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Research on performance optimization of Loess-based ceramic membrane supports with Bauxite-dolomite-talc as additives

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Abstract

Inorganic ceramic membranes have attracted much attention due to their advantages of long life, acid and alkali corrosion resistance, and easy cleaning, but they have not been widely used due to the high cost. In order to obtain a ceramic membrane with low cost and good performance, in this paper, loess-based ceramic membrane support was prepared by using loess as raw material, bauxite as aluminum source, dolomite as pore-forming agent and talc as plasticizer. Response surface optimization was used to determine the optimal addition parameters of ingredients. In addition, the factors affecting the performance of the supports were analyzed in terms of material composition, microscopic morphology, and porosity. The results show that the addition of talc conducive to avoid the negative impact of the pore-forming effect of dolomite on the mechanical strength of the support, and the optimum proportioning parameters are 48.85 wt% of bauxite, 9.93 wt% of dolomite and 8 wt% of talc. Under this condition, the bending strength was 42.24 MPa, the pure water flux was 8323.73 l m⁻² h⁻¹ MPa⁻¹, and the average pore throat radius was 5.15 μm. The performance of the best sample reaches the application performance of porous ceramic supports, which provides a theoretical basis for the wide application of low-cost ceramic membrane supports.

1. Introduction

The ceramic separation membranes belong to the solid membrane material in the inorganic membrane separation technology. The achievement of separation, purification, impurity removal, and sterilization is realized according to the screening theory [1, 2]. Ceramic membranes offer several better beneficial options than polymeric membranes including good combinations of thermal, mechanical and chemical stability, so that the research and development of inorganic membranes have been developed rapidly [3, 4]. They have played a significant role in environmental protection [5, 6], natural resources [7, 8], medical drugs [9], and other fields. In addition, data from the United States BCC shows that ceramic membranes have been used in more than half of the inorganic membranes and are increasing at a rate of 36% each year.

The support is the main component of the ceramic membrane, and its performance has a great impact on the overall performance of the ceramic membrane product. However, the preparation cost of traditional supports is relatively high. Commercial support materials are mainly made of materials such as Al₂O₃ and SiC. The sintering temperature of these two materials with the sintering aid still needs to reach above 1300 °C, so that its energy consumption is huge [10, 11]. In order to reduce the cost and enrich the types of supports, many scholars are committed to finding new raw materials, such as cheap industrial-grade cordierite powder, diatomite, clay, and fly ash [12, 13]. After testing, it was found that the main components of loess are SiO₂ and Al₂O₃, and it also contains good fluxes (such as Na₂O, K₂O and other alkali metal oxides). During the sintering process, these fluxes are easy to form a solid solution and generate a liquid phase, promote particle rearrangement, make the support sintered and compact and effectively reduce the sintering temperature. In recent years, our research
Table 1. Chemical composition of loess (wt%).

| Materials | SiO₂  | Al₂O₃  | CaO  | Fe₂O₃  | Na₂O  | K₂O  | MgO  | TiO₂  | Other |
|-----------|-------|--------|------|--------|-------|------|------|-------|-------|
| Loess     | 56.70 | 15.60  | 12.20| 6.86   | 1.19  | 3.54 | 2.37 | 0.92  | 0.62  |

group has been devoted to the cost-effectiveness investigation of loess materials as porous support materials, and successfully prepared loess-based ceramic membrane supports. However, the comprehensive performance of the ceramic membrane prepared by the previous preparation method needs to be further improved. Therefore, we adopt the corresponding method to optimize the preparation process of the previous stage.

Nowadays, many researches have focused on the effects of a single additive on the properties of supports, Cheng et al [14] prepared a ceramic membrane support using fly ash as the basic raw material and talc powder as the additive. Liu et al [15] studied the effect of dolomite addition on the performance of the supports, and found that adding dolomite helps to increase the porosity of the supports, but it has an inhibitory effect on the sintering behaviour. Fan et al [16] enhanced the bending strength of fly ash-based ceramic supports by the addition of bauxite, and found that when the sintering temperature and bauxite content were controlled at 1300 °C and 40 wt%, respectively, the obtained membrane supports exhibited a high pure water permeability of approximately 5.36 m²·m⁻²·h⁻¹·MPa⁻¹ and a high bending strength of about 69.6 MPa. However, the interaction mode of various factor are extremely complicated in a multi-component system. Therefore, when optimizing the preparation process of the support, it is necessary to consider that the interaction of various factors to improve the comprehensive performance of the support. Response surface methodology (RSM) is an optimization method that combines experimental design and mathematical modelling to find out the quantitative rule between the experimental indexes and factors, and to find out the best combination of each factor level. RSM is widely used in the optimization of biotechnology [17], food processing technology [18] and material processing technology [19]. Therefore, the application of the response surface optimization method to the preparation of the support can effectively improve the comprehensive performance of the support.

In this study, roll-forming method and solid particle sintering method were used to prepare the loess-based ceramic porous support. The interactions among bauxite, dolomite, and talc with respect to the pure water flux and bending strength of the support were analysed by response surface optimization. At the same time, a prediction model is established to determine the optimal preparation process. Moreover, the crystalline phase composition, micro-morphology and pore size distribution of the porous support prepared under the optimal process conditions were analysed.

2. Experimental

2.1. Materials

Lochuan Loess was taken from Heimuya, Lochuan Loess land in Lochuan County, Shaanxi Province. Bauxite was purchased from Gongyi Wanyuan Water Purification Material Trading Co., Ltd. Dolomite was purchased from Hebei Yanxi Mineral Processing Factory. Talc was purchased from Yongshun Mineral Processing Factory in Lingshou County, Shijiazhuang, Hebei.

Table 1 presents the chemical composition of raw materials used in experiments. It can be observed that the main components of loess are SiO₂ and Al₂O₃, which meets the support requirements of raw materials, and is also the theoretical basis for selecting aggregates in experiments. In addition, alkali metal oxides such as Na₂O, K₂O in the raw materials are good fluxes, which can form solid solution or liquid phase during sintering, which is beneficial to sintering.

The main component of bauxite is Al₂O₃, and it also contains a small amount of clay minerals, iron minerals, titanium minerals, etc. The alumina content is very high, so it can be used as an auxiliary aluminium source to combine with SiO₂ in the raw material to form mullite (Al₂O₃–SiO₂) during the sintering process to improve the physical and chemical stability of the support. Zhiwen et al prepared the ceramic membranes with abundant natural minerals including coarse bauxite and fine kaolin powders. They found that mullite was formed during sintering, and the growth of mullite whiskers has a positive impact on the improvement of porosity and mechanical strength of ceramic membranes [20].

The main components of natural dolomite are MgCO₃ and CaCO₃, which decompose easily at the temperature of 700 °C ~ 900 °C to produce a mixture of CaO and MgO, and escape carbon dioxide gas, thereby forming a pore-structure in the support. So, it is often used as a Pore former. Cheikh et al [21] prepared ceramic membrane supports with natural quartz sand as the main raw material, and it was found that the addition of dolomite can increase the porosity to a certain extent.

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Talc, a layered silicate mineral, is a hydrous magnesium silicate mineral with the theoretical chemical formula \( (\text{Mg}_6)\left[\text{Si}_8\right] \text{O}_{20}(\text{OH})_4 \). It is generally in block, leaf, fibrous or radial shape, with the characteristic of soft, easy to be cutted and smooth feel, which helps to support the forming of wet blanks and can be used as a pasted plasticizer to reduce the roughness of the outer surface of the support. At the same time, talc can react with \( \text{Al}_2\text{O}_3 \) and \( \text{SiO}_2 \) to form cordierite during the sintering process. This material has a small thermal expansion coefficient, which can improve the ability of ceramic materials to resist rapid cooling and heating, and can reduce the risk of cracking of the support due to temperature changes during the sintering process. So, it is widely used in the manufacture of ceramics, glass, etc. Qi et al.\cite{22} used kaolin as the main raw material to prepare the ceramic membrane support and added talc to improve the plasticity of the mud. In addition, the addition of talc is also helpful to reduce the sintering temperature.

In addition, studies have shown\cite{23, 24} that talc is also a sintering aid. Adding talcum to the ceramic body can reduce the firing temperature, widen the firing temperature range, and make the sintering system easier to control.

Figure 1 presents the XRD diffraction patterns of bauxite, Luochuan loess, and dolomite. The phase composition of Bauxite includes corundum, sillimanite, cristobalite, hematite and so on. Corundum is the main phase, and its hardness is second only to diamond, which is conducive to improving the mechanical properties of the support. Phase composition of the loess from Luochuan include quartz, plagioclase and calcite. Quartz is the main phase and has stable physical and chemical properties. Dolomite crystalline phase is single. Studies have shown that\cite{25}, during the firing process, dolomite has the effects of increasing porosity, reducing the product firing temperature, and promoting the development of mullite crystalline phase.

2.2. Preparation of ceramic support

Loess-based ceramic porous supports were prepared by roll forming method and solid particle sintering method with loess as aggregate, bauxite as additional aluminum source, dolomite as pore former, talc as lubricant and sintering aid and A ceramic porous support was prepared as follows: every kind of material was accurately weighed according to the mass ratio of the experimental reagent.

After all the materials were mixed in a beaker, distilled water was added. The mixture was stirred mechanically for 40 min to mix well. Then the beaker was put into a water bath heater at temperature of 80 °C ~ 90 °C and continue stirred until the moisture content of slurry was about 15%. The mixture was wrapped tightly with plastic wrap and aged in a biochemical incubator at 30 °C for 48 h. Then, 25 g of the aged mud was weighed with an electronic balance and wrapped on the surface of the wooden rod with a diameter of 4 mm. Then, it was rolled on a smooth flat plate to make it evenly attached to the wooden rod and form a single channel tube with an outer diameter of about 7 mm and a length of about 17 cm. The wet billet was dried in a 30 °C environment for 48 h, and then placed in a muffle furnace for sintering under certain sintering procedures in an air atmosphere. Figure 2 shows the process flow to prepare a support.

2.3. Experimental design

The influences of three independent variables, bauxite(%) , dolomite(%) and talcum(%) on the properties of the support were investigated. A box–behnken design (BBD) for a two-variable, three combinations coded \(-1, 0, 1\)
was employed to study the combined effect of these independent variables. (By using Box-Behnken design (BBD), the combination effects of two variables and three combinations of two variables and three combinations were studied.). Experimental design and actual values for the preparation of the loess-based support are shown in table 2. This design required seventeen sets of randomized experiments, which included 12 points at the midpoint of the cube and 5 central points. The model proposed for the response is

\[ Y = b_1A + b_2B - b_3C - b_{12}AB - b_{13}AC - b_{23}BC + b_{11}A^2 + b_{22}B^2 - b_{33}C^2 \]  

Among them, Y is the response calculated by the model; A, B and C are the coded bauxite, dolomite and talcum, respectively, and \( b_1, b_2 \) and \( b_3 \) are linear, \( b_{11}, b_{22} \) and \( b_{33} \) are quadratic, \( b_{12}, b_{13} \) and \( b_{23} \) are interaction coefficient, respectively.

2.4. Characterization of the ceramic support
The phase composition of sample was analyzed by x-ray diffraction (EMPYREAN, PANAlytical B.V. company, Holland); the microstructure of the sample was analyzed by scanning electron microscope (TM-3000, Hitachi company, Japan); the sintering properties of the raw materials were determined by thermal analyzer(TGA/SDTA851e, Metter Toledo company, Switzerland); the bending strength of the sample was measured by a microcomputer controlled electronic universal testing machine (CMT5105, MTS company, America) with reference to GB/T 2833-1996 using the three-point bending method; the porosity and pore size distribution of the sample were determined by capacitor Mercury pressure meter(YG-97A, JWGB SCI. & tech. company, China); the pure water flux of the sample was measured by a homemade pure water device.

3. Results and discussion

3.1. Establishment and analysis of mathematical models
The green body of the support was prepared according to the test design table, and the finished product of the support was prepared under the optimal sintering system (sintering temperature of 1140 °C for 9 h). The pure water flux and bending strength of the support were used as response values, and the optimal conditions were analyzed to determine the higher pure water flux and bending strength.
The RSM was used to evaluate the effect of bauxite (A), dolomite (B) and talcum (C) on performance of the support, and then a model was built to describe the behavior of bending strength and pure water flux, and the process was optimized by finding the best concentration of bauxite, dolomite and talcum. In this case, non-significant terms can be removed to make the regression equations simple. Therefore, the model equation for each performance is in the form:

\[
Y = 33.64 - 7.57A + 0.40B - 3.31C - 1.80AB + 0.19AC + 7.42BC + 2.44A^2 \\
+ 6.88B^2 - 5.58C^2
\]

\[
Y = 3133.18 - 1238.18A + 75.35B - 489.22C - 338.33AB + 1637.63AC \\
+ 508.80BC
\]

Among them, \(Y_1\) = bending strength (MPa), \(Y_2\) = pure water flux (L·m\(^{-2}\)·h\(^{-1}\)·MPa\(^{-1}\)), \(A\) = bauxite (%), \(B\) = dolomite (%), \(C\) = talcum (%).

Figures 3 and 4 show the results of the analysis of variance. The significance of the model can be further judged by the significance of the coefficients in the analysis of variance regression equation [26]. It was observed that the experimental residual distribution was within the normal range, which indicated that the model selected in the experiment was appropriate. Figures 5 and 6 show a comparison of actual and predicted values. It was observed that the experimental values were basically evenly distributed around the prediction line, indicating that the experimental values agree well with the predicted values. These indicated that the experimental model using BBD could well reflect the relationship between the independent variable (additive amounts) and the response value (bending strength and pure water flux) during the preparation of the support.
After fitting and analyzing the experimental data, the regression equation and the prediction values of response surface were obtained. It was predicted highest pure water flux was $8482.78 \text{ l m}^{-2} \text{ h}^{-1} \text{ MPa}^{-1}$, and bending strength was $43.10 \text{ MPa}$. In the raw material ratio of this sample, bauxite accounts for $48.85 \text{ wt}\%$ and dolomite accounts for $9.93 \text{ wt}\%$, talc accounts for $8 \text{ wt}\%$. And the measured results show that the pure water flux was $8323.73 \text{ l m}^{-2} \text{ h}^{-1} \text{ MPa}^{-1}$ and the bending strength was $42.24 \text{ MPa}$. The errors between the predicted values of pure water flux and bending strength and the measured values of pure water flux and bending strength were $-1.87\%$ and $-1.32\%$, respectively. These errors were all less than $10\%$, indicating that the prediction model is reliable.

### 3.2. Analysis of interaction between factors

Figure 7 shows a contour plot of the interactive effects of three factors on bending strength. With the increase of bauxite, the bending strength of the support decreases. When the amount of bauxite was too large, the proportion of the loess decreased, resulting in a decrease in the components that played a viscous role in the support blank. Because bauxite has a weak water holding capacity, when the amount of bauxite was excessive, the moisture of the support body will be loose quickly during the drying and sintering process, and the surface was prone to crack, reducing the bending strength. As the increase of dolomite addition, the bending strength of the support increases or decreases. On the one hand, dolomite can increase the porosity of the support and reduce the coherence of the support, resulting in a reduction in bending strength. On the other hand, a large number of high-melting substances (calcium oxide and magnesium oxide) are generated, and these substances are filled in the aggregate to make the better mechanical stability of support. With the increase of talc, the bending strength of the support decrease and change greatly, which was consistent with the findings of
Yang et al [27]. This was because MgO contained in talc reacts with SiO₂ and Al₂O₃ in loess at high temperatures to form cordierite with poor mechanical properties.

From the three-dimensional response surface graph, it showed that the influence of bauxite and dolomite on the mechanical strength of the support was more significant. Bauxite strengthens it, but dolomite weakens it. However, when the amount of talc and dolomite was increased at the same time, the bending strength also gradually increased, which was different from the change law when the two exist alone due to the contribution of talc and dolomite to the production of calcareous feldspar, which can reduce the temperature of mullite formation, thereby improving the bending strength of the support.

Figure 8 shows a contour plot of the interactive effects of three factors on pure water flux. With the increase of bauxite, the pure water flux of the support generally showed a downward trend. This was due to the increase in the amount of aluminum caused by the increase of bauxite, and the excess aluminum reacted with the magnesium element in the loess to form magnesium aluminate spinel. Because the spinelization reaction was dominated by liquid phase sintering, the porosity was slightly reduced [28]. With the increase of dolomite, the contour line changed from dark blue to yellow, the colour span was larger, and the pure water flux of the support showed an upward trend. This shows that dolomite had a better pore-forming effect. With the increase of talc, the contour colour changed from yellow to blue, and the pure water flux of the support decreased. Because the melting point of talc is only 800 °C, it is easy to form a pore structure filled with molten material, which reduces the permeability of the support.

It can be seen from the three-dimensional response surface graph that the pure water flux of the support was more sensitive to the changes of dolomite and talc. Without talc, the pure water flux of the support was proportional to the amount of dolomite added. With the addition of talc, the pure water flux of the support
increased slowly with the increase of dolomite. This shows that the presence of talc can inhibit the pore-forming effect of dolomite slightly.

3.3. Characterization of the best sample

3.3.1. XRD analysis of samples

Figure 9 shows the x-ray diffraction pattern of the best sample. It can be seen that the main crystal phases were quartz, corundum and mullite. Quartz is a framework structure composed of silicon-oxygen tetrahedra connected with each other at the top angle and extending to three spaces. Since they are connected by covalent bonds and then connected very closely, the pores are small and other particles are not easy to invade into the net holes, resulting in high crystal purity and strength. Quartz has strong acid resistance, which increases the acid resistance of the support body to some extent. Corundum belongs to the tripartite crystal system, oxygen ions for hexahedral compact accumulation, formed by 6 O$^{2-}$ ions an octahedron, octahedral space into the center of a small radius of Al$^{3+}$. Since Al$^{3+}$ is $+3$ valence and O$^{2-}$ is $-2$ valence, only four Al$^{3+}$ are filled in the six octahedral. One out of every three octahedrons is empty and is regularly distributed in the accumulation of crystals, so the crystal cells of corundum are rhomboids at the center of the face. This structure makes the phase crystal shape of corundum stable and ensures the high bending strength of the support body. In addition, part of the dose-Al$_2$O$_3$ and SiO$_2$ had reacted to produce acicular or columnar trapezium mullite with high hardness and good resistance to chemical corrosion. The crystalline phases of quartz and feldspar and chromium alumina were also shown in the figure, which could increase the mechanical strength of the support body.

Figure 8. 3D response surface and contour plots showing interactive effects of various factors on pure water flux.
3.3.2. SEM and energy spectrum analysis of the best sample

In order to further explore the reason for the improvement of mechanical strength of the support, the samples with poor mechanical strength during the experiment were selected and compared with the best samples, taking sample No. 4 as an example. Figure 10 is the SEM image of No. 4 sample (a) and the best sample (b) cross-section magnified. It can be clearly observed that the link structure between the raw material particles in the cross-section of the No. 4 sample is fine, while the particles of the best sample have a melting phenomenon and fuse with each other. In addition, during the formation of mullite, cordierite and some other substances, the grains grew continuously. The molten material wrapped the grains gradually, which may also lead to the formation of a neck-shaped structure between the raw material particles, making the link structure thicker and more densified between the particles, thereby enhancing the mechanical strength of the support body macroscopically. The biggest difference between sample No. 4 and the best sample raw material was that there was no dolomite added. However, dolomite contributed to the formation of mullite, which was one of the reasons for the weak link between the raw material particles.

Figure 11 shows the surface topography and element composition analysis diagram of the best sample measured by SEM and EDS. Figure 10(a) shows the surface topography of support body magnified. It can be seen that the surface was not high in smoothness, and the particles of different sizes were scattered and irregular, with the bulk particles occupying up to 1/2 of the volume. In figure 10(c), EDS energy spectrum analysis of these small particles revealed that the main elements were O, Si, Mg, Al and Ca, with the percentage of oxygen atoms accounting for 64.13%, followed by the percentage of silicon atoms accounting for 15.05%. It can be inferred that at this time, the support body was mainly composed of Al₂O₃, SiO₂, MgO and their composites. Figure 10(b) shows the SEM of support body by 20000 times. It can be seen from the diagram that a large number of acicular crystal had been precipitated. And the whiskers were relatively uniform in length, of which diameter was about 0.1 μm. By EDS spectrum analysis, acicular crystal precipitation mainly contained Al, Si and O elements, so it was speculated that the crystals formed are mullite, silica and alumina. Mullite crystals are spindly acicular and radially clustered, which can be identified as mullite by XRD. The low temperature of mullite formation in this study was due to the liquid phase produced by feldspar in the sintering process of loess, which promote the formation of mullite. The detection results of EDS further confirmed the analysis results of the reasons for the enhancement of mechanical strength in the above-mentioned SEM analysis.

3.3.3. Pore size distribution of the best sample

In order to further explore the influencing factors of the porosity of the support, the samples with poor pure water flux during the experiment were selected, taking sample No. 1 as an example, and it was compared with the best samples. Figure 12 shows the pore throat radius distribution curves of the best sample and No. 1 sample after mercury intrusion test analysis. As shown in the figure 12, there was only one peak in the pore throat radius distribution of the both samples. The pore throat radius of the best sample was concentrated in the range of 1–17 μm, the average pore throat radius was 5.154 μm, the median pore-throat radius was 4.86 μm, and the porosity was 49.9%. While the radius of sample No. 1 was concentrated in the 1–13 μm range, the average pore-throat radius was 3.97 μm, the median pore-throat radius was 3.16 μm, and the porosity was 27.6%. It can be seen that...
Figure 10. SEM images of No. 4 sample and the best sample.

Figure 11. SEM image and energy spectrum of the best sample.
the average pore size and porosity of sample No. 1 were both smaller than those of the optimal sample, mainly because of the absence of dolomite and talc in sample No. 1.

Talc had little effect on pore formation. But when talc and dolomite were added at the same time, it was easy to react with the raw materials to form anorthite at high temperature. In the presence of anorthite, the formation temperature of mullite was lowered, which facilitated the formation of molten mass, further promoting the formation of neck structures between the raw material particles. The connection between the raw material particles became stronger, thereby improving the mechanical strength of the support. Dolomite is the main pore-forming material. It can generate CO₂ gas to escape during the sintering process, resulting in the formation of pores in the support, which can effectively improve the pure water flux. However, the formation of pores will inevitably have a negative impact on the mechanical strength of the support, but the addition of talc enhanced the mechanical strength. Therefore, dolomite was the main factor for the formation of pores, and talc avoided its negative effect on mechanical strength while increasing the porosity.

4. Conclusion

(1) Loess can be used as a low-cost ceramic membrane support aggregate. In order to improve the mechanical strength, it was necessary to add aluminum source, such as bauxite; Adding dolomite was helpful to increase the porosity of the support; The addition of talc was helpful for the forming of the support wet blank, and at the same time, it conducted to avoid the negative impact of the pore-forming effect of dolomite on the mechanical strength of the support.

(2) The comprehensive performance of ceramic membrane support was measured, which combined with pure water flux and flexural strength. The response surface optimization method was used to determine the optimal addition parameters of ingredients: 48.85 wt% of bauxite, 9.93 wt% of dolomite, 8 wt% of talc. Under this condition, the bending strength was 42.24 MPa, the pure water flux was 8323.73 l m⁻² h⁻¹ MPa⁻¹, and the average pore throat radius was 5.15 μm, which met the application performance of porous ceramic supports.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).
Ethical statement
There is no conflict of interest. All data that support the findings of this study are included within the article (and any supplementary files).

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