

Microwave-assisted Isopropanol-to-Propylene Process with A Structured $\text{Co}_3\text{O}_4@\text{S1}/\text{SiC}$ Foam Catalyst

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ABSTRACT

Microwave has a significant promotion effect in gas-solid phase catalysis due to its unique advantages of heating process. This study introduces an innovative approach that combines microwave irradiation with a structured foam catalyst, $\text{Co}_3\text{O}_4@\text{S1}/\text{SiC}$, to enhance the isopropanol-to-propylene (ITP) process. The effectiveness of $\text{Co}_3\text{O}_4@\text{S1}/\text{SiC}$ foam was investigated under various reaction temperatures (50-200 °C) and space velocities (1200 h^{-1}), in comparison to $\text{Co}_3\text{O}_4@\text{S1}/\text{SiC}$ pellets. The $\text{Co}_3\text{O}_4@\text{S1}/\text{SiC}$ foam exhibited superior performance, achieving a higher selectivity towards propylene (approximately 99%) compared to $\text{Co}_3\text{O}_4@\text{S1}/\text{SiC}$ pellets (93%). This enhancement can be attributed to improved mass and heat transfers facilitated by the foam structure. $\text{Co}_3\text{O}_4@\text{S1}/\text{SiC}$ foam for ITP process has a milder condition with microwave irradiation (121 °C for 90% conversion) than conventional heating (184 °C for 90% conversion).

Keywords: Microwave, SiC foam, Silicalite-1, Structured catalyst, Isopropanol-to-propylene (ITP)

NONMENCLATURE

Abbreviations

ITP	Isopropanol to propylene
S1	Silicalite-1

Symbols

h	hours
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applications such as packaging materials, solvents, anti-freezing agents, and synthetic fibers [1]. Traditionally, the production of lower olefins heavily relies on energy-intensive methods such as steam cracking and fluid catalytic cracking of petroleum feedstock [2, 3]. However, the limited supply of petroleum feedstock has prompted the exploration of alternative and environmentally friendly processes for their production. Among these alternatives, the conversion of short-chain alcohols (such as methanol, ethanol, butanol, and isopropanol) to olefins has garnered significant interest due to their easy synthesis from diverse natural sources like coal, biomass, and natural gas. Zeolite and zeotype catalysts have emerged as promising candidates for the methanol or ethanol to olefins conversion reaction [2, 4, 5]. Nevertheless, achieving high product selectivity and maintaining catalyst stability (including resistance to coke formation, hydrothermal stability, and activity) pose significant challenges in these processes [6].

Microwave is an electromagnetic wave with a very strong penetrating power. This unique reverse heating has many advantages, which can improve the energy transfer efficiency and reduce the heating time, which is conducive to the control the heating process and eliminates the risk of material surface overheating[7, 8]. Microwave technology in the chemical industry has brilliant applications, such as biomass pyrolysis[9, 10], water treatment materials[11, 12], CO_2 methanation[13] [14], catalytic combustion[15, 16], etc. Cobalt oxide is an brilliant catalyst for numerous catalytic applications, such as hydrolysis, oxidation and dihydrogen, due to its unique properties, such as high reducibility, abundant surface oxygen species, and multiple oxidation states[17, 18]. Silicon carbide (SiC), as a new catalyst carrier with microwave-absorption capacity, has been attempted to be used in various reactions, such as fast pyrolysis[19],

1. INTRODUCTION

Lower olefins play a crucial role in the chemical industry due to their increasing demand in various

bio-oil upgrading[20], methane conversion[21], desulphurization[22], etc.

In this work, we aimed to investigate microwave-responsive Co-loaded structured catalysts for the preparation of propylene from isopropanol. The contrasting catalytic performance of structured catalysts can be observed when comparing conventional heating with microwave heating. Microwave intensification enables the reactants to achieve high conversion rates and exceptional selectivity towards propylene, even at low temperatures. This phenomenon highlights the significant advantages of microwave heating in enhancing the performance of structured catalysts. The mechanism is discussed by characterization techniques. Additionally, we also explored the effect of the structure of the carriers (foam and pellet) on the ITP process.

2. EXPERIMENT

2.1 Material and methods

2.1.1 Preparation of S1/SiC foams composites

In this experiment, cylindrical Silicon Carbide (SiC) foams, measuring 25 mm in diameter and 50 mm in height with 2 mm open-cell diameter, served as the catalyst support. To prepare the synthesis solution, deionized (DI) water, sodium hydroxide (NaOH), and tetrapropylammonium bromide (TPABr) were combined and stirred for 15 min, leading to a molar composition ratio of Si: TPABr: NaOH: H₂O = 2.5:0.15:0.3:75. The Si component was sourced from SiC foam's residual silicon. The hydrothermal synthesis was carried out at 175 °C over 72 h for complete reaction. The S1/SiC composites were then processed by boiling in DI water to remove contaminants, and then dried at 100 °C and calcined at 600 °C for 6 h in the presence of air.

2.1.2 Synthesis of structured Co₃O₄@S1/SiC foams, Co₃O₄@SiC foams and Co₃O₄@S1/SiC pellets.

To synthesize Co₃O₄@S1/SiC foam composites procedure involved placing S1/SiC foam in 80 ml solution of 10 mmol cobalt nitrate hexahydrate, 20 mmol Ammonium fluoride, and 20 mmol urea. The autoclave was sealed and subjected to a 50-hour hydrothermal synthesis process at 95 °C. Post-synthesis, the catalyst was rinsed with DI water, then dried at 100 °C and calcined at 400 °C for 2 h. The cobalt deposition on the S1/SiC foam was found to be approximately 2.5 wt%, ascertained by comparing the weight of the catalyst before and after the synthesis. For control purposes, a Co₃O₄@SiC foam was synthesized using the same method, replacing S1/SiC with SiC.

The experiment was also designed to produce Co₃O₄@S1/SiC pellet catalysts. Silicalite-1 (S1) was synthesized via hydrothermal process using tetraethyl orthosilicate (TEOS), sodium hydroxide (NaOH), tetrapropylammonium hydroxide (TPAOH), and water, in a ratio of 2.5 : 3 : 0.15 : 75. Following a similar method, Co₃O₄@S1 was created and then calcined to stabilize its structure. The Co₃O₄@S1 powder was blended with SiC pellets to attain roughly 50 wt% of cobalt content. This final product was compared to Co₃O₄@S1/SiC foam catalysts.

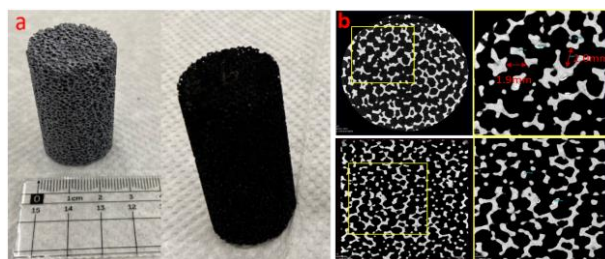


Fig. 1. a) the pictures of bare SiC (left) and Co₃O₄@S1/SiC foam (right); b) the CT scan image of SiC foam

2.2 Microwave-assisted catalytic ITP process

The experiment set-up was purposed for testing either Co₃O₄@S1/SiC foam, Co₃O₄@SiC foam or Co₃O₄@S1/SiC pellets, with the latter possessing a size of approximately 2 mm and a total volume of 25 ml. A graphical depiction of the foam catalysts' size and structure can be observed in Fig.1. The Isopropanol Thermal Process (ITP) was conducted at atmospheric pressure across a varied temperature range, extending from 50 to 300 °C. Infrared measurements provided real-time temperature data for the catalyst bed during the process. In each test, isopropanol vapor, with a concentration of about 38 mg l⁻¹, was generated using a bubbler in a cold-water bath maintained by the cooling system. Nitrogen served as the carrier gas, feeding the reactor. A feed flow rate of 500 ml min⁻¹ was maintained in the system. This corresponded to a gas volume hourly space velocity of 1200 h⁻¹. A parallel control test was performed under identical conditions using bare SiC and S1/SiC foam exposed to microwave radiation.

3. RESULTS AND DISCUSSION

3.1 Temperature rising test in microwave

The temperature rising test under microwave heating was carried out in order to test the microwave-absorbing properties of SiC and S1/SiC foam. In a very short time (less than 2 minutes), the temperature can exceed 300 °C (as shown in Fig. 2). Compared with SiC, S1/SiC has better microwave absorbing ability with the same microwave radiation power. That is because S1 modified the morphology of SiC foam when it grew on the SiC surface, which improved the microwave radiation absorption with more reflection and refraction on the support surface. SiC foam has excellent thermal conductivity, which is attributed to material intrinsic properties and spatial structure. The CT scanning of the internal structure of SiC is shown in Fig 1b. It can be seen that the spatial distribution of the SiC skeleton is relatively uniform, which is conducive to homogeneous heat transfer.

3.2 Properties of materials

Fig. 3.a) presents an in-depth view of the bare Silicon Carbide (SiC) foam support, revealing that its surface is embedded within a silicon (Si) core. The abundance of Si infiltrating the SiC support matrix during the hydrothermal synthesis process served a dual purpose. It not only provided the Si source for zeolite crystallization but also facilitated a robust interaction between the support and the gel. Further micrographic observations of the S1/SiC and Co₃O₄@S1/SiC composite are displayed in Fig. 3.a) a3 and a5. These images elucidate the development of a uniform S1 coating on the SiC foam support, a factor instrumental in promoting the subsequent growth of Co₃O₄ during the hydrothermal synthesis process. Supplementing the visual evidence, a SEM-EDS analysis was conducted to scrutinize the properties of the resulting composites. The

findings, demonstrated in Fig. 3.b), show the formation of a uniform and dense coating of flower-like Co₃O₄ structures on the surface of the S1/SiC. The analysis confirmed that the hydrothermal synthesis process

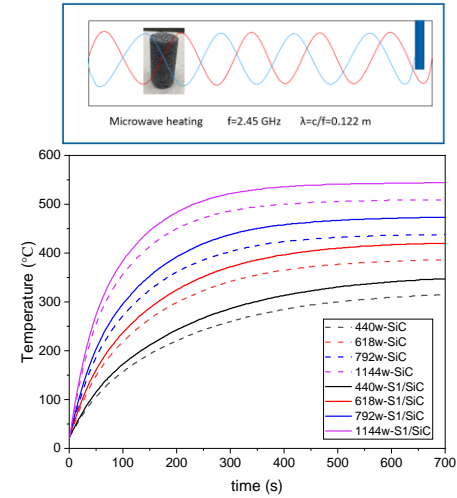


Fig. 2. Simulation diagram of Microwave heating (above) and microwave absorption performance (below) of bare SiC foam and Silicalite-1/SiC (S1/SiC)

successfully led to the desired composite's formation.

X-Ray Diffraction (XRD) analysis was utilized to explore the components of the S1/SiC composite, which includes cobalt oxide, silicalite-1 (S1), Silicon Carbide (SiC), and Silicon (Si), as shown in Fig. 3.d). The results from this analysis were corroborated by SEM-EDS mapping of the Co₃O₄@S1/SiC composite, ensuring reliability in the findings

High-Resolution Transmission Electron Microscopy (HRTEM) was employed for an in-depth inspection of Co₃O₄@S1/SiC, as demonstrated in Fig. 3 c). It unveiled that the unique flower-like structure of the cobalt phase was, in fact, composed of cobalt oxide nanocrystals. The

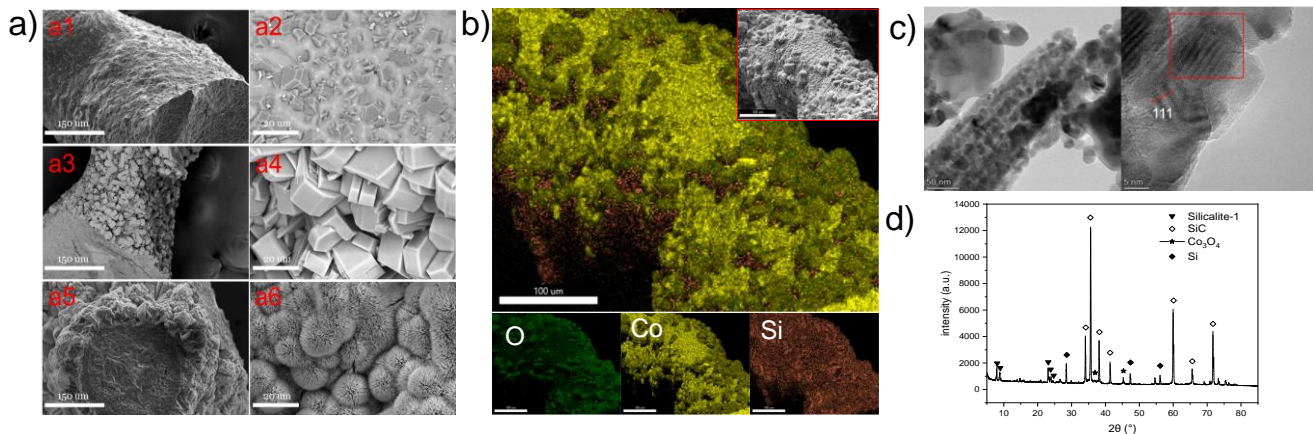


Fig. 3. a) SEM micrographs of (a1, a2) the bare SiC foam, (a3, a4) S1/SiC and (a5, a6) Co₃O₄@S1/SiC with different magnifications; b) EDS-maps of Co₃O₄@S1/SiC; c) HRTEM micrographs of Co₃O₄@S1/SiC; d) XRD patterns of the ground Co₃O₄@S1/SiC catalyst.

cobalt oxide demonstrated a face-centered cubic structure with a lattice constant of $a = 0.48$ nm. This confirmed the crystalline phase as Co_3O_4 spinel. The findings from the HRTEM analysis were congruent with the XRD results, reinforcing the consistency and accuracy of the experimental data.

The textural characteristics of the bare SiC and S1/SiC composite were investigated using N_2 physisorption analysis (Fig. 4.a). The SiC foam demonstrated non-porosity with a relatively low surface area of $0.61 \text{ m}^2 \text{ g}^{-1}$. On the contrary, the S1/SiC composite showed a considerably larger BET surface area of $29.95 \text{ m}^2 \text{ g}^{-1}$. It was evident that the initial crystallization of the Co phases occurred within the S1 framework, followed by the subsequent coverage of the S1 coating by Co_3O_4 . These findings were consistent with the results obtained from the XRD, SEM, and HRTEM analyses, thereby validating the experimental observations and conclusions.

velocity. It was found that the conversion of isopropanol could reach nearly 99%, increasing with temperature.

The catalytic activity of various materials, including bare SiC, S1/SiC, Co_3O_4 @SiC, and Co_3O_4 @S1/SiC, was meticulously evaluated under microwave irradiation as depicted in Fig.4.b). Among them, Co_3O_4 @S1/SiC emerged as notably thermally stable and demonstrated superior performance in the dehydration reaction. This assessment was conducted across a broad temperature spectrum, spanning from 50 to 300 °C, under atmospheric pressure and at an almost constant space velocity. A remarkable observation from this evaluation was that the conversion rate of isopropanol surged with increasing temperature, eventually nearing a staggering 99%. This impressive conversion efficiency underscores the potency of the Co_3O_4 @S1/SiC catalyst under the given conditions, showcasing its potential for high-performance applications. The findings also hint at the potential of fine-tuning these variables to optimize catalytic performance further and Co_3O_4 has a brilliant

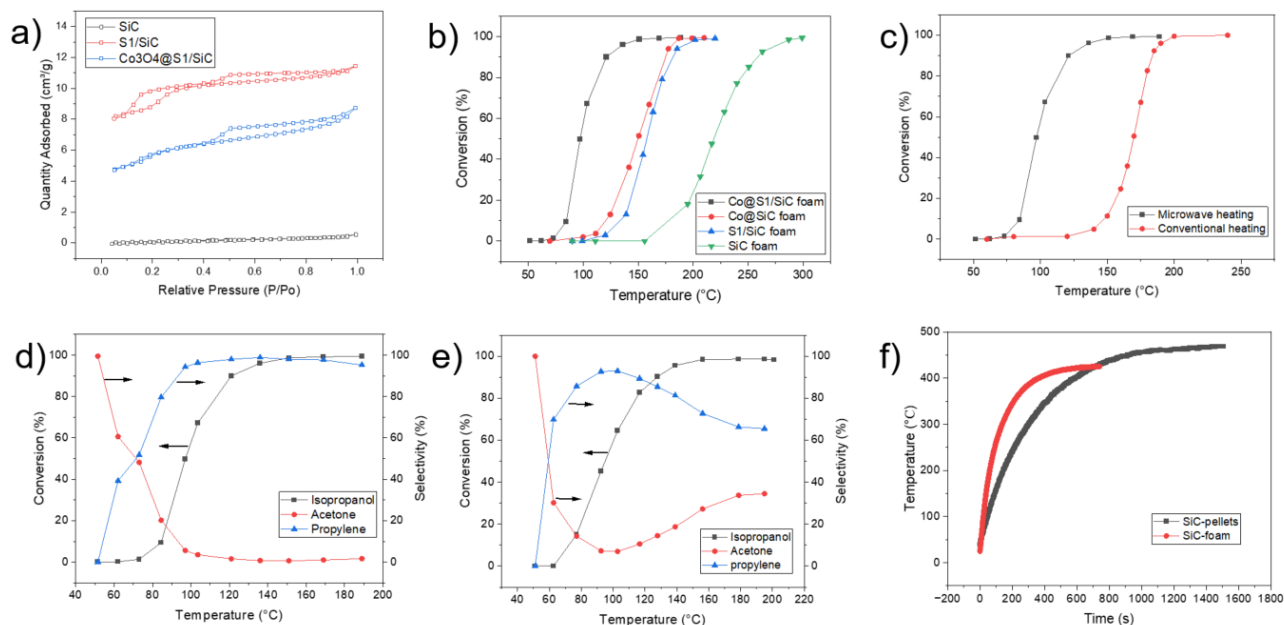


Fig. 4. a) N_2 adsorption/desorption isotherms of the SiC, S1/SiC and Co_3O_4 @S1/SiC composites; b) The conversion of Isopropanol dehydration process over SiC, S1/SiC Co_3O_4 @SiC and Co_3O_4 @S1/SiC composites; c) The comparison of conversion of Isopropanol on Co_3O_4 @S1/SiC under microwave and conventional heating; d) The conversion and selectivity for ITP process on Co_3O_4 @S1/SiC foam and e) on Co_3O_4 @S1/SiC pellets under microwave heating; f) Microwave absorption performance of SiC foam and SiC pellets.

3.3 Catalytic ITP process

Here the catalytic activity has been evaluated over bare SiC, S1/SiC, Co_3O_4 @SiC and Co_3O_4 @S1/SiC catalysts by microwave, as shown in Fig.4.b). which was thermally stable and showed its best performance in the dehydration reaction. The catalytic activity was evaluated in a wide range of temperatures (50–300 °C) at atmospheric pressure with almost constant space

prospect for the dehydration of isopropanol. Second support S1 also played an important role in conversion, not only because it expands the surface area of the carrier, but also improves the microwave absorption capacity and the adsorption of organic gases, which was confirmed from Fig.2 and Fig. 4.a).

In a comparative study, the performance of Co_3O_4 @S1/SiC foam composites was evaluated under microwave-assisted catalysis and conventional heating.

As depicted in Fig. 4.c), the process monitored the catalytic dehydration of isopropanol over $\text{Co}_3\text{O}_4@\text{S1}/\text{SiC}$, with the reaction temperatures corresponding to isopropanol conversion rates of 50% and 90% (T_{50} and T_{90}) and propylene selectivity rates of 50% and 90% (S_{50} and S_{90}) being the comparison metrics, as summarized in Table 1. Under conventional heating, T_{50} , T_{90} , S_{50} and S_{90} values were observed at 170 °C, 184 °C, 152 °C, and 172 °C, respectively. However, when subjected to microwave heating, these key performance indicators shifted to lower temperatures, underlining the microwave's effectiveness in facilitating the catalytic reaction. In essence, microwave heating accelerated the conversion of isopropanol, proving more efficient even at lower, milder temperatures. This highlights the potential for microwave-assisted catalysis as a more energy-efficient and productive approach in such chemical transformations.

Table 1 Relevant activity data of catalytic dehydration of isopropanol over $\text{Co}_3\text{O}_4@\text{S1}/\text{SiC}$ catalysts.

Catalyst $\text{Co}@S1/\text{SiC}$	$\text{C}_3\text{H}_8\text{O}$ Conversion		C_3H_6 selectivity	
	$T_{50}(\text{°C})$	$T_{90}(\text{°C})$	$S_{50}(\text{°C})$	$S_{90}(\text{°C})$
Microwave heating	97	121	72	93
Conventional heating	170	184	152	172

Fig. 4.d) shows the products of the isopropanol catalytic system including acetone and propylene. Acetone was the main product over the catalysts at very low temperatures. At temperatures around 50 °C, these catalysts facilitated isopropanol's dehydrogenation, resulting in the production of acetone. This outcome aligns with previous studies which demonstrated that Co_3O_4 nanoparticles could yield near-total selectivity (about 99%) for acetone at such relatively low temperatures. The $\text{Co}_3\text{O}_4@\text{S1}/\text{SiC}$ catalysts exhibited particular effectiveness for isopropanol dehydrogenation. This improved activity can likely be attributed to increased surface oxygen vacancies. As illustrated in Fig. 4.d), the dehydration of isopropanol (leading to propylene production) began at temperatures just over 50 °C and became dominant at around 80 °C. Once temperatures exceeded 120 °C, these catalysts displayed selectivity towards a total dehydration reaction, with propylene being the principal product. To summarize the findings represented in the

product condition curves, acetone serves as an intermediary product, while propylene is the end product in this catalytic transformation process.

Fig. 4. e) shows the conversion and the selectivity to acetone and propylene respectively over $\text{Co}_3\text{O}_4@\text{S1}/\text{SiC}$ pellets. The behavior of selectivity was different from foam catalysts, only up to 93% for propylene (~99% for foam catalysts). The conversion of isopropanol was also a little bit lower than foam catalysts (99%). This can be explained by the improved mass/heat transfer for foam catalysts, which was confirmed by the microwave absorption capacity between SiC foam and pellets as shown in Fig. 4.f).

4. CONCLUSIONS

This study introduces structured catalysts, $\text{Co}_3\text{O}_4@\text{S1}/\text{SiC}$ foam, as an effective solution for the conversion of isopropanol into propylene. Utilizing microwave irradiation, this innovative method shows significant improvements over conventional processes using packed $\text{Co}_3\text{O}_4@\text{S1}/\text{SiC}$ pellets. The benefits of this methodology are primarily attributed to two key factors. Firstly, the SiC foam's interaction with the microwave radiation ensures rapid and uniform heating of the catalyst. Secondly, the SiC foam's open-cell structure and the $\text{Co}_3\text{O}_4@\text{S1}$'s thin coating layer enhance both global and local mass transfers. Furthermore, when compared with traditional heating processes, the microwave-assisted procedure required lower temperatures to achieve equivalent conversion rates and propylene yields. These advancements indicate the promising potential of microwave-absorbing structured catalysts for process intensification, paving the way for more efficient chemical synthesis under microwave irradiation.

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DECLARATION OF INTEREST STATEMENT

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper. All authors read and approved the final manuscript.

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