A novel preparation of high permeation SiC supports for NaA zeolite membrane by in situ reaction bonding

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Abstract
The performance of zeolite membrane supports directly determines the synthesis performance and industrial application of zeolite membranes. In this study, a novel and high permeation SiC supports for NaA zeolite membrane were prepared by carrying out in situ reaction bonding at high temperature in the air atmosphere to coat the SiC particles with aluminum sol. The effects of process parameters on the formation of porous structure of SiC supports were systematically investigated, and the underlying mechanism was explored. During the sintering process, high-temperature Al2O3 from aluminum sol reacted with SiO2 generated by oxidation of the surface of SiC particles to form mullite, which exhibited excellent chemical stability and improved the mechanical properties and chemical stability of the supports. The porous SiC supports for zeolite membrane displayed obvious three-dimensional connected pore structure, with smooth inner surface of the pores and obvious connection neck between the particles. The porous SiC supports for zeolite membrane showed good in situ mullite bonding between the particles, and high permeability. It has a porosity of 36.3%, an average pore size of 1.42 µm and a bending strength of 32.4 MPa. All these figures indicate good comprehensive properties that are consistent with the rigorous requirements of zeolite membrane synthesis and industrial application.

Keywords SiC · Low cost · Zeolite membrane supports · Zeolite membrane

Introduction
As a new separation methodology, membrane technology is characterized by high efficiency, lower cost of energy, easy process control and convenient operation (Fan et al. 2021; Hua et al. 2021; Park et al. 2017). Currently, the industrialization of some new membrane technologies has been realized, and pervaporation technology is under rapid development. NaA zeolite membrane has become one of the most popular inorganic membrane materials due to its unique advantages and good application prospects in terms of organic dehydration, azeotrope separation, pollutant removal and gas separation (Gao et al. 2021; Chen et al. 2020; Wang et al. 2019).

In spite of progress that has been made in the research of NaA zeolite membranes, the technical challenges in industrial application, such as the control of micro-morphology of supports, the poor adhesion strength between supports and zeolite layers, the control of synthesis conditions and the repeatability of synthesis, could not be dismissed (Zhu et al. 2021; Chen et al. 2021; Liang et al. 2021; Morigami et al. 2001; Cho et al. 2010; Caro et al. 2000; Li et al. 2013; Huang et al. 2013; Shao et al. 2014). Among them, the development of zeolite membrane supports restricts the industrial application of zeolite membranes. Alumina (Al2O3), mullite (3Al2O3·2SiO2), porous glass, and stainless steel, etc., are commonly used as supporting materials for zeolite membranes. Silicon carbide (SiC) ceramic membrane materials are similar to oxide ceramic material with regard to excellent properties and have strong acid and alkali resistance, high thermal shock resistance and almost zero wetting angle (Liu et al. 2021; Xiaohong et al. 2021; Jiang et al. 2021). Such hydrophilic property ensures that SiC porous material as NaA zeolite...
membrane support wetting well with water during industrial solvent dehydration, thereby greatly improving the dehydration efficiency of zeolite membrane and reducing the cost.

However, pure porous SiC ceramics are usually prepared by using recrystallization method, which manifests complex production process, high sintering temperature and high production cost. A number of studies have been carried out to overcome these disadvantages. Dey et al. reported a preparation method based on in situ reaction to produce SiO₂ as binding phase, in which the sintering temperature could be lowered to merely 1300 °C (Dey et al. 2013). The membrane support prepared by this method has high strength and good oxidation resistance. However, the thermal expansion coefficient of SiO₂ is notably larger than that of SiC (Dey et al. 2013). In other words, the high-temperature strength and thermal shock resistance of porous ceramic membrane support bonded with SiO₂ will be reduced. In addition, SiO₂ has poor alkali corrosion resistance in hydrothermal synthesis of zeolite membranes. It cannot meet the application requirement of membrane support materials of zeolite membranes. In contrast, mullite and SiC, by virtue of low and similar thermal expansion coefficient, high creep resistance and thermal stability, and good chemical compatibility at high temperature, have become popular. The surface of SiC will be carbonized during high-temperature sintering in the air atmosphere. Then, SiC will be decomposed into SiO₂ and CO₂. The existence of SiO₂ will greatly reduce the mechanical strength and alkali resistance of silicon carbide ceramic support (Xiaohong et al. 2021; Dey et al. 2013). If the aluminum source is added to the SiC support during the process of high-temperature air atmosphere sintering, it can react with SiO₂ to consume excessive SiO₂ and form mullite phase with excellent chemical stability in situ, thereby greatly improving the mechanical properties and chemical stability of SiC ceramic supports. The addition of aluminum sol not only consumes SiO₂ produced by high-temperature oxidation decomposition of SiC, but also greatly improves its surface physical and chemical properties. Its good pore structure and binding phase contribute to high porosity, high strength and toughness. All these features improve the performance of the supports. In addition, porous SiC ceramic zeolite membrane supports with different pore sizes and porosity can be obtained by adjusting the preparation process parameters. Nevertheless, these procedures are few reported.

In this study, the porous SiC zeolite membrane supports were prepared by in situ reaction and sintering in high-temperature air atmosphere by taking SiC as powder raw material and aluminum sol as aluminum source. The effects of process parameters on the formation of porous structure of silicon carbide and the underlying mechanism of porous SiC zeolite membrane supports were systematically investigated.

**Experimental**

**Sample preparation**

SiC particles (d50 = 8 μm, Hebei Tenai Welding Material Co., Ltd., China) were used as the main raw materials and aluminum sol (alumina content of 25 wt%, Shandong Aluminum Co., Ltd., China) was used as the additive source of aluminum. Two types of starting powders were prepared: SiC and SiC + aluminum sol (amount of Al₂O₃ = 5 wt%), referred to as S and SA, respectively. Pure SiC was used to form the green body. The prepared raw materials were fully mixed in a ball mill until the additives were completely coated on the surface of SiC particles. After the even mixing, a certain amount of binder and lubricant were added, the flake body sized of φ 30×2 mm and the strip body sized of 50×6×6 mm were compressed in the powder crystal pelleting machine (model 769YP-24B, Tianjin Keqi HIGH and New Technology Corporation, China). After being dried, the body was heated in the air at a heating rate of 4 °C min⁻¹ in a programmed electric furnace.

**Characterization of samples**

A scanning electron microscope (Quanta 200, FEI Company, the Netherlands) equipped with EDX was used to characterize the micro-morphology and chemical composition of the samples. X-ray diffraction (XRD, D8 advance, Bruker Instrument Co., Ltd., Germany) was used to analyze the phase composition of the samples at different sintering temperatures. The porosity of the sample was determined by using Archimedes method (GB1996-80), and the average pore size of the sample was evaluated by using gas bubble-pressure method. Three-point bending method (GB1965-80) was used to characterize the bending strength of the carrier on a tensile testing machine (model CMT6203), with a pivot span of 40 mm and a loading rate of 0.5 mm/min.

**Results and discussion**

**Sintering behavior with sintering temperature**

The TG-DTA analytical curves of green samples S and SA in air atmosphere are illustrated in Fig. 1. Apparently, the mass loss rate of the sample S prepared by ultrafine SiC powder without aluminum gel was the highest, reaching 4.7% at 510–930 °C. The mass of the sample S slowly increased along with higher temperature after reaching 930 °C, while the mass of the sample increased rapidly along with the temperature after reaching 1210 °C and recovered to
98.8% at 1500 °C. This could be attributable to the loss of water and the combustion of organic binder added in the preparation process before 510 °C. Meanwhile, the sample quality remains stable at the temperature of 510–930 °C, owing to the sole presence of SiC. Due to the formation of amorphous silica by the slow oxidation of SiC, the sample mass increased slowly when the temperature increased to 930 °C. As shown in formula (1), a dense SiO₂ layer was formed on the surface of SiC. As a matter of fact, the diffusion coefficient of oxygen in SiO₂ was rather small at lower temperatures, and however, oxygen would diffuse to the surface of SiC particles swiftly after the temperature exceeding 1210 °C, which would lead to thickened oxide layer. The formation of SiO₂ became prevalent when SiC surface was oxidized at lower temperature in glassy state (Dey et al. 2013). However, SiO₂ in glassy state would be transformed into cristobalite with increasing temperature, according to formula (2). Owing to volume expansion during phase transformation, the locally produced higher stress caused poor thermal shock resistance for cristobalite, thereby resulting in tiny cracks in the surface layer in the fluctuation of temperature. In the event of high temperature, cracks served as the fast channel for oxygen diffusion, thereby promoting further oxidation of SiC surface. The excessive oxidation of the surface layer of particles narrowed down the pore structure of materials and led to rapidly increased sample quality. The mass loss rate of the sample SA prepared reached 4.78% at the temperature of 510 °C after aluminum gel was added. The mass loss rate was strictly maintained at 4.7–4.8% at 510–660 °C. When the temperature was 660–875 °C, the sample mass decreased rapidly again, and then, the sample mass loss rate became stable at around 5.1% before the temperature reached 1140 °C. After that, the sample mass began to increase slowly along with rising temperature, and the sample mass increased rapidly along with the temperature before it reached 1290 °C. The sample mass was recovered to 96.3% at 1500 °C, which results from the fact that the mass loss of samples SA decreases rapidly before 510 °C due to loss of water and combustion of organic binder added in the sample preparation process before 510 °C. Meanwhile, the quality of samples remained stable at 510 °C–660 °C due to the existence of aluminum sol and SiC. After the temperature reached 660–875 °C, a large endothermic peak could be observed in the DTA curve, indicating that the aluminum sol in the sample began to decompose and the loss of structural water. Hence, sample quality could decrease rapidly again. When the temperature was elevated to 1140 °C, the surface of SiC particles began to oxidize slowly to form amorphous silica, as shown in formula (1). At this moment, the sample mass began to increase slowly. When the temperature reached 1290 °C, the surface of SiC particles would be oxidized rapidly (Jiang et al. 2021). Meanwhile, the aluminum source began to react with SiO₂ generated by oxidation of SiC particles, yielding mullite in situ and rapidly enhancing sample mass, as shown in formula (3).

\[
\text{SiC} + 2\text{O}_2 \rightarrow \text{SiO}_2 + \text{CO}_2 \tag{1}
\]

\[
\text{SiO}_2(\text{amorphous}) \rightarrow \text{SiO}_2(\text{cristobalite}) \tag{2}
\]

\[
3 \text{Al}_2\text{O}_3 + 2 \text{SiO}_2 \rightarrow (3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2)(\text{mullite}) \tag{3}
\]

In order to understand the reaction mechanism, samples SA were sintered at 1350 °C, 1400 °C, 1500 °C and 1550 °C for 2 h, respectively. The corresponding XRD diffraction patterns revealed the formation of mullite phase at 1350 °C (Fig. 2). However, cristobalite phase was observed in the support, and the content of cristobalite phase increased along higher temperature. Such a trend could be attributable to the transformation of amorphous SiO₂ glass phase pressed forward by oxidation on the surface of SiC particle, according
to formula (2). With increased sintering temperature, the diffraction peak intensity of mullite phase in the sample was elevated. Such a pattern could be explained by the formation of activated alumina in the aluminum sol and the formation of silicon oxide due to high-temperature oxidation on the surface of SiC, the reaction of which yielded more mullite, according to formula (3). Consequently, the amount of mullite in the binding phase between particles was increased to improve the mechanical strength and chemical stability of the supports.

**Microstructure analysis and phase composition of the supports**

Figure 3 shows SEM images of the sample SA prepared at 1500 °C for 2 h. Noticeably, the sample SA exhibited excellent pore structure, as evidenced by the formation of an obvious three-dimensional interlocking pore structure in the sample (Fig. 3a). In addition, the inner surface of the pore was relatively smooth, revealing the clear neck connection between the particles. The high porosity was also consistent with results of pore structure characterization and high permeability performance of the samples. Chemical composition analysis was carried out on the microscopic composition of the sample (Fig. 3b). C, Si and Al element were found at the neck joint between the sample particles, yet no alkali metal element was found. This indicates that the neck and particle surface of the sample are mainly composed of SiC, quartz and mullite.

The XRD patterns of the phase composition of the samples at this temperature (Fig. 4) showed that the diffraction peaks of phase composition of sample S were mainly SiC diffraction peaks and partial cristobalite diffraction peaks. However, it is found that the main phase composition of sample SA at this temperature was SiC phase, during which the samples were accompanied by a small amount of mullite and cristobalite. The intensity of cristobalite in XRD

Fig. 2 XRD patterns of the specimens sintered at 1350–1550 °C for 2 h in the air

Fig. 3 SEM images of the sample SA prepared at 1500 °C for 2 h in the air

Fig. 4 XRD patterns of the specimens sintered at 1500 °C for 2 h in the air
diffraction peak was obviously weaker than that of the sample S. Such disparity could mostly be caused by in situ reaction between the additional aluminum source and the glass phase formed by high-temperature oxygen on the surface of SiC particles to form mullite phase. The XRD patterns further confirmed the formation of mullite phase on the surface of SiC, which greatly reduced the content of SiO2 facilitated by high-temperature oxidation of SiC in the air. Such procedure improved the mechanical properties and chemical stability of the sample.

**Pore structure parameters of the supports**

As shown in Fig. 5, the porosity of the samples SA was notably higher than that of the samples S, and the porosity of both samples decreases when the sintering temperature soared. The porosity of the samples S decreased from 36.7% at 1350 °C to 32.6% at 1550 °C. The extent of increased sintering degree between particles could be attributed the continuous oxidation of silicon dioxide glass phase on the surface of SiC particles. However, the porosity of the samples SA decreased from 39.8% at 1350 °C to 33.2% at 1550 °C, a pattern consistent with the sintering behavior of samples SA with aluminum source.

Figure 6 displays the association between the average pore size of sample and sintering temperature. Apparently, the average pore size of the sample SA gradually became larger with higher sintering temperature, from 1.21 μm at 1350 °C to 1.42 μm at 1500 °C, yet decreased to 1.29 μm as the sintering temperature continued to increase to 1550 °C. The addition of aluminum source facilitated the in situ reaction between SiC particles to form mullite, and thus neck connection. The growth of mullite crystals led to expanded pores between silicon carbide particles (Chen et al. 2015). However, when the sintering temperature exceeds a certain temperature, excessive silicon dioxide glass phase was formed due to the rapid oxidation of SiC surface, and the amorphous glass phase diffused into the pores in the support, inducing decreased porosity and pore size of the support. In contrast, the surface of SiC particles in sample S was oxidized continuously to form silica glass phase. The occurrence of densification behavior led to decreased pore size along with higher sintering temperature.

**Filtration and permeability of samples**

The filtration permeability of the sample is determined by using the filtration device and deionized water acted as medium. As shown in Fig. 7, the pressure difference of filtration operation ranges from 0.1 to 0.3 MPa, and three groups
of data are measured under each operation pressure to obtain the average flow rate under the corresponding pressure. The apparent specific permeability (Kp) of the porous material is expressed in Eq. (4) (Lukasiewicz and Reed 1988; Reed 1993):

$$K_p = \frac{dQ}{dP} \frac{\eta L}{A_{eff}}$$  \hspace{1cm} (4)

where $Q$ represents the volume flow rate of deionized water, $P$ represents the operating pressure, $\eta$ represents the viscosity of deionized water, $L$ represents the thickness of the sample, $A_{eff}$ represents the effective filtration membrane area of the sample, and $r$ represents the average pore size of the sample. The samples have two kinds of pore structures: open pore structure and closed pore structure. In the pore structure, the aperture could be subdivided into one end closed aperture and interlocking pore structure connected at both ends. In the filtration process of porous materials, the through-hole structure with through ends works as filter, and its volume fraction could be calculated according to Eq. 5 (Lukasiewicz and Reed 1988; Reed 1993). Where $r$ represents the average pore size of the sample.

$$\varepsilon_{eff} = 24 \frac{dQ}{dP} \frac{\eta L}{A_{eff} r^2}$$  \hspace{1cm} (5)

The fraction of the penetrating porosity to the open porosity can be calculated according to Eq. 6 (Lukasiewicz and Reed 1988; Reed 1993) where $\varepsilon_o$ represents the open porosity of the sample as measured by the Archimedes method.

$$\tau = \frac{\varepsilon_{eff}}{\varepsilon_o} \times 100\%$$  \hspace{1cm} (6)

As shown in Fig. 8, the volume flow rate of pure water through the sample has a good linear relationship with the operating pressure, which is in agreement with Darcy law. Moreover, the sample sintered at 1500 °C with alumina source has a higher volume flow rate of pure water compared with that of other sintering temperatures. Such pattern is consistent with the change of pore structure of the sample at this temperature.

The through porosity of samples heated at different temperatures could be calculated according to Eq. 5 (Fig. 9). The maximum penetrating porosity of SA samples sintered at 1500 °C was 31.1%. According to Eq. 6, the penetrating porosity of SA samples sintered at this temperature accounts for 85.7% of the total open porosity. Given that the samples were sintered at high temperature without mullite formation, the surface of SiC was partially oxidized to form amorphous glass phase, and high-temperature viscous flow became obvious with higher sintering temperature. Alumina sol acted as a pore-forming agent due to the dehydration with increased temperature. With the continuous increase in sintering temperature, the alumina particles filled in the pores between SiC particles, blending into the glass phase to form a rigid mullite structure in situ. Subsequently, the pore structure between particles in the sample was opened up to form a through pore structure, resulting in increased open porosity and higher permeability of the sample (Colombo et al. 2008).

The three-point flexural strength of the samples sintered at different temperatures is shown in Fig. 10. In the range of 1350–1550 °C, the mechanical strength of the samples increased with higher sintering temperature. When the sintering temperature exceeded 1450 °C, the mechanical strength of the sample SA increased drastically from 22.3 to 38.5 MPa. Such a trend was probably caused by in situ
reaction between alumina and silicon dioxide formed by oxidation of SiC surface in the sample, which led to shortened glass phase and rapid increase in mullite phase. However, the three-point flexural strength of sample S sintered at 1300 °C/2 h was 8.5 MPa and increased to 17.9 MPa when the sintering temperature surpassed 1550 °C/2 h. The strength increase is not obvious in the whole temperature range, owing to the continuous oxidation of the surface of SiC, the thickening of silica glass phase and higher sintering degree between SiC particles with soaring temperature.

Conclusion

In this study, SiC powder was used as raw material, and aluminum sol was used as aluminum source to prepare mullite-bonded porous SiC supports for zeolite membrane during in situ reaction at high temperature in air atmosphere by coating SiC particles with aluminum sol. The effects of process parameters on the formation of porous structure of porous SiC supports were systematically investigated, and the formation mechanism was explored. During the high-temperature sintering procedure, the addition of aluminum sol consumed SiO2 produced by oxidation decomposition of SiC, but also facilitated the formation of mullite phase to greatly improve its surface physical and chemical properties. The good pore structure and binding phase ensure high porosity and high strength and toughness, all of which improve the performance of the support. The porous SiC supports for zeolite membrane prepared by using this method showed obvious three-dimensional connected pore structure, with smooth inner surface of the pores and apparent connection neck between the particles. The porous SiC supports with good in situ mullite bonding between particles and high permeability were prepared at 1500 °C, the porosity reached 36.3%, the average pore diameter was 1.42 μm, and the bending strength was 32.4 MPa. All these figures confirm the outstanding comprehensive properties that fulfill the requirements of zeolite membrane synthesis and industrial application.

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Declarations

Conflict of interest

All authors certify that they have no affiliations with or involvement in any organization or entity with any financial interest or non-financial interest in the subject matter or materials discussed in this paper.

Ethical approval

This article does not contain any studies with human participants or animals performed by any of the authors.

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