Desalination and Water Treatment
www.deswater.com

doi: 10.5004/dwt.2022.28493

257 (2022) 96–102
May

Processing and characterization of titania ultrafiltration ceramic membrane: response surface methodology optimization

M. Ait Baih[a], H. Saffaj[b], A. Adam[a], A. Bakka[a], H. Zidouh[a], R. Mamouni[a], N. Saffaj[a,*]

[a]*Laboratory of Biotechnology, Materials and Environment, Ibn Zohr University, Agadir, Morocco, emails: saffaj@gmail.com (N. Saffaj), liraqimoh@gmail.com (M. Ait Baih), adam.abdeljalil@gmail.com (A. Adam), abdelhamid.bakka@gmail.com (A. Bakka), r.mamouni@uiz.ac.ma (R. Mamouni)*

[b]*Laboratory of Algebra Analysis and Applications, Department of Mathematics and Computer, Faculty of Sciences Ben M'Sik, University Hassan II, Casablanca, Morocco, email: saffajhamza@gmail.com (H. Saffaj)*

Received 23 January 2022; Accepted 16 April 2022

ABSTRACT

In the present study, the effect of titania sol concentration, hydroxy ethyl cellulose (HEC), and sintering temperature of Titanium ultrafiltration membrane elaboration were evaluated using Box–Behnken and investigated by pores diameter. The titania ultrafiltration membrane has been prepared by the sol gel technique. A deflocculated suspension of titania was obtained by mixing 20 mL of titania sol (0.5–1 mol/L) and 10 g of HEC (2%–5% w/w aqueous solution) as binder. The titania layer was deposited on the inner surface of clay support by slip casting with a contact time (15 min). After drying at room temperature, the TiO_2 membrane was sintered (300°C–600°C) for 2 h. The optimal factors to elaborate the TiO_2 membrane with pores diameter of 5 nm by using Box–Behnken design include a sintering temperature of 600°C, TiO_2 sol concentration of 0.5 mol/L and 2% of HEC. The interaction between the factors were relatively less important.

Keywords: Titanium membrane; Ultrafiltration; Optimization; Response surface methodology

1. Introduction

Membrane technology find now a wide application in the desalination processes, in food industry, in the medical industry, and in separation effluents, among other process [1]. This increase of the application of membrane in industry can be attributed to the various advantage such as simple operation, good product quality, no chemical alteration of constituents, easy scale up and better stability [2]. Ceramics membrane have many advantages comparing to polymeric ones in terms of resistance to aggressive conditions, maintenance, and life span [3,4].

The choice of materials is very important for the membrane preparation as it defines various properties like pore size, porosity, thickness and strength [2,3]. The chemical and physical characteristics of these materials are responsible for these membrane properties. Ceramics membranes are prepared from different material, including spinel, silica, titania, alumina, zirconia. Ceramics membranes are generally prepared by using the following steps: (i) powder or sol preparation; (ii) shaping; (iii) temperature treatment of deposited layer [3,4].

The sol gel method is the most commonly used for the ceramic ultrafiltration and nanofiltration membrane. It was believed us one of the most important techniques in fabrication of ceramic membrane. The advantage of the sol–gel techniques is that the pores size of the membrane can be desirably controlled, especially for small pores [4–6].

* Corresponding author.

Presented at the Second International Symposium on Nanomaterials and Membrane Science for Water, Energy and Environment (SNMS-2021), June 1–2, 2022, Tangier, Morocco

The sintering is usually the final step in ceramic membrane production. It consists of three important steps, the initial stage, the intermediate stage and the final stage. The membrane precursor and the particles have different features and movement at each stage, including full densification, grain coarsening and closing of pores. The temperature regulation depends upon the membrane precursor and especially the material type [7].

However, many trials are required for evaluating the effect of these factors on the ceramic ultrafiltration membrane elaboration. The optimization of experimental conditions to elaborate the ultrafiltration membrane cannot successfully done by using factor by factor optimization only. For this reason, the application of the experimental design methodologies can result in improved conditions of the elaboration with lesser number of experiments. In addition, response surface methodology (RSM) is a powerful and widely used Mathematical method suitable for modeling and optimizing chemicals reactions and or industrial process. The objective of the optimization is to determine the optimum value of variable factors from the model obtained via experimental design and analysis [8–14].

In this work, an asymmetric ceramic ultrafiltration multilayer membrane was elaborate. The first part consists to elaborate a new membrane support using natural abundant clay from south of Morocco (Laayoune, Sahara); The formation of microporous interlayer from zirconia and formation of a thin ultrafiltration separation top layer from TiO_2 sol will be described. The estimation of the best elaboration conditions of titanium ultrafiltration membrane and evaluate the effect of TiO_2 sol concentration, HEC content, and sintering temperature will be studied by using BBD.

2. Materials and methods

2.1. Preparation of macroporous support clay and ZrO_2 microfiltration membrane

The elaboration of ceramic support and zirconia interlayer has been reported in our previous work. For ceramic support, the grain size of Moroccan Sahara clay used to prepare the paste is 30 µm. The sample was performed by extrusion of the mixture of clay and organic additives. The extruded pieces were dried at ambient temperature for 24 h and transferred in an oven at 50°C for 24 h. The Optimized response to elaborate the ceramic support of porosity 38.79% and Mechanical strength of 12 MPa include a starch of 4% and sintering temperature of 1,014.36°C [15,16].

For zirconia interlayer, the powder suspension technique was used to prepare the zirconia microfiltration membrane layer. A deflocculated suspension of zirconia was obtained by mixing zirconia powder, PVA (12% w/w aqueous solution) as binder and water (with dispersant 0.2% w/w) like dispersing agent. The zirconia membrane layer was deposited on the inner surface of Moroccan clay support by slip casting (Fig. 1). After drying at room temperature, the ZrO_2 membrane was sintered for 2 h. The optimal factors to elaborate the microfiltration membrane of a pores diameter of 0.24 µm and thickness of 24 µm include a sintering temperature of 1,000°C, zirconia content of 5%, polyvinyl alcohol content of 30% and contact time of 2 min [17,18].

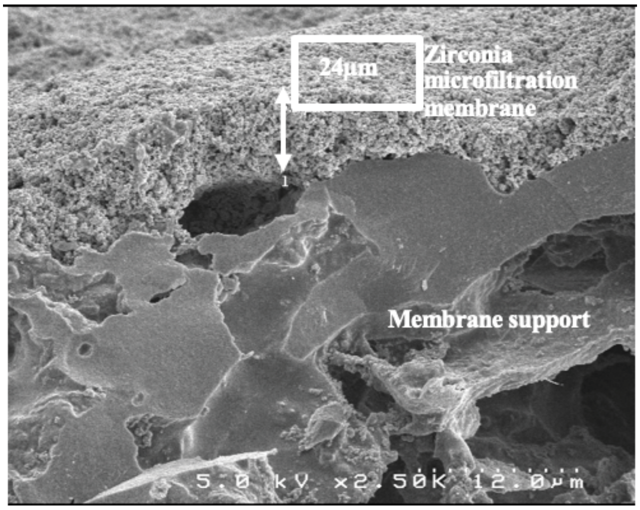

Fig. 1. Scanning electron micrographs of membrane support and zirconia microfiltration layer.

2.2. Preparation of TiO_2 membrane ultrafiltration

The ultrafiltration layer was prepared using sol gel route:

TiO_2 sol is prepared from the hydrolysis of $Ti(iOPr)_4$ by 111.6 g of water. After stirring for 1 h, 65.2 g of a nitric acid solution is added. The solution is stirred and heated at 60°C for 24 h to obtain the peptization. The sol is sieved at 125 µm and diluted to 250 mL.

The sol TiO_2 mixed with hydroxyethyl cellulose (aqueous solution) as binder.

Ultrafiltration layer preparation: the former sol was deposited in the inner part of the clay tubular support by slip casting. The coating time was 2 h. The coated support was then dried for 24 h at room temperature, then sintered for 2 h, after bonding at 250°C for 2 h.

2.3. Methods of characterization

Different techniques were used to investigate the properties of titania ultrafiltration ceramic membrane. The pore size distribution of the ultrafiltration layer was determined by nitrogen adsorption/desorption (BET method-Micrometrics Asan 2010). The morphology of titania membrane was examined by scanning electron microscopy (Hitachi, S-4500).

2.4. Box–Behnken design

The RSM using Box–Behnken (BBD) design was performed to obtain the details regarding the significant effects and to study the possible interactions between the shortlisted significant factors with a positive effect on the elaboration of ceramic support membrane and to determine the optimal value of every factor [9]. A three-level three factorial Box–Behnken experimental design was applied. The factor levels were coded as –1 (low), 0 (central point) and 1 (high) [19–21]

In the optimization process, an empirical model was developed to correlate the response of the Elaboration of ultrafiltration membrane process and is based on second-order quadratic model given in Eqs. (1) and (2):

$$Y = b_0 + \sum_{i=1}^{k} b_i X_i + \sum_{i=1}^{K} b_{ii} X_i^2 + \sum_{i=1}^{k} \sum_{j=1}^{K} b_{ij} X_i X_j + \varepsilon \quad (1)$$

$$Y = b_0 + b_1 X_1 + b_2 X_2 + b_{12} X_1 X_2 + b_{11} X_1^2 + b_{22} X_2^2 \quad (2)$$

where Y is the response, b_0 is the constant; b_i is the linear coefficient, b_{ii} represents the quadratic coefficient, b_{ij} is the interaction coefficient, X_i is the coded variable level and i or j is the number of the independent variables.

The results of the experiments design were analyzed using statistical software to evaluate the effect as well as the statistical parameters, the statistical plots. BBD having 15 experiments for titania ultrafiltration ceramic membrane elaboration was studied and a matrix was established according to their high and low levels, represented by +1 and –1 respectively. The coded value of variables with the response (Pores diameter) are illustrated in Table 1. The main effect of the microfiltration ceramic membrane elaboration was identified based on the P-value with 95% of confidence level.

3. Results and discussion

3.1. Effects of the processing factors on the physical properties of TiO_2 ultrafiltration membrane

The measured value of the technological properties (pore diameter) of the sintered test specimens as a function of titania sol concentration, hydroxy ethyl cellulose content (HEC), and sintering temperature are given in Table 2.

A regression equation was obtained for each titania membrane using BBD at a 5% level of significance. Analysis of variance (ANOVA) and plots of observed values vs. predicted one were used to confirm the validity and precision of model.

The results equations of pore diameter (nm) (Y_1) are reported after:

$$Y_1 = 7.750 + 0.235A - 2.156B + 0.358C - 0.265AA + 0.452AB + 0.047CC - 0.063AC - 0.032AC - 0.250BC \quad (3)$$

Table 1
Factor levels tested using Box–Behnken design

Parameters	Experimental value		
	Low (–1)	Central (0)	High (+1)
Titania sol concentration (mol/L)	0.5	0.75	10
HEC content (%)	2	3.5	5
Sintering temperature (ST) (°C)	300	450	600

Table 2
Experiment design matrix and measured values of the considered responses using Box–Behnken

Run	Coded values			Experimental values			Experimental response	Predicted responses
	A	B	C	$[TiO_2]$ sol (mol/L) (A)	Sintering temperature (°C) (B)	HEC content (%) (C)	Y_1 (nm) pore diameter	Y_1 (nm) pore diameter
1	1	–1	0	1	300	3.5	10.5	10.391
2	0	1	–1	0.75	600	2	6	5.985
3	0	0	0	0.75	450	3.5	7.75	7.750
4	0	–1	–1	0.75	300	2	9.5	9.797
5	–1	0	–1	0.5	450	2	7	6.906
6	1	0	1	1	450	5	8	8.093
7	1	1	0	1	600	3.5	5.75	5.953
8	–1	–1	0	0.5	300	3.5	10	9.796
9	–1	0	1	0.5	450	5	7.5	7.688
10	0	0	0	0.75	450	3.5	7.75	7.750
11	–1	1	0	0.5	600	3.5	5.5	5.608
12	0	0	0	0.75	450	3.5	7.75	7.750
13	0	1	1	0.75	600	5	6.5	6.202
14	0	–1	1	0.75	300	5	11	11.015
15	1	0	–1	1	450	2	7.63	7.441

Y_1: pore diameter (nm).

Based on the experimental data, regression models were fitted for Y_1 as shown in Eq. (3). The adequacy of the initial model was tested via parity plot for observed vs. predicted values, as demonstrated in Fig. 2. As seen in Fig. 2, the high values of the correlation coefficient ($R^2 = 0.99$) for pore diameter demonstrates a good correlation between the observed and the predicted responses by initial models.

Analysis of variance (ANOVA) was employed to investigate the adequacy and significance of the model. The effect of a factor is defined as the change in response produced by a change in the level of the factor. This is frequently called the main effect because it refers to the primary factors of interest in the experiments. The ANOVA results showed that the equation adequately represented the actual relationship between each response and the significant variables. The F-value implies that the models are significant and value of Prob. > F less than 0.05 indicate that the models terms are significant. Especially larger F-value with the associated P-value (smaller than 0.05, confidence intervals) means that the experimental system can be modelled effectively with less error [22].

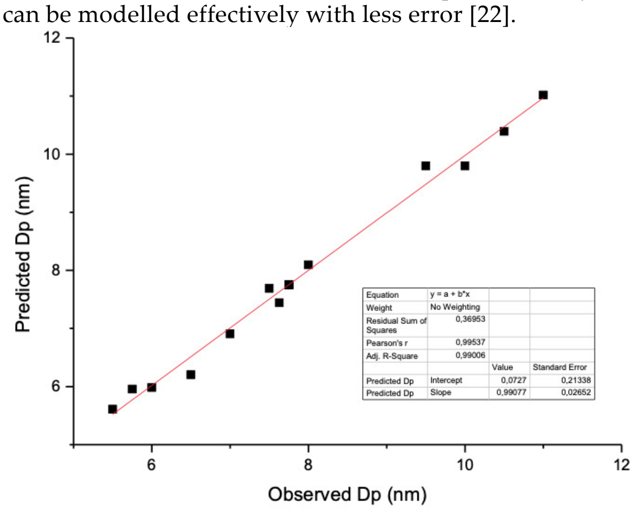

Fig. 2. Parity plot of predicted vs. observed responses for pore diameter (D_p).

According to the ANOVA results (Table 3), the values of F_{cal} (59.63 for pore diameter (Y_1) was higher and P-value s were lower than 0.05 which shows the significance and suitability of BBD model. Moreover, the normal probability of the residuals almost indicated no departures from the normality (Fig. 3). Among all the terms, the linear effect of sintering temperature and HEC content were to have a predominant effect owing to the low p-value (<0.05) for the factors. However, the quadratic and the 2-way interaction were not statistically significant on the elaboration of ultrafiltration membrane.

As shown in the Table 4, High coefficient of determination (R^2: 0.990) and adjusted coefficient of determination (R^2_{adj}: 0.974) indicate the good agreement of experimental response values with model predicted values. The predicted R-squared (R^2_{pred}: 0.852) was also in reasonable agreement with adjusted R-squared and showed a good prediction of model.

Factors that influence the pore diameter (nm) was evaluated by using factorials plots: the main effect [23,24].

Taking into consideration the value of linear coefficient shown in the above equations, the weight effect of the considered parameters followed the order: $B > C > A$ for the pore diameter

The main effect which are helpful in visualizing which factors most affects the response of each parameters represent deviations of the average between high and low levels of each one of them as shown in Figs. 6 and 7.

Each level of factor effects the response differently; if the slop is close zero, then the magnitude of the main effects will be small. As the results show, the sintering temperature appears to have a great effect on the response on pore diameter as indicated by steeply slope due the great surface followed by HEC and titania sol concentration.

The increase of the sintering temperature causes an enlargement of the pore diameter but at high value of sintering the pores tend to decrease [26,27]. Such effects are related to the strong influence of temperature on the melt formation and consequently on the sintering process [3,7]. However, the HEC affects the properties of the ultrafiltration layers, the increase of HEC content provokes

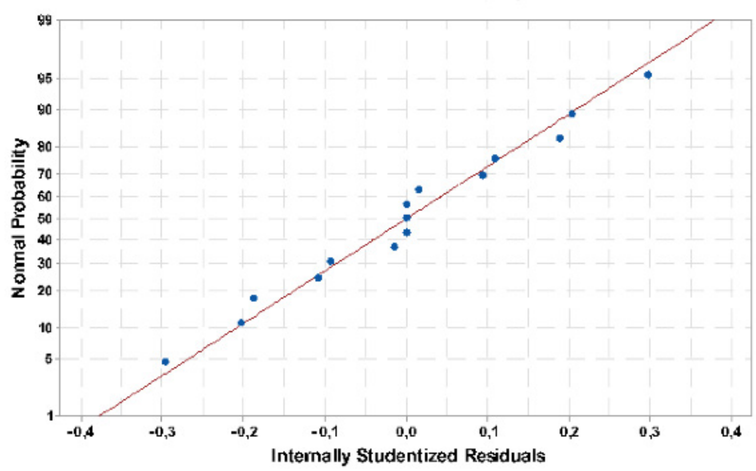

Fig. 3. Normal probability of the residuals of pore diameter.

Table 3
Analysis of variance (ANOVA) for response surface for the prediction of pore diameter

Source	Sum of square (SS)	Df	Mean squares (MSS)	F-value	P-value probability $(P) > F$
Model	40.0308	9	4.4479	59.63	0.000
Linearity	38.667	3	12.8889	172.79	0.000
$[TiO_2]$ sol (mol/L)-A	0.4418	1	0.4418	5.92	0.059
Sintering temperature (°C)-B	37.1953	1	37.1953	498.63	0.000
HEC content (%)-C	1.0296	1	1.0296	13.80	0.014
Square	1.0942	3	0.3647	4.89	0.060
$A \times A$	0.2593	1	0.2593	3.48	0.121
$B \times B$	0.7560	1	0.7560	10.14	0.024
$C \times C$	0.0083	1	0.0083	0.11	0.752
2-way interaction	0.2683	3	0.0899	1.21	0.398
$A \times B$	0.0156	1	0.0156	0.21	0.666
$A \times C$	0.0042	1	0.0042	0.06	0.821
$B \times C$	0.2500	1	0.2500	3.35	0.127
Error	0.3730	5	0.0746		
Lack-of-fit	0.3730	3	0.1243		
Pure error	0.000000	2	0.00000		
Total	40.4038	14			

Table 4
Values of correlation coefficient (R^2) related to the adopted models

Response	R^2 coefficient of determination	R^2 adjusted	R^2 predicted
Y_1: pore diameter (nm)	0.990	0.974	0.852

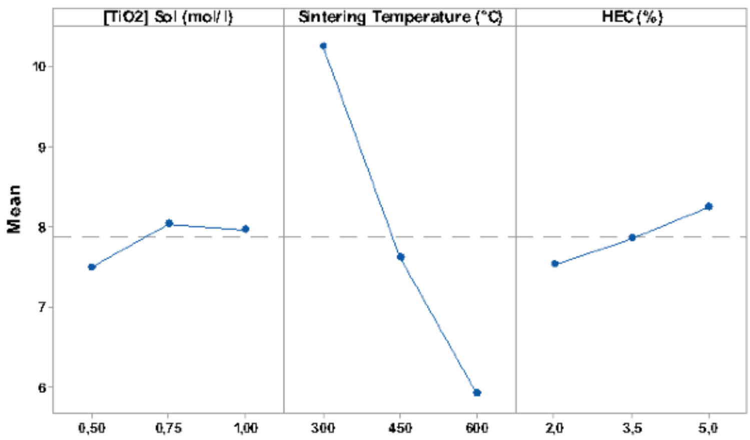

Fig. 4. Main effects for pore diameter.

an increase of pore diameter. This effect is due of pore forming during burning out around 250°C–300°C of HEC [28,29]. The HEC is used as binder in casting process to provide sufficient strength to the body so that the green bodies can be modeled and retained in the desired shape without breaking or damage, before and during sintering process. Also, they cause to achieve a higher thickness in function of time contact with desired support [1–4].

Figs. 5 and 6 show the graphical interaction indication the combined effect of (sintering temperature and HEC content) and (sintering temperature and $[TiO_2]$). From this figure it is clear that the increase in sintering temperature

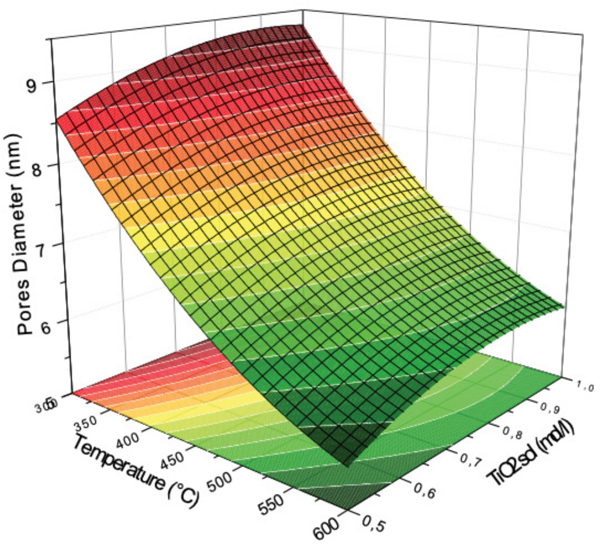

Fig. 5. Response surface plot of pores diameter of ultrafiltration membrane vs. temperature and TiO_2 sol.

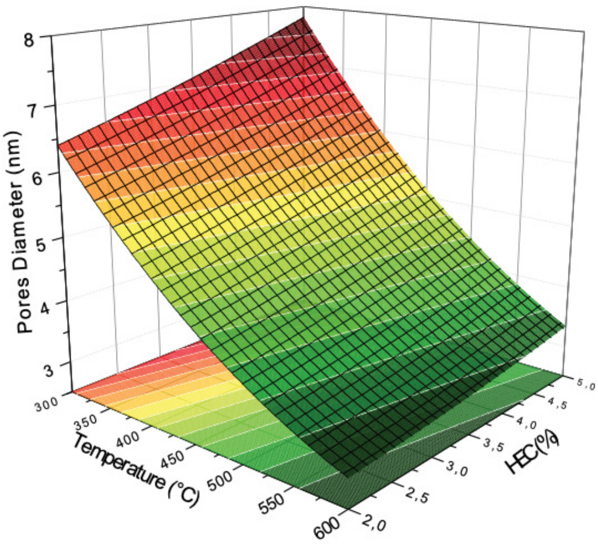

Fig. 6. Response surface plot of pores diameter of ultrafiltration membrane vs. temperature and HEC.

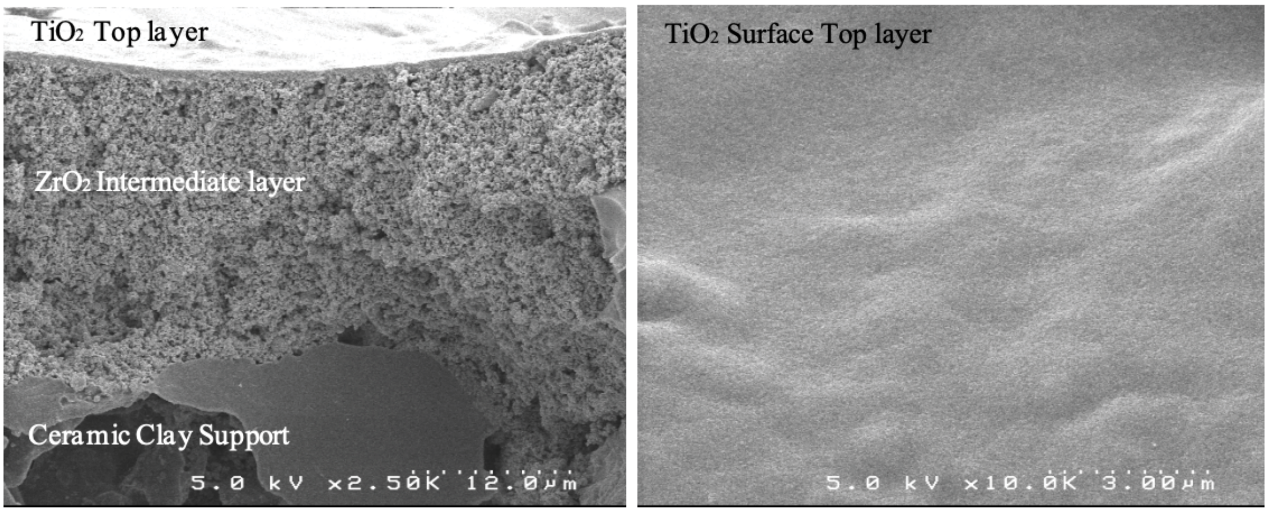

Fig. 7. Scanning electron micrographs of TiO_2 membrane.

decrease dramatically the pores diameter. HEC as a pore former improve the pores diameter specially at low sintering temperature 300°C [28–30].

3.2. Response optimization

Performance of ceramic membrane is governed by its morphology which depends on the physical, chemical, and thermal properties. Porous texture in the ceramic is controlled by pore-former that, under sintering conditions release carbon dioxide gas. The path taken by the released CO_2 gas thereby creates the porous texture of ceramic support membrane and contributes to membrane porosity. Response optimization for the current model was performed with the target as minimum of diameter of pores (high retention). The optimized predicting response to elaborate the TiO_2 ultrafiltration membrane with pores diameter of 5 nm include a TiO_2 sol concentration of 0.5 mol/L, sintering temperature of 600°C and a HEC content of 2% (Fig. 7).

4. Conclusion

The application of Box–Behnken design is an effective tool to evaluate the important significant factors influencing the elaboration of TiO_2 ultrafiltration ceramic membrane. 3 factors namely the sintering temperature, HEC content and titania sol concentration were found to exert a significant effect on the process of pores diameter. A detailed analysis of the results was conducted using a multilinear regression method based on the analysis of variance using Fisher test and the validation by the coefficient of determination R^2 were found to be statically significant and presented low variability. The optimized predicting response to elaborate the TiO_2 ultrafiltration membrane with pores diameter of 5 nm include a TiO_2 sol concentration of 0.5 mol/L, sintering temperature of 600°C and a HEC content of 2%.

References

[1] M.K. Purkait, R. Singh, Membrane Technology in Separation Science, Taylor and Francis Group, LLC, London, New York, 2018.

[2] D. Liang, J. Huang, H. Zhang, H. Fu, Y. Zhang, H. Chen, Influencing factors on the performance of tubular ceramic membrane supports prepared by extrusion, Ceram. Int., 47 (2021) 10464–10477.

[3] L. Kang, Ceramic Membranes for Separation and Reaction, John Wiley & Sons, Ltd., England, 2007.

[4] C. Das, S. Bose, Advanced Ceramic Membrane and Application, Taylor and Francis Group, LLC, London, New York, 2017.

[5] B. Yuzer, M. Guida, F. Ciner, B. Aktan, M. Iberia Aydin, S. Meric, H. Selcuk, A multifaceted aggregation and toxicity assessment study of sol–gel-based TiO_2 nanoparticles during textile wastewater treatment, Desal. Water Treat., 57 (2016) 4966–4973.

[6] A. Zanurin, N.A. Johari, J. Alias, H. Mas Ayu, N. Redzuan, S. Izman, Research progress of sol-gel ceramic coating: a review, Mater. Today:. Proc., 48 (2022) 1849–1854.

[7] M.N. Rahaman, Sintering of Ceramics, Taylor and Francis Group, LLC, London, New York, 2007.

[8] R.L. Plackett, J.P. Burman, The design of optimum multifactorial experiments, Biometrika, 33 (1946) 305–325.

[9] J. Goupy, L. Creighton, Introduction to the Experimental Design, Dunod, Paris, 2006.

[10] R. Venkataraghavan, R. Thiruchelvi, D. Sharmila, Statistical optimization of textile dye effluent adsorption by *Gracilaria edulis* using Plackett-Burman design and response surface methodology, Heliyon, 6 (2020) e05219, doi: 10.1016/j.heliyon.2020.e05219.

[11] Y. Achour, A. El Kassimi, I. Nadir, H. Yazid, A. Hafid, M. Khouili, M. El Himri, M.R. Laamari, M. El Haddad, Simultaneous removal of binary mixture of cationic dyes onto Bombax Buonopozense Bark: Plackett–Burman and central composite design, Biointerface Res. Appl. Chem., 12 (2022) 326–338.

[12] A. Azari, M. Yeganeh, M. Gholami, M. Salari, The superior adsorption capacity of 2,4-dinitrophenol under ultrasound-assisted magnetic adsorption system: modeling and process optimization by central composite design, J. Hazard. Mater., 418 (2021) 126348, doi: 10.1016/j.jhazmat.2021.126348.

[13] A. Azari, M.H. Mahmoudian, M.H. Niari, I. Eş, E. Dehganifard, A. Kiani, A. Javid, H. Azari, Y. Fakhri, A.M. Khaneghah, Rapid and efficient ultrasonic assisted adsorption of diethyl phthalate onto $Fe^{II}Fe_2^{III}O_4$@GO: ANN-GA and RSM-DF modeling, isotherm, kinetic and mechanism study, Microchem. J., 150 (2019) 104144, doi: 10.1016/j.microc.2019.104144.

[14] S.Y. Hashemi, A. Azari, M. Raeesi, K. Yaghmaeian, Application of response surface methodology (RSM) in optimization of fluoride removal by magnetic chitosan/graphene oxide composite: kinetics and isotherm study, Int. J. Environ. Anal. Chem., (2021), doi: 10.1080/03067319.2021.1938021.

[15] M. Ait Baih, H. Saffaj, K. Aziz, A. Bakka, N. El baraka, H. Zidouh, R. Mamouni, N. Saffaj, Statistical optimization of the elaboration of ceramic membrane support using Plackett–Burman and response surface methodology, Mater. Today:. Proc., 52 (2022) 128–136.

[16] M. Ait Baih, N. Saffaj, A. Bakka, R. Mamouni, N. El baraka, H. Zidouh, N. El Qacimi, Clay ceramic support membrane optimization using factorial design approach, J. Appl. Membr. Sci. Technol., 25 (2021) 1–15.

[17] M. Ait Baih, H. Saffaj, A. Adam, A. Bakka, N. El baraka, H. Zidouh, R. Mamouni, N. Saffaj, Application of the experimental design for the optimization of microfiltration membrane, J. Appl. Membr. Sci. Technol., 26 (2022) 95–106.

[18] A. Ait Taleb, N. El Baraka, N. Saffaj, A. Laknifli, R. Mamouni, A. Fatni, A. El Hammadi, N. El Qacimi, New tubular ceramic membranes from natural Moroccan clay for microfiltration application, E3S Web Conf., 37 (2018) 01011, doi: 10.1051/e3sconf/20183701011.

[19] E.S. Akpan, M. Dauda, L.S. Kuburi, D.O. Obada, Box–Behnken experimental design for the process optimization of catfish bones derived hydroxyapatite: a pedagogical approach, Mater. Chem. Phys., 271 (2021) 124916, doi: 10.1016/j.matchemphys.2021.124916.

[20] G.E. Box, D.W. Behnken, Some new three level designs for the study of quantitative variables, Technometrics, 2 (1960) 455–475.

[21] J.N. Miller, J.C. Miller, Statistics and Chemometrics for Analytical Chemistry, Pearson Education Limited, England, 2010.

[22] E. del Castillo, Process Optimization: A Statistical Approach, Spring, Pennsylvania, USA, 2007.

[23] W. Misrar, M. Loutou, L. Saadi, M. Mansori, M. Waqif, C. Favotto, Cordierite containing ceramic membranes from smectetic clay using natural organic wastes as pore-forming agents, J. Asian Ceram. Soc., 5 (2017) 199–208.

[24] M. Loutou, M. Hajjaji, M. Mansori, C. Favotto, R. Hakkou, Heated blends of clay and phosphate sludge: microstructure and physical properties, J. Asian Ceram. Soc., 4 (2016) 11–18.

[25] M. Loutou, M. Hajjaji, M. Mansori, C. Favotto, R. Hakkou, Heated blends of phosphate waste: microstructure characterization, effects of processing factors and use as a phosphorus source for alfalfa growth, J. Environ. Manage., 177 (2016) 169–176.

[26] J.D. de Oliveira Henriques, M.W. Pedrassani, W. Klitzke, A.B. Mariano, J.V.C. Vargas, R.B. Vieira, Thermal treatment of clay-based ceramic membranes for microfiltration of *Acutodesmus obliquus*, Appl. Clay Sci., 150 (2017) 217–224.

[27] N. El Qacimi, N. El Baraka, N. Saffaj, R. Mamouni, A. Laknifli, S. Alami Younssi, A. Faouzi, H. Zidouh, Preparation and characterization of flat membrane support based on Sahara Moroccan clay: application to the filtration of textile effluents, Desal. Water Treat., 143 (2019) 111–117.

[28] M.-M. Lorente-Ayza, M.J. Orts, V. Pérez-Herranz, S. Mestre, Role of starch characteristics in the properties of low-cost ceramic membranes, J. Eur. Ceram. Sci., 35 (2015) 2333–2341.

[29] M.-M. Lorente-Ayza, E. Sanchez, V. Sanz, S. Mestre, Influence of starch content on the properties of low-cost microfiltration ceramic membranes, Ceram. Int., 41 (2015) 13064–13073.

[30] M. Messaoudi, N. Tijani, S. Baya, A. Lahnafi, H. Ouallal, H. Moussout, L. Messaoudi, Characterization of ceramic pieces shaped from clay intended for the development of filtration membranes, S. Afr. J. Chem. Eng., 37 (2021) 1–11.