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Application of an Experimental Design for Optimizing the Conditions of Ceramic Membranes Elaboration

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ABSTRACT

The present work is focused on studying the porosity of a ceramic membrane elaborated using aspects of the experimental design. A complete factorial design 2^3 was developed in order to study the effect of physical and chemical characteristics of flat or tubular membranes based on Moroccan clay. The main factors investigated are the sintering temperature as a thermal parameter, the granulometry for the particle size distribution, and the starch content being an organic additive that contributes to the final porosity. The experimental results showed that the powder of clay has to be crushed and sieved at 125 µm, the sintering temperature support as 850 °C and the starch content as 10%. The optimization of the results revealed that the response value of porosity is 40.775%. Finally, the experimental design allows us to better understand the correlation between process variables and to interpret test results.

1. Introduction

In the last few years, the elaboration of the ceramic porous membrane has attracted a lot of research due to their different applications like desalination of seawater [1], domestic wastewater treatment [2], gas separation [3] and catalysis [4]. The development of ceramic membranes based on cheap natural materials to reduce the cost of conventional ceramic membranes made from expensive materials like alumina, silica, titania and zirconia, waste materials have been investigated by several authors [5–12]. The previous works of our laboratory describe the utilization of local materials such as clays [13–16], animal bones [17,18], phosphate [19,20] in both tubular and flat ceramic membranes.

The optimization of development conditions by experimental designs [21] allows to express clearly and quantitatively the impacts on membrane performance and to detect the interactions between factors that influence their quality, which will allow in the future to make a prototype optimization which develops Moroccan natural resources to ensure sustainable and continuous development. Moroccan clay was selected mineral as raw material and different formulations were used to optimize the limit conditions of the design of the experiments [22]. These formulations were then processed to develop the ceramic membrane support: powder preparation (wet grinding, drying, granulation, and humidification), preparation support (pressing and drying) and characterization.

In this work, a factorial experimental design 2^3 has been used to analyze the effect of thermal treatment parameter sintering temperature, the amount of starch and the granulometry (particle size distribution).

2. Experimental Methods

2.1 Experimental Procedure

The plastic pastes were prepared from natural Moroccan clay powder homogeneously. These powders will be mixed with organic additives and water. Plasticizer and binder are required to prepare a paste with rheological properties allowing the shaping by extrusion.

The mixture of clay and organic additives was obtained by the mixing of clay and starch (Corn starch RG03408, Cerestar) as the lead factor, with Methocel 4% w/w (The Dow Chemical Company), Amijel 4% w/w (Cplus 12072, Cerestar) and PEG 1500 0.3% w/w (Prolabo). The mixture was agitated 250 rpm, for 30 min in order to obtain a good homogeneity. The water (32% w/w of powders) and Zusoplast 0.24 % w/w (Zschimmer and Schwartz) were added too and pugging for 30 min.

The pastes were kept in a closed box for 2 days under high humidity to avoid premature drying and to ensure complete diffusion of the water and organic additives. Thereafter they were shaped by extrusion and calendared into a thin film that was segmented to format flat disk supports with a diameter of 4.9 cm. Later they were dried at temperature 40 °C during 24 h of the flat support after extrusion. Finally, the extruded pieces were sintered in the furnace. The process of ceramic preparation is described in Fig. 1.

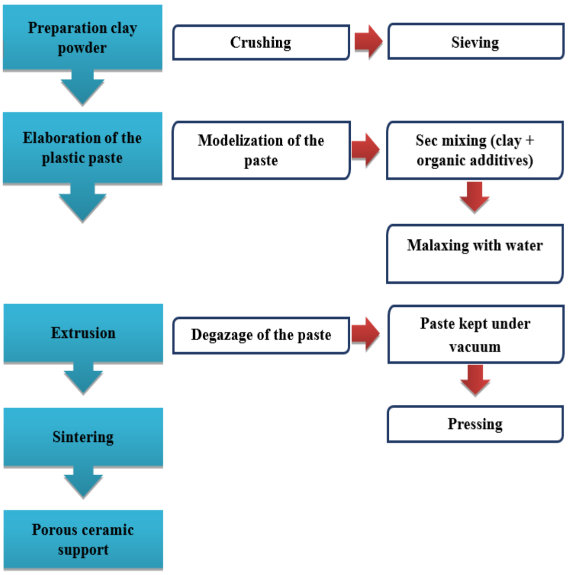

Fig. 1 Diagram of porous support elaboration by extrusion method [13]

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2.2 Experimental Design

Different experimental design methods exist. These include the method "one factor at a time". This consists of the successive study of each of the factors while leaving the others constant. However, this methodology neglects the possible existence of interactions between the factors, it is, therefore, to be prescribed [21]. The method used to carry out this work is that of the complete plans. Nevertheless, this last method is also to be prescribed when the number of controlled factors becomes important.

Using a complete factorial design (Fig. 2), we performed all the combinations of factor levels involved [23]. In the case where the k factors have 2 levels, we proceeded to the realization of 2^k treatments and the complete factorial plan is then called 2^k plane.

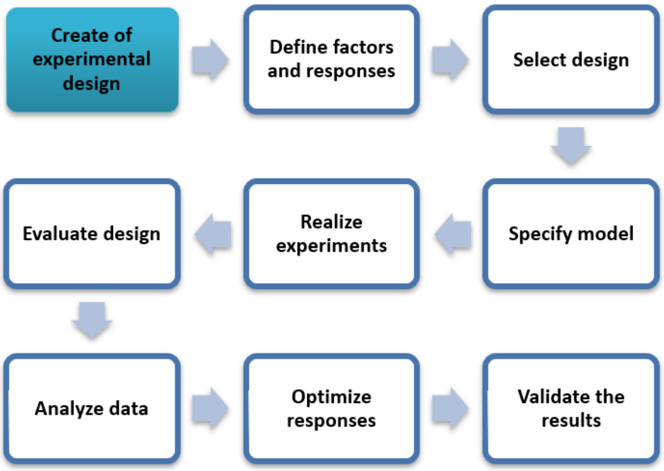

Fig. 2 Schematization of an experimental design [24]

The complete experimental design adopted in our study involves three factors, namely sintering temperature (X_1), the starch content (X_2) and granulometry (X_3), whose objective is to optimize the porosity (Y) considered as an answer this factors which will be evaluated at two levels (a lower level marked -1 and a higher level marked +1) showed in Table 1.

Table 1 Factorial experiment design 2^3

Level	Granulometry (µm)	Starch content (%)	Sintering temperature (°C)
	X_1	X_2	X_3
-1	100	6	600
+1	125	10	850

3. Results and Discussion

The construction of a complete plan, it suffices to vary the first factor on all its levels while the second remains at one, and to copy the block obtained on all the levels of the second factor. This gives a second block that is copied on all levels of the third factor and so on. Table 2 shows the experimental analyzes in the order they were executed.

Table 2 Results of experiments

Exp.	Factors			Response
	X_1	X_2	X_3	Y
	Granulometry (µm)	Starch content (%)	Sintering temp. (°C)	Porosity (%)
1	100	10	600	26.1
2	100	10	850	32.6
3	125	10	600	29.0
4	100	5	600	20.8
5	125	10	850	40.9
6	100	5	850	26.3
7	125	5	850	39.4
8	125	5	600	28.0

There are three factors and each taking two levels, and since it is thought that the first-degree model with interactions is sufficient to explain the results, it is advisable to choose a 2-factor 3 complete factorial design scheme.

In Table 3, a P-value < 0.05 represents a significant effect of the corresponding factors on the porosity. All the significant terms have positive effect exhibiting that increasing level of these factors results in higher porosity. This P-value > 0.05 represents non significant effect of the corresponding factors on the porosity.

Table 3 Analysis of variance for response (porosity)

Source	Sum of Squares	Degree of freedom	Mean Square	F-Ratio	P-Value
X_1	124.031	1	124.031	3969.00	0.0101
X_2	24.8513	1	24.8513	795.24	0.0226
X_3	155.761	1	155.761	4984.36	0.0090
X_1 X_2	10.3513	1	10.,3513	331.24	0.0349
X_1 X_3	15.9612	1	15.9612	510.76	0.0282
X_2 X_3	0.28125	1	0.28125	9.00	0.2048
Total error	0.03125	1	0.03125	_	_
Total (corr.)	331.269	7	_	_	_

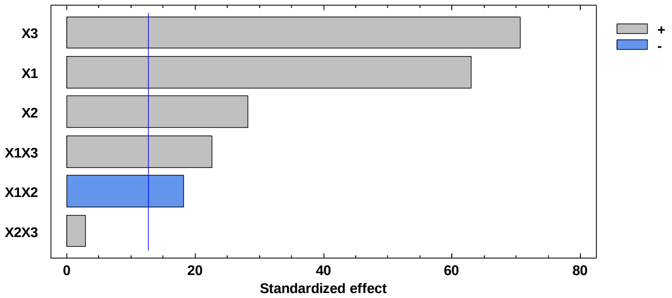

Fig. 3 Pareto chart for porosity

Analysis of the variance of the operating parameters studied that are translated by the Pareto diagram (Fig. 3) reveals that the simple effect of sintering temperature, granulometry, starch content, and the sintering temperature - granulometry conjugate effect have a significant effect positive on performance. The simple effect of the granulometry - starch content conjugate has a significant negative effect on the performance. On the other hand, the effects of the sintering temperature - starch content conjugate has no significance.

3.1 The Equation of the Model

In the example below, X_1, X_2 and X_3 will be considered as the three main factors of the studied system. The system equation becomes,

$$\text{Porosity (\%)} = 7.09 - 0.0674X_1 + 4.8X_2 - 0.0664X_3 - 0.0364X_1X_2 + 0.000904X_1X_3$$

where, 7.09 is the independent value (term), which is equal to the average of all the results of a single answer.

Table 4 Analysis of experimental results

Model	Porosity
Model d.f.	5
P-value	0.0024
Errordergeefreedomdf	2
Std. error	0.395285
R-squared	99.91
Adj. R-squared	99.67

The value of the coefficient of determination R-squared is equal to 99.91%, which means a good adjustment of the proposed model.

3.2 Optimization of the Responses

Table 5 shows that the theoretical values of the parameters are in the range of experimental values and the theoretical optimum corresponds to the experimental optimum.

Table 5 Factor settings at optimum

Factor	Setting
Granulometry	125.0 µm
Starch content	10.0%
Sintering temperature	850.0 °C

Table 6 Response values at optimum

Response	Optimum value	Lower 95.0% Limit	Upper 95.0% Limit	Desirability
Porosity (%)	40.775	39.3021	42.2479	0.993857

The graph of response surface in Fig. 4, built after determining the most influential factors, makes it possible to define all the combinations of operating conditions that make it possible to obtain the target value of the

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response. They are therefore very practical to delineate a desirable or optimal work area.

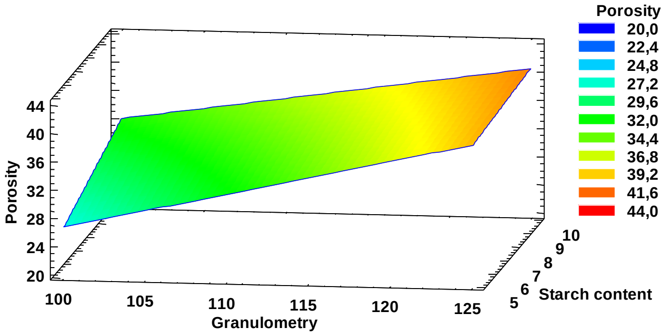

Fig. 4 Response surface at sintering temperaute 850 °C

4. Conclusion

The objective of this work was to optimize the operating conditions of the elaboration of ceramic membranes namely sintering temperature, granulometry and starch content. The experimental results showed that the powder of clay has to be crushed and sieved at 125 µm, the sintering temperature support could reach 850 °C and the starch content addition as 10%. The ceramic support presents the porosity of 41%. The study by plan of experiments made it possible to reach optimal conditions of the elaboration of ceramic membranes and to propose a model that describes an optimum value of porosity is 40.775%. It has also shown that there is a strong interaction between granulometry - starch content and sintering temperature - granulometry.

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