes used in electrochemical supercapacitors, including aqueous, organic, ionic liquid, solid state, and redox.[22] Tan et al. focused on electrolyte engineering for high-voltage aqueous energy storage devices, discussing strategies to expand the electrochemical stability window.[23]

The use of experimental design in historical data evaluation is a valuable tool, as demonstrated by Gaudard et al. who applied recursive partitioning to narrow down potential factors for a Six Sigma project.[24] Santis et al. emphasized the importance of Bayesian sample size determination, particularly in the use of historical data, and discussed the use of power priors to account for heterogeneity.[25] Krange et al. and Pedersen et al. further emphasized the importance of considering the nature of the design experiments method and the potential of design historical cases.[26,27] In strong similarity, ANOVA has a rich historical background and its application in unbalanced factorial designs has been traced from the 1930s to 1960s.[28] More recently, ANOVA has also been used in the analysis of proportions, as a statistical method for incorporating historical control data.[29,30]

Although the research on materials for AEM is widely increasing, systematic studies regarding the comparison among many different assemblies are rare because of the many variables that must be taken into account. Thereafter, in this study, with the aim of underlying the performance of AEM electrolyzers under the employment of different materials, and as an innovative part with respect to previous literature, analytical mathematical models related to the selection of cathode, electrolyte, and membrane materials have been developed through multivariate analysis. Using these analytical methods, it is possible to calculate numerically how much an input factor (e.g., cathode or membrane material) affects the response variables, thus estimating not only the combinations of variables that result in better performance but also by how much. As further innovative aspect of this article, the coordinated research on AEM electrolysis herein proposed is focused to improve not only the performance of the assembly by analyzing the current density but also the stability overtime by reducing the H_2 production costs.

2. Results

2.1. Input Factors Selection

For the fruitful application of the design of experiments (DoE) method, first, practical considerations on available data in literature were done, and seven input factors were chosen among materials and operation conditions.[2,31–34] In particular, membrane backbone material and its thickness and electrolyte were considered as ones of the possible major factors that could affect stability overtime as well as costs. Furthermore, with the aim of limiting the input factors and, consequently, the needed dataset population, at the same time improving the innovative part of this study, only cathode materials and their loading were considered. In fact, it is recognized in literature that cathode materials are actually less investigated with respect to anode materials. In addition, operation conditions such as applied temperature and voltage were included as environmental conditions of employment that could affect the AEM performance. In **Table 1** and **2**, the levels of each factor (numerical and categorical) are detailed among those found in literature.

2.2. Current Density

Recent advancements in AEM electrolyzers have significantly improved their current density and durability. Chen et al.

Table 1. Limits and levels of the input variable considered.

Name	Units	Type	Subtype	Minimum	Maximum	Levels
Backbone structure		Categoric	Nominal			6
Thickness	μm	Numeric	Continuous	25	130	
Electrolyte		Categoric	Nominal			4
Cathode		Categoric	Nominal			4
Loading	$mg\,cm^{-2}$	Numeric	Continuous	0.40	85	
Voltage	V	Numeric	Continuous	1.59	2.29	
Temperature	°C	Numeric	Continuous	25	80	

Table 2. Levels of the categorical factors.

Backbone structure	Electrolyte	Cathode
Hydrocarbon (FAA-3-50)	KOH	Pt
Quaternary Ammonium - QA (A201)	H_2O	Ni
PSF (Polysulfone)	NaOH	$CuCoO_3$
Copolymer (Sustainion X37-50)	K_2CO_3	Pt/C
Mg–Al		
Polyphenylene		

achieved a current density of 7.68 A cm^{-2} and a durability of 1000 h, while Lee et al. demonstrated a high-efficiency AEM water electrolyzer with a cell current density of 2.1 A cm^{-2} at a voltage of 1.8 V.[35] Nevertheless, the quantitative correlation between materials and current density is not clearly assessed in literature. Regarding the current density response, in this study, five reliable factors have been detected by ANOVA analysis, having a p-value < 0.001. These are electrolyte, loading, temperature, loading–temperature interaction, and loading–electrolyte interaction. The overall R^2 of the model is 0.69. The mathematical models are expressed graphically in **Figure 1**. Different behaviors can be evaluated depending on different electrolytes; in fact, KOH, the most commonly employed electrolyte, is capable of yielding the highest current density (>1123 mA cm^{-2}) for a wide range of loading at temperatures above 69 °C, whereas for the other electrolytes only low loading are capable to assess enhanced values of current density, always at relatively high temperatures (over 65–70 °C). Among those conditions, the worst is given for water employed as electrolyte. It is also interesting to note that cathode loading seems the most valuable factor to consider, as it is statically reliable in single and in interaction with two other factors (electrolyte and temperature).

2.3. Costs

The evaluation of this response is related to the H_2 cost ($ Kg^{-1}) and is referred to an assembly based on RuO_2 as anode from which the numerical values have been collected and details

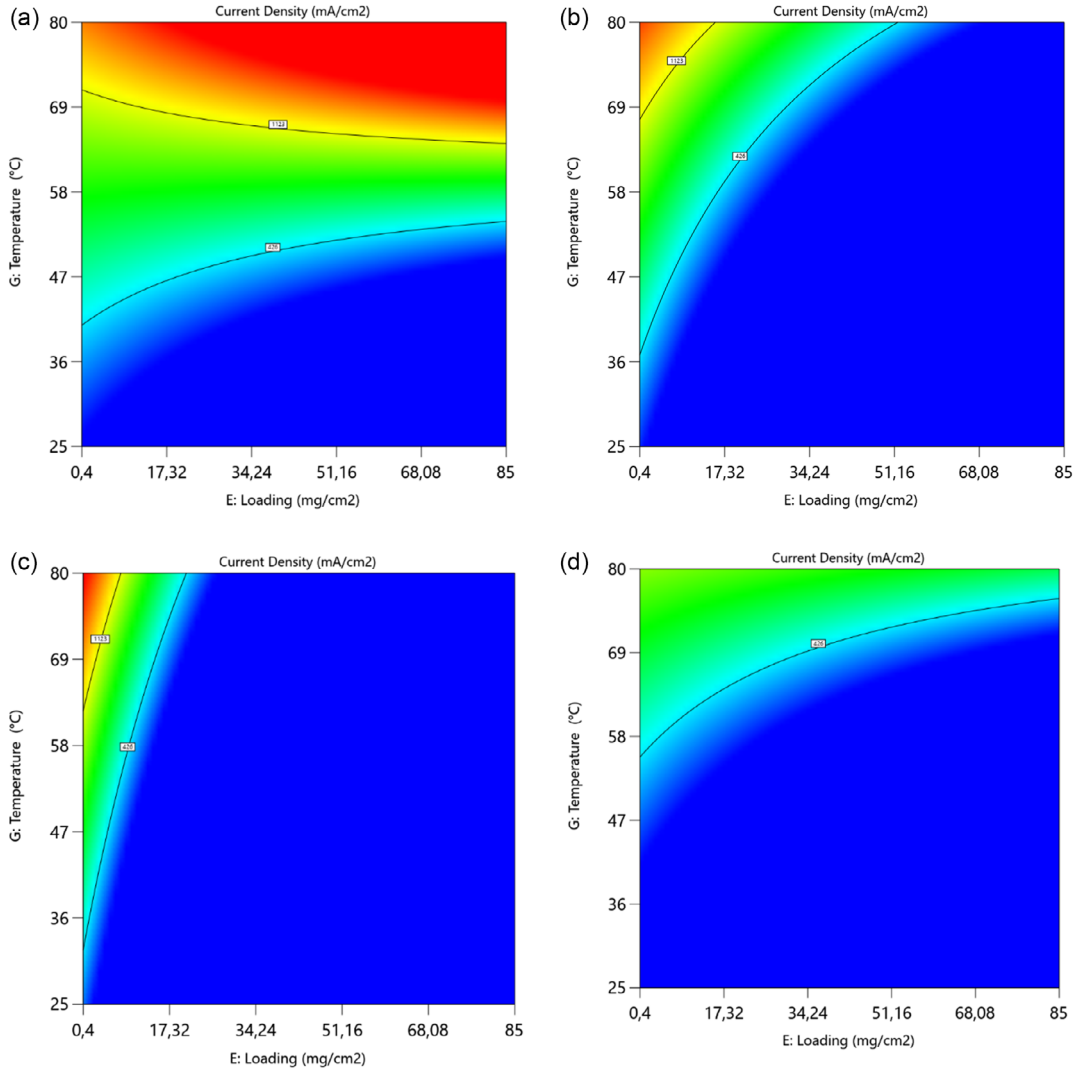

Figure 1. Contour plots of the response current density considering the behavior of different electrolytes: a) KOH; b) NaOH; c) K_2CO_3; and d) H_2O.

are reported in Abbasi et al.[36] The results of the mathematical analysis agreed that the statistically significant factors are backbone structure, thickness, cathode, and the backbone structure–thickness interaction. This model has a p-value < 0.001 and an $R^2 = 0.99$. Regarding the trend of this response, as shown in **Figure 2–4**, it is clear that the lowest H_2 costs are due to specific materials employed as cathode, in particular Pt and Pt/C, independently from the other considered factors. Even if the absolute lowest value of the cost response depends on the membrane thickness and backbone interaction as well. In fact, for thickness equal to or below 75 μm (Figure 2), hydrocarbon, QA, and copolymeric membranes represent the less expensive solutions; instead of over 75 μm (Figure 3 and 4) of thickness, other types of membranes should be considered, such as PSF, to reduce costs. This trend demonstrates that the membrane materials structure plays a key role in cost reduction, but it can't be separated from other factors such as, first, its own thickness and cathode material. In contrast, factors considered in the experimental design, but not significant for this model, such as type of electrolyte and temperature, are not relevant and must be evaluated as not influencing the H_2 cost.

2.4. Time Stability

The AEM stability overtime is the more challenging response to evaluate as it is often missing from an experimental evaluation due to the high time needed to achieve those types of data; but even in this case, through the methodology applied in this study, a reliable mathematical model with R^2 well above 0.9 has been obtained. For this model, the reliable factors are backbone structure, thickness, and backbone structure–thickness interaction. In this case, it is clear that only two factors are significant from the statistical point of view, and as indicated in **Figure 5**, the leading factor is the backbone structure as higher stability overtime

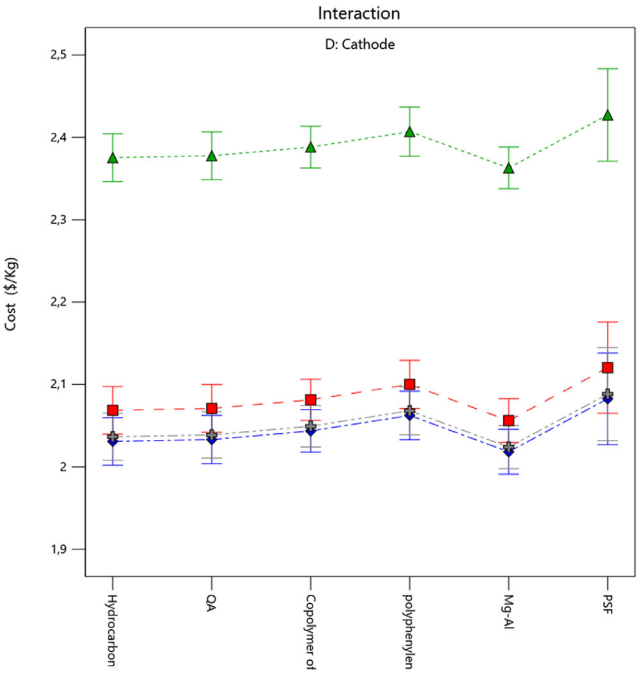

Figure 2. Contour plot for the response cost and different cathodes when a membrane having a 25 μm of thickness is considered: $CuCO_3$ (green), nickel (red), platinum (grey), and platinum/carbon (blue).

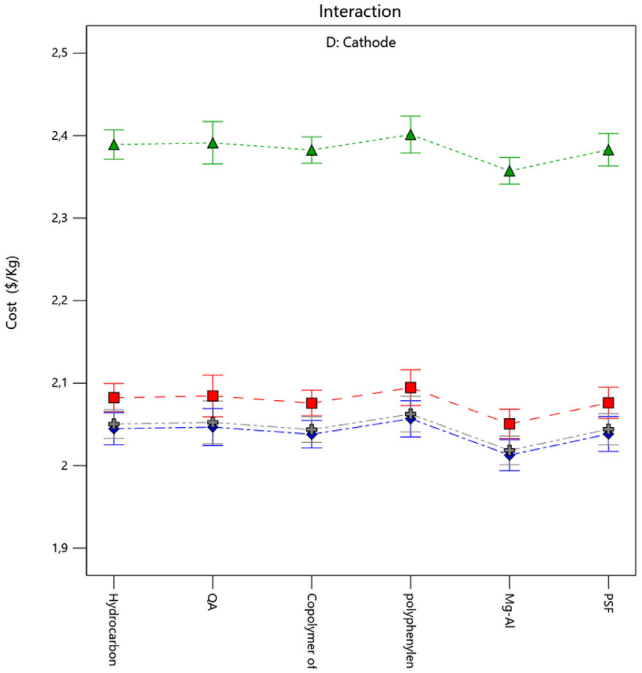

Figure 3. Contour plot for the response cost and different cathodes when a membrane having a 75 μm of thickness is considered: $CuCO_3$ (green), nickel (red), platinum (grey), and platinum/carbon (blue).

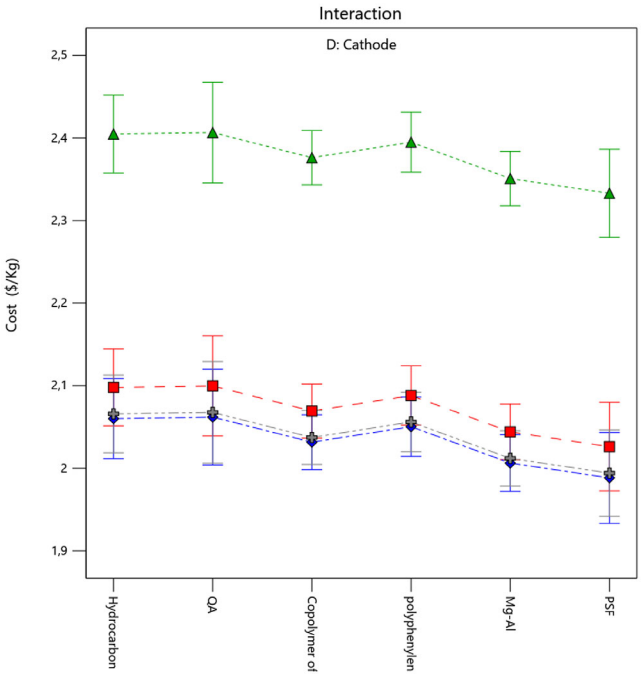

Figure 4. Contour plot for the response cost and different cathodes when a membrane having a 130 μm of thickness is considered: $CuCO_3$ (green), nickel (red), platinum (grey), and platinum/carbon (blue).

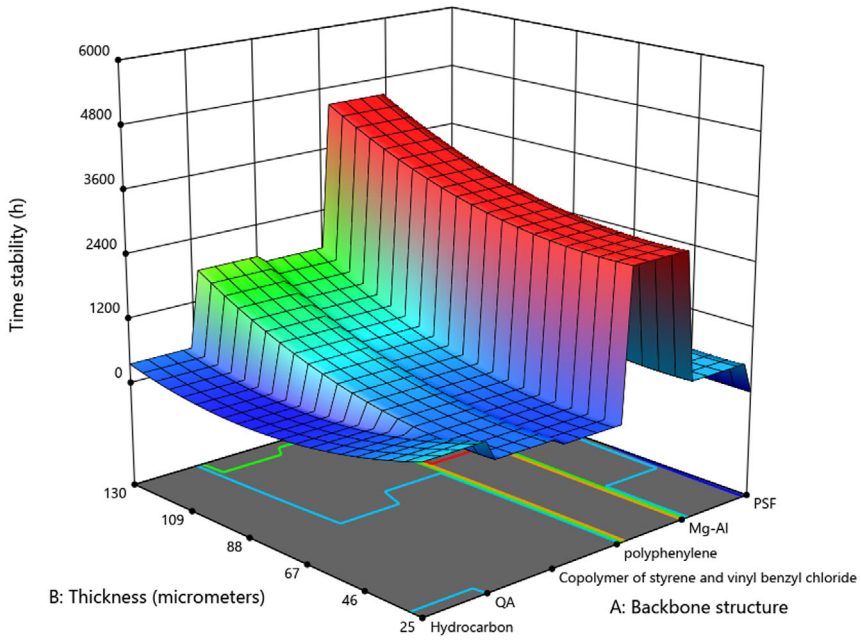

Figure 5. Contour plot for the time stability response.

is given from polyphenylene membrane. Nevertheless, also thickness must be considered as, in general, higher thickness provides higher time stability, and this is the case for polyphenylene membranes. Polyphenylene membranes, including polyethylene (PE) and polyphenylene sulfide (PPS) membranes, offer a range of benefits in various applications. Zuo et al. highlighted the impressive separation performance and stability of PE membranes in desalination, while Gao et al. discussed the unique properties of PPS membranes, such as corrosion resistance and thermal stability, in membrane separation processes.[37,38] In electrochemical devices, polyphenylene-based membranes, with their high C—C bond dissociation energy, are being explored as alternatives to perfluorosulfonic acid ionomers.[37] Sutton-Sharp et al. further underscored the cost-effectiveness and reliability of polymeric membranes, including polyphenylene membranes, in oil- and gas-produced water treatment.[39] These membranes offer high oil rejection rates, good resistance to fouling, and a longer life expectancy compared to conventional technologies.

2.5. Desirability Function Discussion

Several studies have explored the interaction among the assembly constituents on the cost-effective fabrication of AEMs for various applications. Vincent et al. identified plasma-sprayed electrodes as key components of AEM electrolysis that contribute to achieving high dialysis coefficients and separation factors,[40] while Cheng et al. proposed simple and economical methods for AEM fabrication, demonstrating good chemical and thermal stability.[41,42] Haq et al. further improved AEM performance by synthesizing a membrane with high ion-exchange capacity and low water uptake, leading to enhanced salt adsorption capacity and current efficiency.[43] In this context, the present study proposes

Table 3. Desirability function limits.

Name	Units	Importance	Goal
Current density	mA cm^{-2}	3/5	To maximize
Cost	\$ Kg^{-1}	4/5	To minimize
Time stability	h	4/5	To maximize

with mathematical models an objective choice among several different materials and operating conditions. However, considering the obtained mathematical models, as reported in Section 2.2–2.4, it is clear that each response is driven by different factors, and, thereafter, more effort is required to calculate the overall best combination among the three responses. Having as final objectives, the conditions shown in **Table 3** for the response variables, in **Figure 6**, the factors' combination that satisfies all of them is reported as well as a prediction of the obtained responses.

3. Conclusion

Employing the DoE approach, it was possible to uncover rules and paths that lead to promote current density and long-term stability with lower cost in AEM electrolyzers. Normally these paths are difficult to accomplish by conventional methods, due to the complex relationships of these variables with the performance of the electrolyzer. With the help of this method, we were able to quantify how current density, costs, and time duration are affected by appropriate combinations of materials and environmental factors. We demonstrated in a statistically reliable way ($p < 0.001$) that current density, electrolyte, loading, and

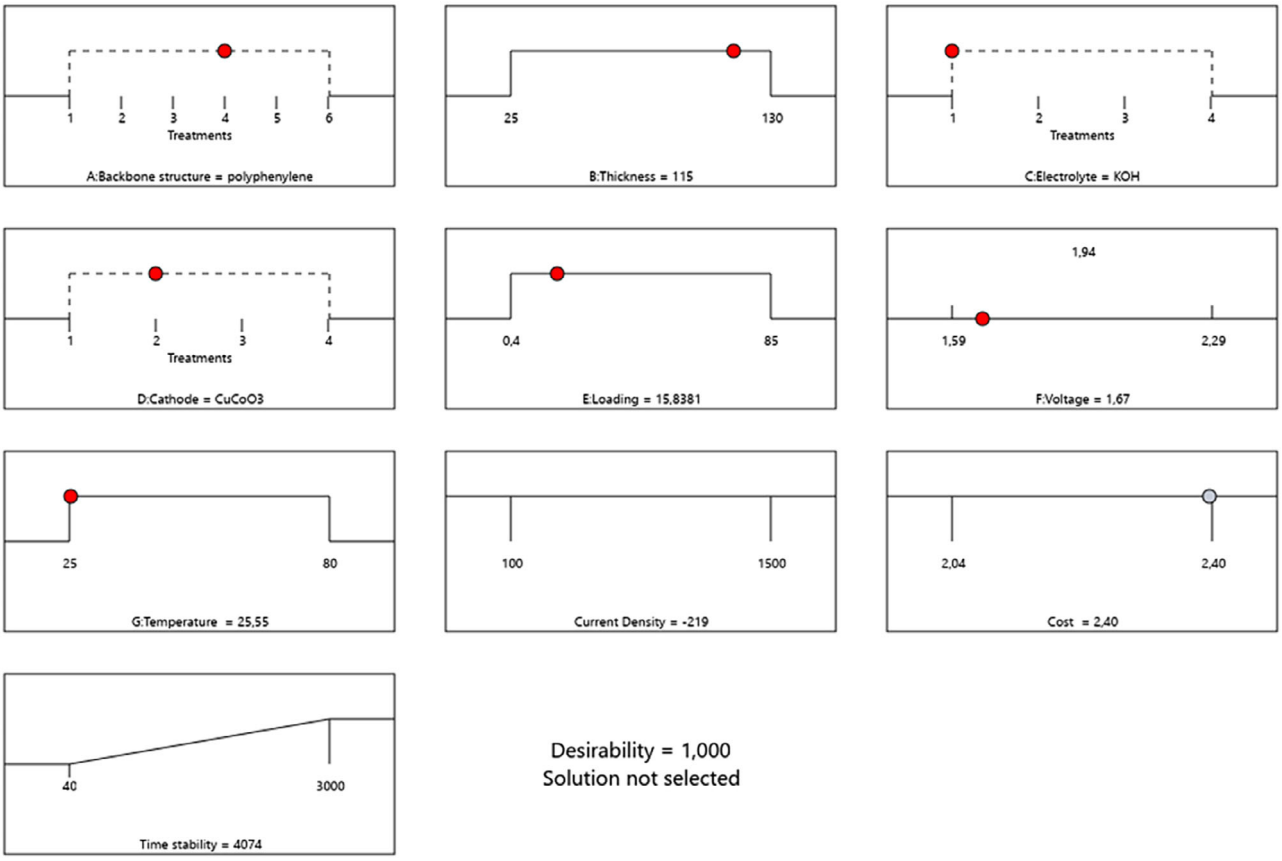

Figure 6. The desirability function results in terms of input and output (calculated) variables.

temperature are the most important factors to consider. On the opposite, costs and time stability are mainly affected by the membrane (materials and geometrical factor) and cathode materials. Further optimization should involve additional variables such as support and surface elements, as well as gas diffusion layers of both cathode and anode, despite their increased complexity to codify within this framework This methodical selection of materials will aim to assemble the most suitable components based on objective criteria, that will enable the advancement of electrolyzers toward enhanced efficiency and economic sustainability.

4. Experimental Section

In this work, the DoE approach and analysis of variance were employed for historical data analysis. To analyze data as homogeneous as possible, five datasets were considered, as a collection of dozens of research articles.[2,31–34] Loading in particular was estimated through the difference between the mass of the support loaded before and after catalyst deposition for all the considered samples.[31] A database of 30 experimental data entries was prepared, reporting information from the more recent and complete literature on AEM electrolyzer materials. Only articles published in the last 10 years were used to get the data. This was done so that the database would be in accordance with current trends in this field. Because it was necessary to have a database completely filled, to apply DoE techniques, the articles that were lacking data were eventually ignored. Data analysis was conducted through a rational approach based on multivariate data analysis (ANOVA) capable of deriving objective results from these literature data.[44] This approach was herein employed to obtain the maximum amount of information possible to support the generation of robust and predictive mathematical models without further experimental work to determine the positive or negative effects of a variety of descriptor variables on current density, cost, and working time. According to this method, it was also possible to evaluate discrepancy among the employed data, which were unexplained by the selected input variables, indicating that unreliable models relied on a greater part of the variance that remained unexplained. In this sense, the obtainment of reliable methods suggested also the substantial homogeneity of the data and vice versa. The Design Expert 13 software was used to implement the data set and perform the analysis. Three responses were evaluated to assess the cost and performance of the selected materials and environmental factors: cost, time stability, and current density. In addition, for each response, also goal and importance were defined to build the desirability function, the mathematical equation that summed up all three responses through a weighted average. The highest importance was given to cost and time stability, as were the two requirements more difficult to satisfy, whereas average importance was given to the current density. Regarding the goal, for each response, the expected objective was defined as reducing H_2 cost as much as possible but increasing current density and time stability (Table 3).

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Conflict of Interest

The authors declare no conflict of interest.

Author Contributions

S.B.: writing—original draft preparation and investigation; G.D.: data curation, writing–original draft preparation, and investigation; L.M.: conceptualization, data curation, resources, and project administration; M.M.: conceptualization, supervision, and resources; M.M.: resources, writing—review and editing, and supervision.

Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

Keywords

assemblies, cathodes, design of experiments, hydrogens, membranes

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