Preparation of Pt-ZSM-5 zeolite membrane catalysts for isomerization of linear alkane

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Abstract

A ZSM-5 supported membrane was synthesized by secondary growth method. The Pt-ZSM5 membrane was prepared by impregnation method. The membrane was characterized by single gas permeation step at room temperature. The isomerization of n-pentane was chosen as a probe reaction for evaluating the catalytic performance of the membrane. In particular, the effect of the space velocity and the time on stream were considered. After the catalytic tests, the membrane was characterized by SEM, EDX and XRD. N₂ permeance for the membrane, after calcination, was equal to $2.9 \times 10^{-7}$ mol/m$^2$.s.Pa indicating a coverage of the larger support pores by the zeolite crystals. This results was also confirmed by the SEM investigation. In addition, XRD analysis showed as the ZSM-5 was the desired zeolite-type. During the catalytic tests, it was observed a decrease of the nC₅ conversion and an increase of the iC₅ selectivity with WHSV. The nC₅ conversion was decreased from 2.5 to less than 0.5, with an enhancement in weight hourly space velocity (WHSV), while the selectivity increases from 30 to over 70. On the other hand, it's conversion on catalyst enhanced from 10% to approximately 38%, with an increase in the reaction temperature from 250 to 450°C.

Keywords: catalytic zeolite membrane; Pt catalyst; Pent; Isomerization.
1. Introduction

The isomerization of catalytic linear alkanes to branched alkanes are an important industrial reaction due to the increasing demand of iso alkanes, which are used as a substitute for octane boosters instead of aromatic and oxygenate compounds, which are subjected to stringent environmental limitation (Al-Kandari, Al-Kandari, Al-Kharafi and Katrib, 2009, Ramos, Gómez, Dorado, Sánchez and Valverde, 2005, Villegas, Kumar, Heikkilä, Lehto, Salmi and Murzin, 2006, Dhar, Vekariya and Sharma, 2017). Gasoline is formed of a large number of linear-chain compounds with low octane number. Nevertheless, these compounds can be rises when the gasoline is placed under an isomerization reaction by which linear-chain molecules are transformed to branched molecules (Yoshioka, Garetto and Cardoso, 2005, Noh, Shi, Zones and Iglesia, 2018). In isomerization reactions a mixture of di-branched, mono-branched and linear molecules are formed, therefore converting linear paraffins in the isomerization reaction is limited by a thermodynamic equilibrium (Baudot and Bournay, 2009, Ejtemaei, Charchi Aghdam, Babaluo, Tavakoli and Bayati, 2017). The linear paraffins from the isomerization process effluent are separated and recycled at the entrance of isomerization reactor. A membrane reactor (MR) consists of a combination of the distribution process or membrane separation with a chemical reactor in one system. Chemical processes are easier due to the integration of distribution/separation and reaction that leading to a much lower processing costs than more conventional processes (Daramola, Deng, Pera-Titus, Giroir-Fendler, Miachon, Burger, Lorenzen and Guo, 2010, Li, 2007). There are many scientific reports on the synthesis, characterization and development of catalysts for pentane isomerization. Aboul-Gheita et al. evaluated the effect of Pt concentrations on SZ catalyst performance (Aboul-Gheita, Gad, Abdel-Aleem, El-Desouki, Abdel-Hamid, Ghoneim and Ibrahim, 2014). The Ir/Pt-HZSM5 zeolite
catalyst was prepared by saturation of iridium on Pt-HZSM5 zeolite. The influence of Si/Al ratio on n-pentane isomerization using Ir/Pt-HZSM5 was studied by Setiabudi et al. The highest activity for isomerization of n-pentane was achieved by Ir/Pt-HZSM5 zeolite catalyst for Si/Al ratio of 23 (Setiabudi, Jalil, Triwahyono, Kamarudin and Mukti, 2012, Setiabudi, Jalil, Triwahyono, Kamarudin and Jusoh, 2013). The n-pentane isomerization on the Zn/HZSM-5 zeolite catalyst was carried out by Triwahyono et al. Their results indicated that the conversion to iC5 relevant of the promotive effect of H2 as a carrier gas (Setiabudi, Jalil, Triwahyono, Kamarudin and Jusoh, 2013). Our best knowledge, there is no existing report related to pentane isomerization using catalytic membrane reactors.

Zeolites with their regular structure, thermal and chemical stability have shown a major potential for combined systems of separation/reaction as membrane reactor (Deshayes, Miró and Horowitz, 2006, Zhang, Hong, Chen, Gu, Jin and Xu, 2012, Anbia and Aghaei, 2018, Medrano, Garofalo, Donato, Basile, De Santo, Gallucci, Cofone, Ciuchi and Algieri, 2018, Dong, Wang, Rui and Lin, 2015). These advantages propose zeolite membranes as a new technique for organic synthesis and particular separation in the chemical industry (Sato, Kumagai and Itoh, 2010, Ejtemaei, Charchi Aghdam, Babaluo, Tavakoli and Bayati, 2018, Charchi Aghdam, Ejtemaei, Babaluo, Tavakoli, Bayati and Bayat, 2016, Algieri, Comite and Capannelli, 2013). The MFI-type zeolite (ZSM-5 and aluminum-freesilicalite-1) with a pore size of 0.58nm can separate nC5 (minimum kinetic diameter = 4.3Å) from 2-methyle pentane (minimum kinetic diameter = 5Å) selectively using the difference in adsorption strength and size. Thus, MFI membrane is employed as a separator and as well as a membrane reactor for diverse reactions like linear alkane (C5-C7) isomerization (Setiabudi, Jalil, Triwahyono, Kamarudin and Jusoh, 2013, Song, Feng, Liu, Kang, Zhou, Xu and Yu, 2009, Kumar, Masloboischikova, Kustov,
Heikkilä, Salmi and Murzin, 2007), whereby is provided better product selectivity. A study of alkanes isomerization using MFI membrane reactor was performed by Gora et al. (Gora and Jansen, 2005). They passed a feed (iso-hexane/n-hexane) during the outer of a tubular membrane was investigated. The conversion of n-hexane to iso-hexane is achieved by the permeation of nC6 through the membrane and within the catalyst phase.

In this study, a zeolite catalytic membrane reactor, for the first time, was used for the nC5 isomerization. The prepared membrane was characterized by SEM, EDX and XRD analyses. Moreover, the permeation property was also evaluated before and after calcination step. Finally, the membrane reactor performance for nC5 isomerization was studied as a function of the space velocity and time on stream.

2. Experimental

2.1. Materials

The chemicals used for the membrane preparation were tetraethoxysilane (TEOS, ACROS) as silica source, aluminumisopropoxide (AIP, Merck) as aluminum source, tetrapropylammonium hydroxide ((TPAOH, 1 M aqueous solution, Merck) as the organic structure-directing agent the template, triethylenetetramine (Trien, Merck) as a chelating agent. All of the solutions were prepared by also adding sodium hydroxide (Merck) and distilled water.

Membrane supports were made of porous α-Al2O3 asymmetric tubes (IKTS, I.D. = 7 mm, O.D. = 10 mm, L = 100 mm, dpore = 100 nm). The tubular support, for removing possible dust and oil
materials inside the pores, were cleaned with acetone in an ultrasonic cleaner for 1 hour. Afterwards, the support was boiled in distilled water for 1 hour and then dried in a furnace at 120 °C for 2 hours.

### 2.2. Membrane preparation

ZSM-5 zeolite membranes were synthesized by using the secondary growth method. This method, separating the nucleation from crystal growth, permits the optimization of each step independently. It provides two stages: seeding and growth. In this work the membranes were seeded by means of the procedure reported by Algieri and coworkers (Garofalo, Carnevale, Donato, Drioli, Alharbi, Aljlil, Criscuoli and Algieri, 2016). After the seeding, the growth of the crystals was performed by pouring the synthesis solution on the inner support positioned in a Teflon-lined autoclave. The formation of the zeolite layer on the outer surface of the support was avoided wrapping it with Teflon tape. Finally, the hydrothermal treatment was carried out putting the autoclave in a preheated furnace. The operating conditions used for the growth are reported in table 1:

**Table 1**

<table>
<thead>
<tr>
<th>Condition</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature</td>
<td>120°C</td>
</tr>
<tr>
<td>Time</td>
<td>2 hours</td>
</tr>
</tbody>
</table>

After the hydrothermal synthesis, the synthesized membranes were washed several times with distilled water, afterwards dried at 100°C for 8 hours to remove the water adsorbed in the pores of zeolite. After the synthesis, the prepared membrane was calcined up to 753 K to remove the template. The calcination was performed using a furnace with heating and cooling rates of 1 K/min.

### 2.3. Pt-ZSM-5 catalytic membrane layer
For obtaining the HZSM-5 membrane, Na\(^+\) ions present in the zeolite layer were exchanged with ammonium in a 0.1 M NH\(_4\)Cl solution for 4 hours at 95°C. This procedure was repeated for three times. Then, the cation-exchanged membrane was dried at 50 °C and subsequently heated up to 450 °C for 3 hours (Wang and Lin, 2012). Pt/zeolite catalyst layers were prepared by impregnation with an aqueous solution of hexachloroplatinic acid hydrate for 24 hours. The catalysts were dried after saturation and activated under H\(_2\) atmosphere for 3 h at 623 K.

2.4. Zeolite membranes characterization

Powder X-ray diffraction (XRD, Siemens D500...insert the characteristic of the instrument) was performed on the material scratched off the membrane surface. The morphology of the zeolite membrane was observed by scanning electron microscopy (SEMCamScan MV2300, Check Republic). The energy dispersive spectroscopy (EDS) and element mapping were applied to analyze the element dispersion of in the membrane sample. Before membrane reactor experiments, C\(_5\) isomers permeation properties including separation factor and permeance of the membrane were evaluated in a continuous flow system. The isomerization reaction of \(n\)-pentane was chosen to evaluate the catalytic activity of the prepared zeolite membrane.

The total pressure at permeate side was kept at 1 atm for binary permeation tests and the used membrane pressure difference (\(\Delta p\)) was 0.1–1 bar. A nitrogen stream was applied to eliminate the permeating species by sweep. Both components can permeate the feed solution, and afterwards separation can be achieved due to differences in diffusion rates, sorption rates, and the molecule capability to block or inhibit the other from entering the pores. The experimental setup
used for performing the separation tests and the catalytic reactions is showed in Fig.1. The catalytic zeolite membrane was placed in a membrane module and sealed by O-rings. This reactor was placed inside an electrical oven to control the reaction temperature. The gas feed was controlled by mass flow controller. The injection of pentane into the membrane reactor was carried out by means of a syringe pump. The outlet flow of the pump was evaporated into a gaseous state by using thermal wires that are wrapped around the steel tubes connected to the membrane modulus. Hydrogen gas was applied as a reactant in the processes under research. Nitrogen gas was used as a sweep stream in permeate side of membrane module. A nitrogen stream was applied to eliminate the permeating compounds by the sweep side in the membrane separation tests. The flow rates for gases were controlled using mass flow controllers. Gas was injected to the zeolite catalyst membrane cell, and permeate and the retentate pressures were controlled by back pressure regulators. Also, the differential pressure on the zeolite membrane was controlled using differential pressure gauges. Both of the retentate and permeate were evaluated on-line using a Teif Gostar Company gas chromatograph with a capillary column and a flame ionization detector (Serial. No.: p2085307, Cat. No. TR-110222, ID: 0.25, l: 25 m, TRB-1, Tecknokroma). The experiments were performed in H₂ at 250 °C and constant H₂/HC molar ratio 4.8.

**Figure 1**

The selectivity (or separation factor) of the membrane is defined by the following equation:

\[ \alpha = \frac{y_i/y_j}{x_i/x_j} \]  

1

where \( y_i \) and \( y_j \), \( x_i \) and \( x_j \), are the molar fractions of components \( i \) and \( j \) on the permeate (\( y \)) and the feed (\( x \)) sides, respectively.
The total conversion of the n-pentane \( (X_{n\text{-pentane}}) \) was calculated as:

\[
X_{n\text{-pentane}} = \frac{\sum A_i - A_{n\text{-pentane}}}{\sum A_i} \times 100
\]  

(2)

And the selectivity \( (S) \) is described for the reaction product, or asset of products by Eq. 3:

\[
S_i = \frac{\sum A_i}{\sum A_i - A_{n\text{-pentane}}} \times 100
\]  

(3)

where \( A_i \) is the corrected chromatographic area for a particular compound, used to express the conversion and selectivity as molar percentages (López, Guillén, García, Gómez and Ramírez, 2008).

3. Results and discussion

Before the calcination, the membrane was gas-tight indicating as the synthesized membrane was defect-free. After calcination, Single gas permeation test, at room temperature, was performed by using nitrogen as gas. The \( \text{N}_2 \) permeance for the membrane was equal to \( 2.9 \times 10^{-7} \) mol/m\(^2\) s Pa, for the bare support it was about \( 30 \times 10^{-6} \) mol/m\(^2\) s Pa. The lower membrane permeance with respect to the bare support indicates that, at least, the larger support pores are covered with zeolitic layer.

nC\(_5\)/2MB mixture (70/30) were separated using the synthesized Pt-ZSM-5 membrane. As shown in Fig. 2, the selectivity decreased as the feed pressure increased. A possible explanation of this result is owing to the increased transport through the non-zeolite pores with the feed pressure. This result confirms the presence of defects into the zeolite layer. Same results was found by
using a BZSM-5 zeolite membrane (Bayati, Belbasi, Ejtemaei, Aghdam, Babaluo, Haghighi and Drioli, 2013).

**Figure 2**
The isomerization of \( n \)-pentane was chosen as a probe reaction to evaluate the catalytic activity of the prepared catalytic zeolite membrane. The catalytic test were performed at 250 °C considering the results obtained in the same process in previous works. Fig. 3 showed the experimental results in terms of conversion and selectivity obtained in the isomerization of \( n \)-pentane as a function of the space velocity.

**Figure 3**
The experimental data evidenced as an increase of the space velocity determined a decrease of the conversion and an increase of the selectivity. It is possible to explain this result considering that an increase of the space velocity caused a reduction of the contact between reagents and catalytic sites and so the pentane conversion decrease. At the same time, an increase of the space velocity suppressed the side reaction of 2MB by determining an increase of the selectivity. In general, the nC\(_5\) conversion reduced with rising WHSV and the reaction products shift far away from the thermodynamic equilibrium and achieved to particular value without regard to nC5 flow rate, while isomerization selectivity increases with increasing WHSV. Because the WHSV has a critical impact on reaction kinetics, for instance, high WHSV causing reaction-rate-limited conversion and low WHSV resulting equilibrium-limited conversion (Zhang, Wu, Hong, Gu and Xu, 2012). The conversion of \( n \)-pentane and selectivity as a function of the time on stream was shown in Fig. 4.
It is possible to observe as the nC₅ conversion decayed easily with the time on stream. While, the selectivity remained constant, probably because of coke deposition on acid sites. In fact, in different papers it was reported that strong Lewis acid sites in the absence of hydrogen, become active sites for the formation of dehydrogenated carbonaceous species, which become a precursor of coke on the surface of the catalyst (Ye, Sun, Guo, Zhu, Liu, Zhou and Coppens, 2018, Setiabudi, Jalil, Triwahyono, Kamarudin and Jusoh, 2013). The performance of both Pt-ZSM-5 catalysts powder that was reduced at the temperature of 250 and 450°C are presented in Fig.5. The catalysts that reduced at 450°C were more active in pentane isomerization than those reduced at 250°C. The pentane conversion on the Pt-ZSM-5 catalyst enhanced from 10% to about 38%, with an increase in the reaction temperature from 250 to 450°C. Also the reaction selectivity for catalysts reduced at 450°C was more than those reduced at 250°C. Therefore is expected that reduction of catalytic layer at 450°C can be increase the conversion and selectivity of pentane isomerization.

After the catalytic experiments, the zeolite layers grown on the alumina supports were analyzed by using XRD, SEM and EDX. The XRD patterns of α-Al₂O₃ tubular support (Fig. 6b) and ZSM5 powder (Fig. 6c) were compared with that recorded for powder scratched from the surface of the membrane. Characteristic peaks of ZSM5 zeolite and alumina can be observed in the composite membrane spectrum (Fig. 6a). In (Fig. 6a), the peaks at 7-9° and 22.5-24.5° related to MFI-type zeolite indicating the presence of the desired zeolite. Extra peaks related to the alumina
support and R-TiO₂ (rutile phase) intermediate layer (Ejtemaei, Charchi Aghdam, Babaluo, Tavakoli and Bayati, 2018).

**Figure 6**

SEM micrographs of the membrane are shown in Fig. 7. A mostly uniform and compact zeolite layer was present on the alumina support (see Fig. 7a). In addition, the thickness of the zeolite layer on the support was about 50 mm, as shown in Fig. 7b. For a comparison, also the image of bare support is also reported (Fig. 7c and 7d).

**Figure 7**

EDX analyses were also carried out on the membrane cross section of the ZSM5 layer. The presence of Si, Al and Pt was detected (see Fig. 8).

**Figure 8**

The elemental chemical mapping revealed that Si and Al were the major elements present along the thickness of the membrane. In addition, the oxygen was also present outside the zeolite layer being a major component of the support (see Fig.9).

**Figure 9**

The results presented in this work demonstrated the possibility to perform the n-pentane isomerization by using zeolite membrane loaded with Pt. Usually, a zeolite catalytic membrane offers a good dispersion of catalytically active and nano-sized particles in a thin zeolite layer by improving the contact between the reactants and the catalytic site and reducing the by-pass problem (Medrano, Garofalo, Donato, Basile, De Santo, Gallucci, Cofone, Ciuchi and Algieri, 2018). However, for improving the performance of a process by using catalytic zeolite membrane, it is very important to reduce the concentration of defects into the zeolite layer. In
fact, is well-known that zeolite membrane function very well in process were liquid species are involved while the limits are present when gas species are involved for their small sizes and so the necessity to have membrane defect-free (Bedard and Liu, 2018).

4. Conclusion

ZSM5 supported membranes were synthesized by secondary growth method. Pt-loaded zeolite membranes were prepared by impregnation method. SEM analysis evidenced the formation of almost uniform and compact layer on the alumina support. This result was also confirmed by the $N_2$ permeance of the membrane measured after calcination step. In addition, EDS analysis confirmed in the presence of Pt, Si, Al and O into the zeolite layer.

The catalytic tests showed the possibility to perform the nC$_5$ isomerization by using the Pt-ZSM5 membrane. Moreover, it was also observed that the nC$_5$ conversion decreased and the iC$_5$ selectivity increase with WHSV due to the contact time decrease. It was also found that the nC$_5$ conversion decreased with the time on stream probably owing to the coke formation.
Abbreviations

EDS  energy dispersive spectroscopy
MR   Membrane Reactor
SEM  Scanning Electron Microscope
WHSV Weight Hourly Space Velocity
XRD  X-ray Diffraction

List of symbols

A  [dimensionless]  the chromatographic area
S  [dimensionless]  selectivity
X  [dimensionless]  the total conversion
x\textsubscript{i}  [dimensionless]  molar fractions of components i in the feed
y\textsubscript{i}  [dimensionless]  molar fractions of components i in permeate
\alpha  [dimensionless]  the separation factor
References


Behrouz Bayati received the PhD degree in Chemical Engineering from Sahand University of Technology, Iran, in 2013. He is currently an Assistant Professor of Chemical Engineering at Ilam University. His research interests are in the general area of Membrane and membrane reactor process, zeolite membranes, molecular simulation. He has published over 30 research articles on related subjects. In the last decade, he presented different specialized courses.

Catia Algieri is a researcher at Institute on Membrane Technology, National Research Council of Italy (ITM-CNR) since 2001. She collaborated in several international and national projects focused on membranes preparation and characterization for application in water treatment, catalytic reactions and gas separations. She is coauthor more than 45 peer-reviewed papers and one patent. She also did more than 70 oral presentations (also as invited lecture) in National and International Conferences. She has chaired several sessions in conferences. She is also an associate editor of the Euro-Mediterranean Journal for Environmental Integration by Springer-nature.

Ali Akbar Babalou, after graduating from high school in 1992, continued his education in Chemicalpetrochemical Engineering at Sahand University of Technology (SUT). Then, he continued his education and graduated with PhD degree in 2004. After that, Dr. Babaluo continued his education and research-based activities at SUT as Assistant Professor. He is author of more than 80 journal papers, 150 national and international conference papers 22 and 16 national patents (2 patents have scientific certificate).Dr. Babaluo has also done other different scientific activities such as: publishing a journal; creating research center and incubator; writing book and book chapter; working as an editorial board member of two research journals; presenting scientific comments, corrections, and corrigendum on published articles. Now, he continues his viable activities at Sahand University of Technology as a Full-Professor.

Asma Ghorbani received both her BSc and MSc degrees in Chemical Engineering from Ilam University, Iran in 2016 and 2018, respectively. Her research interests are in the general area of separation science, especially membrane and membrane reactor process, zeolite membranes, molecular simulation. She has published more than 4 research papers and one chapter book. She also did one oral presentation in International conference.
Enrico Drioli Emeritus Professor at the School of Engineering of the University of Calabria. Founding Director of the Institute on Membrane Technology, CNR, Italy. Since 2012 Distinguished Adjunct Professor, CEDT King Abdulaziz University, Jeddah Saudi Arabia; since 2010 WCU Distinguish Visiting Professor, at the Hanyang University, Seoul Korea; His research activities focus on Membrane Science and Engineering, Membranes in Artificial Organs, Integrated Membrane Processes, Membrane Preparation and Transport Phenomena in Membranes, Membrane Distillation and Membrane Contactors, and Catalytic Membrane and Catalytic Membrane Reactors. He is author of more than 800 scientific papers, 22 patents and 24 books on Membrane Science and Technology.
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<table>
<thead>
<tr>
<th>Molar composition</th>
<th>2.7 TPAOH: 46 SiO₂:5 Na₂O: Al₂O₃: 1.3 Trien: 2500 H₂O</th>
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<td>Temperature</td>
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<tr>
<td>Time</td>
<td>24 hrs</td>
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<table>
<thead>
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<th>Element</th>
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<td>Na</td>
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<tr>
<td>Al</td>
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<tr>
<td>Si</td>
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<td>Au</td>
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