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Research Article

The effect of different surface functionalization of SBA-15 catalysts on the production of C_{16} bio-aviation fuel precursor

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Abstract. The increasing global demand for sustainable aviation fuels has driven extensive research on developing efficient heterogeneous catalysts. This study investigates the effect of different surface functionalization methods of mesoporous SBA-15 on its catalytic activity for the production of a C_{16} precursor of bio-aviation fuel. The SBA-15 surfaces were modified by two acid functionalization routes, namely sulfonation and sulfation, to enhance its surface acidity and catalytic activity. Sulfonation was carried out using 3-mercaptopropyltrimethoxysilane (MPTMS) followed by oxidation to obtain the SO_3H–SBA-15 catalyst containing sulfonic acid groups (–SO_3H), while sulfation using ammonium sulfate as a precursor produced the SO_4–SBA-15 catalyst containing sulfate groups (SO_4^{2-}). Both catalysts were characterized using NH_3-TPD and acid-base titration to quantify the total acidity. The catalytic performance was evaluated through hydroxyalkylation-alkylation (HAA) reaction between 2-methylfuran (2-MF) and methyl isobutyl ketone (MIBK) to synthesize a C_{16} bio-aviation fuel precursor, 5,5′-(4-methylpentane-2,2-diyl) bis(2-methylfuran) abbreviated as MPM. The results revealed that both modification methods effectively increased the total acid of SBA-15. However, the sulfated SBA-15 catalyst exhibited superior catalytic activity and stronger acid strength than the sulfonated one due to formation of more acid sites on its surface. Therefore, the sulfation route was identified as a more effective strategy for developing highly active solid acid catalysts. This research demonstrates the superior properties of sulfated mesoporous SBA-15 as a promising and sustainable heterogenous catalyst for converting biomass-derived platform chemicals into advanced C_{16} bio-aviation fuel precursors.

Keywords: SBA-15, sulfation, sulfonation, hydroxyalkylation-alkylation (HAA) reaction, C_{16} bio-aviation fuel

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1. Introduction

The transportation sectors including aviation industry generally use fossil fuels as the main energy source (Lindstad *et al.*, 2023; Muttaqii *et al.*, 2025). However, its use causes many environmental problems including acid rain, global warming and greenhouse gas emissions (Pal *et al.*, 2023; Wang & Tao, 2016; Yoro & Daramola, 2020). The aviation industry alone contributes significantly to 2—3 % of global CO_2 emissions (Lee *et al.*, 2021; Mohd Hasan Wong *et al.*, 2024; Wang *et al.*, 2024). The growing need for fossil fuels along with population growth has led researchers to develop alternative energy such as the use of biomass (Adany *et al.*, 2025; Al Muttaqii *et al.*, 2024; Jayabal, 2024; Manjate *et al.*, 2024; Hadiyanto *et al.*, 2014). One of the modern methods of converting biomass has been done through the hydroxyalkylation-alkylation (HAA) reaction of lignocellulose derivatives compound namely 2-methyl furan (2-MF) which is a key product for producing renewable diesel and bio-aviation fuel (Corma *et al.*, 2011; Yati *et al.*, 2015). 2-MF were reacted with aldehyde or ketone to produce C_{12} to C_{20} compound (Li *et al.*, 2013; Yang *et al.*, 2013; Yati *et al.*, 2024).

Among various aldehyde or ketone, methyl isobutyl ketone (MIBK), which can also be sourced from lignocellulosic biomass, have emerged as promising platform molecules to produce bio-aviation fuels (Che *et al.*, 2022; Chen *et al.*, 2025; Teng *et al.*, 2016). Our previous study demonstrated for the first time that a novel C_{16} bio-aviation fuel precursor, 5,5'-(4-methylpentane-2,2-diyl) bis(2-methylfuran) abbreviated as MPM, can be produced via HAA of 2-MF with MIBK using sulfated Santa Barbara Amorphous-15 (SBA-15) catalyst (Salsabila *et al.*, 2025). The advantage of using MIBK is that it has a branched framework of carbon chain, α-H, and carbonyl functional group which makes

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it favorable for the formation of long branched carbon alkane with a low freezing point, which is essential in fuel properties (Feng *et al*., 2021; Watanasiri *et al*., 2023).

The SBA-15 is a type of hexagonal structure of mesoporous silica which was first synthesized by Zhao *et al*. (Zhao *et al*., 1998). SBA-15 has been modified with various methods to enhance its properties as a heterogeneous catalyst to increase the catalytic activity (Liang *et al*., 2024; Ridwan *et al*., 2024; Singh *et al*., 2018). The sulfation of SBA-15 that was first conducted in our study has proven to be effective in increasing the acidity of the catalyst and significantly improve a novel bio-aviation fuel production (Salsabila *et al*., 2025). Most common strategy to increase the acidity of the catalyst is sulfonation to incorporate sulfonic acid groups (Gebresillase *et al*., 2018; Saini *et al*., 2024). Sulfonation on the carbon surface has been shown to increases the active site on the carbon surface and produces a very stable solid material having high active acid density (Fonseca *et al*., 2022; Yang *et al*., 2021). Both sulfonation and sulfation method can produce higher active side on the catalyst. This can cause more intensive contact between reactants with the acid center of the catalyst, resulting in better catalytic activity (Busca & Gervasini, 2020; Vogt & Weckhuysen, 2022; Wang *et al*., 2022).

To further elucidate the effect of different acid functionalities, a comparative overview of SO_3H- and SO_4-functionalized catalysts previously reported for bio-aviation fuel precursor production is summarized in Table 1. The contribution of this work is primarily material-based and application-driven. From a materials perspective, this study presents the first direct comparison between sulfonated and sulfated SBA-15 catalysts, enabling a systematic assessment of how different surface acid functionalities (sulfonic, -SO_3H versus sulphate, SO_4 group) affect catalyst acidity and performance. From an application standpoint, these catalysts are evaluated in the HAA reaction of 2-methylfuran and methyl isobutyl ketone, targeting the production of a C_{16} bio-aviation fuel precursor, a reaction system that has not been previously explored for comparative sulfonated and sulfated SBA-15 catalysts.

Notably, although sulfonation and sulfated-based catalysts have been individually explored for acid-catalyzed upgrading reactions, a direct comparison of sulfonated and sulfated SBA-15 in the HAA reaction for C_{16} bio-aviation jet fuel precursor production has not been previously reported. In this research, the SBA-15 was modified with two different methods of functionalization, sulfonation and sulfation to enhance the acidity of the catalyst. Sulfonation of SBA-15 to incorporate sulfonic acid groups (-SO_3H) was conducted using MPTMS precursor to produce SO_3H-SBA-15 catalyst. Meanwhile, the sulfation of SBA-15 to incorporate sulfate groups (SO_4) was conducted using ammonium sulfate precursor to produce SO_4-SBA-15 catalyst. The resulting catalysts were analyzed using NH_3-TPD and acid base titration. In order to study the influence of different functionalization methods, both catalysts were then tested in a HAA reaction of 2-MF and MIBK to produce C_{16} bio-aviation fuel precursor to find out which catalyst has the best catalytic activity and effectiveness for bio-aviation fuel precursor production.

This study provides a comparative evaluation of two different surface functionalization methods, namely sulfonation and sulfation, on SBA-15 to systematically determine their influence on acidity and catalytic performance in the HAA reaction. The novelty of this work lies in the direct comparison of these two modification routes on the same SBA-15 framework for C_{16} bio-aviation fuel precursor synthesis, which has not been comprehensively reported in previous studies.

2. Experimental Section

2.1 Materials

Pluronic, P123 (average $M_n \approx 5800$, ≥99 %), hydrochloric acid fuming (HCl, 37 %), tetraethyl orthosilicate (TEOS, ≥99 %), 3-mercaptopropyltrimethoxysilane (MPTMS, 95%), hydrogen peroxide (H_2O_2, 30 %), ammonium sulfate (≥99 %), 2-methyl furan 2-MF (≥99 %), and methyl isobutyl ketone (MIBK, ≥99%) were acquired from Sigma Aldrich. Meanwhile, Sodium hydroxide (NaOH, ≥98 %) and phenolphthalein (PP, ≥99 %) were acquired from Merck. All reagents were used directly without treatment.

2.2 Preparation of SO_3H-SBA-15 catalyst

The SO_3H-SBA-15 catalyst was prepared following the modified procedure of Gabla *et al.* (2019). The sulfonic functionalization of the SBA-15 surface was achieved through a one-pot co-condensation approach, which was selected to ensure a homogeneous incorporation of sulfonic acid groups throughout the silica framework rather than surface-only functionalization. Initially, 4 g of Pluronic P-123 was completely dissolved in 106 mL of 2 M HCl solution at 25 °C, followed by heating the mixture to 40 °C under constant stirring. After complete dissolution, 4.19 mL of tetraethyl orthosilicate (TEOS) was added dropwise to allow for pre-hydrolysis for 45 minutes, a duration chosen to ensure partial hydrolysis while preventing premature condensation. Subsequently, 2.86 mL of (3-mercaptopropyl) trimethoxysilane (MPTMS) was introduced as the organosilane precursor, follow by 0.61 mL of H_2O_2 to promote in situ oxidation of thiol groups into sulfonic acid functionalities. The reaction mixture was continuously stirred at 40 °C for 24 hours to complete hydrolysis and condensation, then transferred to a Teflon-lined stainless-steel autoclave and aged hydrothermally at 130 °C for 24 hours. The resulting solid product was recovered by centrifugation, followed by extensive washing with ethanol to remove unreacted precursors. The surfactant template (P-123) was subsequently extracted using Soxhlet extraction with ethanol for 24 hours to ensure complete

Table 1
SO_3H- and SO_4- functionalized catalysts for bio-aviation fuel precursor production

Catalyst	Reactants	Ref.
SO_4-MOF-808	2-MF + 5-MFUR	(Atayde *et al*., 2024)
SO_4-Zirconia	2-MF + Butanal	(Yati *et al*., 2024)
SO_4-TiO_2	2MF + Furfural	(Yan *et al*., 2023)
Pr-SO_3H-SBA-15	Levulinic acid	(Paniagua *et al*., 2021)
SO_3H-C	Biomass	(Mateo *et al*., 2020)
SO_3H-Nb_2O_5	2MF +Furfural	(Chhabra & Krishnan, 2023)
SO_3H- and SO_4-SBA-15	2-MF + MIBK	This study

template removal. The obtained material was again centrifuged, washed several times with hot ethanol to eliminate residual organics, and dried in an oven at 120 °C for 4 hours. The resulting SO_3H-SBA-15 catalyst was stored in an airtight glass vial for further characterization and catalytic testing. This synthesis route provides a homogeneous distribution of sulfonic acid groups on the mesoporous silica framework, maintaining the ordered hexagonal pore structure characteristic of SBA-15 while introducing strong Brønsted acid sites beneficial for biomass-derived catalytic reactions.

2.3 Preparation of SO_4-SBA-15 catalyst

The SO_4–SBA-15 catalyst was synthesized following the modified procedures reported in our previous studies (Fadilah *et al*., 2023; Salsabila *et al*., 2025). The incorporation of sulfate groups onto the mesoporous silica surface was achieved through a wet impregnation method, which was selected for its simplicity and effectiveness in introducing surface-coordinated sulfate species. Initially, 4 g of Pluronic P-123 was dissolved in a 2 M HCl solution and deionized water mixture at a volume ratio of 1:4 under continuous stirring. Subsequently, 8.8 g of tetraethyl orthosilicate (TEOS) was added dropwise to the solution and stirred at 40 °C for 24 hours to facilitate hydrolysis and condensation processes. The resulting homogeneous gel was transferred into a Teflon-lined autoclave and subjected to hydrothermal treatment at 100 °C for 12 hours. The obtained solid product was recovered by centrifugation, thoroughly washed with deionized water several times, and then dried at 60 °C for 18 hours. The as-synthesized SBA-15 was calcined at 550 °C for 6 hours to remove the P-123 surfactant, producing a highly ordered mesoporous silica support.

For the sulfation step, 1 g of calcined SBA-15 was dispersed in 25 mL of 1 M ammonium sulfate solution and stirred at 25 °C for 4 hours to ensure uniform sulfate ion adsorption on the silica surface. The suspension was centrifuged, and the solid was dried at 100 °C for 18 hours, followed by calcination at 400 °C, which is sufficient to decompose ammonium sulfate and generate thermally stable sulfate species anchored to the silica framework without causing structural collapse. The final SO_4–SBA-15 catalyst was stored in a sealed glass vial prior to use. This synthesis method effectively introduces strong Brønsted and Lewis acid sites associated with coordinated sulfate species, which are known to enhance catalytic performance in acid-catalyzed reactions such as hydroxyalkylation and alkylation also biomass upgrading.

2.4 Characterization of the catalyst

The total acidity and acid site strength distribution of SO_3H–SBA-15 and SO_4–SBA-15 catalysts were determined using ammonia temperature-programmed desorption (NH_3-TPD) and acid–base titration methods. The NH_3-TPD measurements were performed using a Micromeritics Autochem II 2920 instrument to evaluate the nature and strength of acid sites on the catalyst surface. Prior to analysis, each catalyst sample was pretreated by heating at 350 °C for 1 hour under a continuous helium (He) flow to remove physisorbed moisture and impurities. After pretreatment, ammonia gas (5% NH_3 in He, v/v) was introduced into the system at 100 °C for 30 minutes to allow adsorption of NH_3 molecules onto the acid sites. The physisorbed ammonia was then purged with pure He for 30 minutes to ensure that only chemisorbed NH_3 remained. Subsequently, NH_3 desorption was conducted by heating the sample from 100 to 800 °C at a constant rate, and the desorbed NH_3 was monitored by a thermal conductivity detector (TCD). The desorption profile was used to distinguish between weak, medium, and strong acid sites, which reflect the Brønsted and Lewis acidity characteristics of each catalyst.

In addition to NH_3-TPD, the total acid amount of the catalysts was quantified by acid–base titration. Approximately 0.05 g of catalyst was dispersed in 3.75 mL of 0.1 M NaOH solution and stirred for 2 hours at 25 °C to neutralize the surface acid sites. After equilibration, the solid was separated by filtration, and the filtrate was titrated with 0.1 M HCl using three drops of phenolphthalein (PP) as an indicator. The disappearance of the pink color marked the titration endpoint. The total acidity was then calculated according to Equation (1):

$$\boldsymbol{Acidity\ (mmol\ g^{-1})} = \frac{\boldsymbol{n_{NaOH}}}{\boldsymbol{w}} \qquad (1)$$

where $\boldsymbol{n_{NaOH}}$ represents the amount of NaOH consumed (mmol) and $\boldsymbol{w}$ denotes the catalyst weight (g). This combined approach provides both qualitative and quantitative insights into the surface acidity of the catalysts. The correlation between NH_3-TPD and titration results confirms that SO_4-SBA-15 exhibits a higher density of strong acid sites than SO_3H–SBA-15, consistent with its superior catalytic performance in the hydroxyalkylation–alkylation (HAA) reaction.

Furthermore, the surface morphology and elemental composition of the catalysts were characterized using Field Emission Scanning Electron Microscopy (FE-SEM) equipped with Energy Dispersive X-ray Spectroscopy (EDS) and elemental mapping analysis. The measurements were carried out using a JEOL JIB-4610F instrument to observe the surface texture, particle uniformity, and elemental distribution of sulfur, silicon, and oxygen across the catalyst framework. This analysis provided information to confirm the successful incorporation of sulfate and sulfonate functional groups on the SBA-15 surface.

2.5 Hydroxyalkylation-alkylation (HAA) Reactions

Hydroxyalkylation-alkylation (HAA) reactions were performed using reflux condenser to maintain a constant reaction volume and prevent loss of volatile reactants, following the procedure reported by Salsabila *et al.* (2025). A mixture of 2-MF (4 mL, 0.0450 mol) and MIBK (1.4 mL, 0.0225 mol) (4 to 1 molar ratio) was put into a round flask and then added with 1.4 wt% of catalyst (50 mg). The mixture was refluxed at 90 °C for 24 hours with constant stirring. The HAA product was then separated from the catalyst by centrifugation. The liquid product was analyzed using gas chromatography-flame ion detector (GC-FID, HP-88 column). The oven temperature was initially set at 50 °C and then increased to 250 °C at a heating rate of 10 °C min^{-1}. The MIBK conversion (C), selectivity (S) and yield (Y) of MPM were calculated using equations 2 to 4.

$$\boldsymbol{C}\ (\%) = \frac{\boldsymbol{n_x}}{\boldsymbol{n_0}}\ \boldsymbol{x\ 100} \qquad (2)$$

$$\boldsymbol{S}\ (\%) = \frac{\boldsymbol{A_{MPM}}}{\boldsymbol{\Sigma A_{product}}}\ \boldsymbol{x\ 100} \qquad (3)$$

$$\boldsymbol{Y}\ (\%) = \frac{\boldsymbol{C}}{\boldsymbol{100}}\ \boldsymbol{x\ S} \qquad (4)$$

where n_x denotes the moles of MIBK consumed, n_0 represents the initial moles of MIBK, and $A_{(MPM)}$ and $A_{(product)}$ indicate the integrated peak areas of MPM and the total reaction products, respectively, as determined by GC-FID analysis.

3. Results and Discussions

In various catalytic reactions, intrinsic property of the catalyst such as acidity has become a key parameter which determines the catalytic activity (Yati *et al*., 2014; Yati, Ridwan, *et al*., 2024). SBA-15 has been used in various chemical reactions due to its outstanding performance including high BET surface area, ordered mesoporous structure, and excellent thermal stability as catalyst and catalyst support (Akbar *et al*., 2024). In this research, the SBA-15 surface was modified by functionalization using two different methods: sulfonation to incorporate sulfonic acid (SO_3H) groups and sulfation to incorporate sulfate (SO_4) groups in order to increase the total acid of catalyst, thereby enhancing the catalytic potential.

The acidity measurements were performed since acid strength and density play crucial roles in determining catalytic performance in condensation or hydroxyalkylation-alkylation (HAA) reactions. The acidity of the prepared catalyst was analyzed using NH_3-TPD and acid base titration method. Fig. 1 showed the NH_3-TPD result of SO_3H-SBA-15 and SO_4-SBA-15 catalysts, and the acid amount was presented in Table 1. The combination of these two techniques provided complementary information: NH_3-TPD reveals the strength distribution of acid sites, while acid–base titration gives quantitative data on total acidity. These analyses are essential to correlate the acid properties of the catalysts with their catalytic performance in the HAA reaction.

As can be seen in Fig. 1, both SO_3H-SBA-15 and SO_4-SBA-15 catalysts exhibit ammonia desorption peaks at high temperatures (above 350 °C), confirming the presence of strong acid sites on their surfaces. The desorption of NH_3 at these elevated temperatures indicates that both materials possess strong acid sites, which are crucial for promoting the HAA reaction. The SO_4–SBA-15 catalyst displays a significantly larger and sharper desorption peak centered at around 500 °C, suggesting a higher acid site density and stronger acid strength compared to SO_3H–SBA-15, whose desorption maximum appears at approximately 410 °C.

The enhanced acidity of SO_4–SBA-15 can be attributed to the presence of coordinated sulfate species bonded to the silica framework, forming strong S=O and S–O–Si bonds that generate both Brønsted (proton donor) and Lewis (electron acceptor) acid sites (Sekewael *et al*., 2022). Meanwhile, the relatively weaker peak intensity of SO_3H–SBA-15 reflects a lower number of accessible acid sites and moderate acid strength derived mainly from surface-grafted –SO_3H groups. This difference in acid site concentration and strength is consistent with the catalytic results, where SO_4–SBA-15 exhibited higher MIBK conversion and MPM yield compared to SO_3H–SBA-15. The strong correlation between acidity and catalytic performance highlights the crucial role of surface acid density in determining the efficiency of biomass-derived condensation reactions.

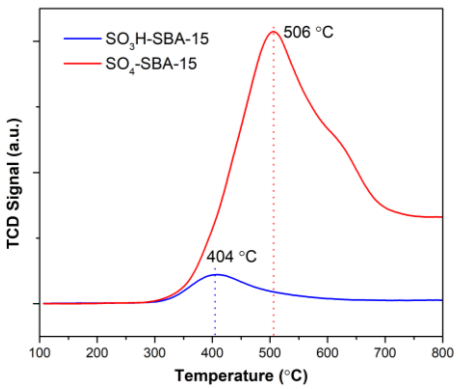

Fig. 1 NH_3-TPD result of SO_3H-SBA-15 and SO_4-SBA-15, showing higher acid strength and acidity for SO_4-SBA-15 (506 °C, 8.9 mmol g^{-1}) compared to SO_3H-SBA-15 (404 °C, 5.3 mmol g^{-1}).

Table 2
Acidity amount of the modified SBA-15 catalysts

Catalyst	Acidity amount (mmol g^{-1})	
	(a)	(b)
SO_3H-SBA-15	5.3	3.8
SO_4-SBA-15	8.9	7.6

*Acidity amounts were calculated by: (a) NH_3-TPD and (b) acid-base titration

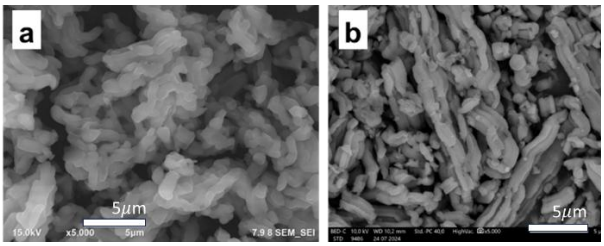

Fig. 2 SEM images of (a) SO_3H-SBA-15 and (b) SO_4-SBA-15 catalysts at a magnification of 5000x.

As presented in Table 2, based on ammonia TPD, SO_3H-SBA-15 catalyst has acidity of 5.3 mmol/g. Meanwhile, SO_4-SBA-15 catalyst has acidity of 8.9 mmol/g. The acid amount of the catalysts was also measured using acid base titration method and the result was in agreement with the ammonia TPD result. The SO_4-SBA-15 catalyst has higher acidity (7.6 mmol/g) compared to SO_3H-SBA-15 catalyst (3.8 mmol/g). It was confirmed that functionalization method to incorporate the sulfate groups into SBA-15 catalyst has increased the acidity more effectively compared to sulfonation method.

To further understand the physicochemical properties of the synthesized catalysts, the surface morphology of both catalysts was examined using scanning electron microscopy (SEM), as shown in Fig. 2. The SEM micrographs revealed that both SO_3H–SBA-15 and SO_4–SBA-15 catalysts maintained the typical rod-like morphology characteristic of the parent SBA-15. This observation suggests that the ordered mesostructured of SBA-15 remained intact after chemical modification, indicating that the sulfonation and sulfation treatments did not cause framework collapse or structural deformation. The preservation of the SBA-15 shape confirms its high structural stability under the functionalization conditions applied.

The successful incorporation and distribution of sulfur-containing functional groups were further verified through energy-dispersive X-ray spectroscopy (EDX) and elemental mapping analyses, as shown in Figs. 3 and 4. The EDX spectrum of SO_3H–SBA-15 (Fig. 3a) exhibited characteristic peaks of Si, O, and S elements, confirming the successful introduction of sulfonic groups. Quantitative analysis revealed a sulfur content of approximately 6 wt%. The corresponding mapping images (Fig. 4b) demonstrated a uniform distribution of Si, O, and S, implying that sulfur atoms were homogeneously dispersed across the catalyst surface without forming agglomerates. Similarly, the EDX analysis of SO_4–SBA-15 (Fig. 3b) confirmed the presence of sulfur species derived from sulfate groups. The sulfur content was measured at 2 wt%. The mapping images

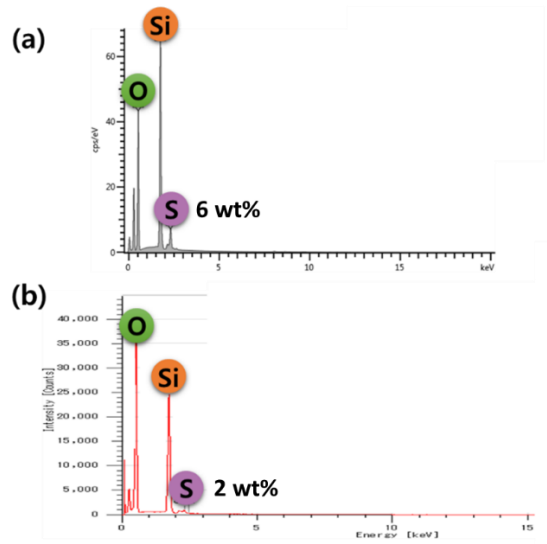

Fig. 3 EDX spectra of (a) SO_3H-SBA-15 and (b) SO_4-SBA-15 catalysts.

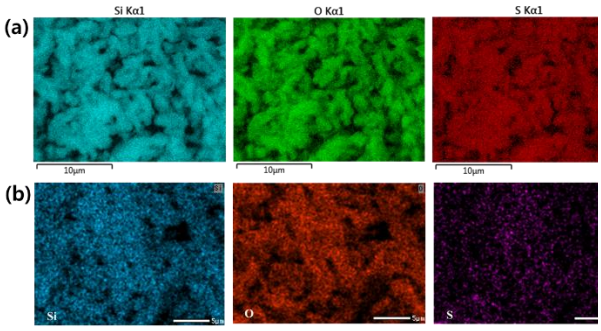

Fig. 4 Elemental mapping images of (a) SO_3H-SBA-15 and (b) SO_4-SBA-15 catalysts. Part (b) is adapted from our previous publication (Salsabila *et al.*, 2025), reproduced with permission from Elsevier.

(Fig. 4b) revealed that sulfur was evenly distributed along the silica framework, confirming the homogeneous grafting of sulfate groups on the SBA-15 surface. This even elemental distribution ensures accessible acid sites and contributes to catalytic uniformity during reactions. Overall, the SEM, EDX, and mapping analyses collectively demonstrate that both surface functionalization methods were successful in incorporating sulfur species into the SBA-15 framework while maintaining the mesostructured integrity. To investigate the effect of different surface modification methods on the catalytic activity of SBA-15, both SO_3H-SBA-15 and SO_4-SBA-15 catalysts were then used in HAA reaction of 2-MF and MIBK to produce C_{16} bio-aviation fuel precursor (MPM). The reaction mechanism between 2-MF and MIBK was illustrated in Fig. 5.

As seen in Fig. 5, HAA reaction between 2-MF and MIBK required strong acid site (H^+) for reaction to proceed and formed MPM. Fig. 5 illustrates the proposed reaction mechanism of the hydroxyalkylation and alkylation (HAA) process between 2-methylfuran (2-MF) and methyl isobutyl ketone (MIBK) over acid catalysts. The reaction proceeds through two main consecutive steps: hydroxyalkylation and alkylation. In the first step, the carbonyl group of MIBK is protonated by the Brønsted acid sites of the catalyst, increasing its electrophilicity and allowing nucleophilic attack by the C5 position of 2-MF to form a β-hydroxy intermediate (1a). This

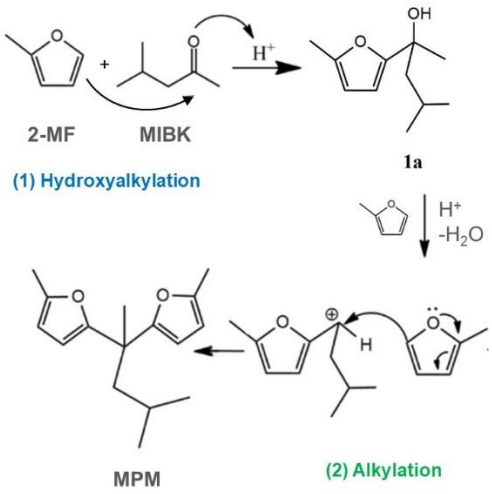

Fig. 5 Hydroxyalkylation–alkylation (HAA) of 2-methylfuran with MIBK to produce MPM via a two-step pathway involving hydroxyalkylation and subsequent alkylation of 2-methylfuran and MIBK.

step is referred to as hydroxyalkylation. In the subsequent alkylation step, the intermediate undergoes acid-catalyzed dehydration, producing an α,β-unsaturated carbonyl compound that can further react with another 2-MF molecule to yield the desired C_{16} product, 2,2'-(4-methylpentane-2,2-diyl)bis(5-methylfuran) (MPM) (Salsabila *et al.*, 2025). The Brønsted acid sites promote both the protonation and dehydration steps, while the Lewis acid sites assist in the activation of the carbonyl group and stabilization of the carbocation intermediates, thus facilitating C–C bond formation. The synergistic interaction between Brønsted and Lewis acid sites plays a key role in enhancing catalytic activity and selectivity toward MPM formation.

The appearance of liquid products of the HAA reaction result between 2-MF and MIBK using SO_3H-SBA-15 and SO_4-SBA-15 catalysts were depicted in Fig. 6. After 24 hours of reaction time at 90 °C using SO_3H-SBA-15 catalyst, the liquid product has light brown color. Meanwhile, when using SO_4-SBA-15 as catalysts under the same reaction condition, darker

Fig. 6 Liquid products of the HAA reaction of 2-MF and MIBK using SO_3H-SBA-15 and SO_4-SBA-15 catalysts.

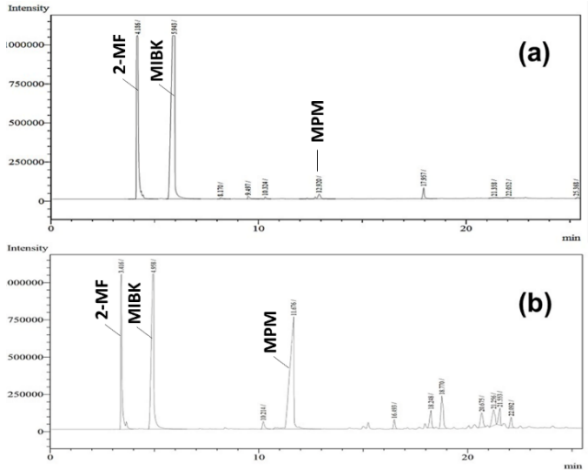

Fig. 7. GC-FID chromatograms of liquid products obtained from the HAA reaction using (a) SO_3H-SBA-15 and (b) SO_4-SBA-15 catalysts, showing the presence of unreacted 2-methylfuran (2-MF), MIBK, and the target product MPM.

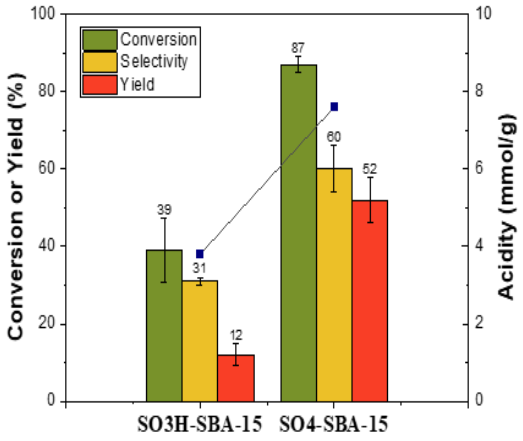

Fig. 8. HAA reaction between 2-methylfuran (2-MF, 0.045 mmol) and MIBK (0.0225 mmol) using SO_3H-SBA-15 and SO_4-SBA-15 catalysts with a 4:1 molar ratio of 2-MF to MIBK at 90 °C for 24 h and a catalyst loading of 50 mg.

liquid product was produced. Both liquid products were then analyzed using GC-FID for quantification.

The liquid products obtained from the HAA reaction using SO_3H-SBA-15 and SO_4-SBA-15 catalysts were analyzed using GC-FID. The chromatogram was presented in Fig. 7, and the calculation result was presented in Fig. 8. through the inclusion of error bars representing standard deviations obtained from triplicate experiments (n = 3). The SO_3H-SBA-15 catalyst consistently exhibits higher average conversion, selectivity, and yield compared to SO_4-SBA-15. Importantly, the magnitude of the performance difference between the two catalysts is significantly larger than the corresponding standard deviations, indicating that the observed enhancement is systematic and reproducible rather than arising from experimental uncertainty. The relatively small error bars for both catalysts further demonstrate good repeatability and experimental reliability.

As seen in Fig. 7a, the chromatogram of SO_3H-SBA-15 displays two dominant peaks at retention times around minute 4 and 5, corresponding to unreacted MIBK and 2-MF, with minor main product peaks appearing at retention time around minute 12. This indicates that the SO_3H-SBA-15 catalyst exhibited limited catalytic activity, which is consistent with the lower MIBK conversion (39%) and MPM yield (12%) presented in Fig. 8. In contrast, the chromatogram of SO_4-SBA-15 (Fig. 7b) shows a marked decrease in the intensity of the reactant peaks and the emergence of major peaks between 11 and 22 minutes, corresponding to the formation of heavier condensation products such as MPM and other side products. These new peaks confirm the higher catalytic performance of SO_4-SBA-15, as reflected by its higher MIBK conversion (87%) and MPM yield (52%) shown in Fig. 8. The enhanced product yield marked by an increased main product peak intensity is directly related to the higher total acidity of SO_4-SBA-15, which effectively promote both condensation and dehydration reactions in the HAA process (Sudarsanam *et al.*, 2019) (Zhang *et al.*, 2023). The total acid amount of the catalyst greatly influences the catalytic activity (Osazuwa & Ng, 2025). As the acidity of the catalyst increased, activity was also increased. It was also worth noting that SO_4-SBA-15 catalyst performed two times higher selectivity (60%) compared to SO_4-SBA-15 (31%) due to its higher acid value.

The results in this research showed that functionalization of SBA-15 is a crucial step to enhance the catalytic activity of SBA-15 catalyst. It was found that functionalization of SBA-15 with SO_4 groups was far more efficient and effective for the high production of bio-aviation fuel precursor compared to functionalization with SO_3H groups. To place the present results in the context of state-of-the-art solid acid catalysts for HAA of 2-methylfuran with aldehydes or ketones, the catalytic performances of SO_4-SBA-15 in this study were compared with recent studies reported in the literature. The SO_4-SBA-15 catalyst exhibited a competitive performance, producing a C_{16} product with a yield of 52% when MIBK was used as the ketone substrate which is comparable to those reported catalysts such as SO_4-ZrO_2 (Yati *et al.*, 2024) and Amberlyst-15 (Lin *et al.*, 2024), indicating efficient utilization of acid sites. Although this yield is lower when compared to SO_4-MOF-808, H-Y Zeolite and Nafion-212, their study employed highly reactive aldehydes or shorter ketone such as 5-hydroxymethylfurfural (Atayde *et al.*, 2024), furfural (Siakpebru *et al.*, 2025), and acetone (Li *et al.*, 2013) which generally favor the HAA reaction This trend is consistent with previous findings (Salsabila *et al.*, 2025) that ketones, particularly sterically hindered and longer carbon chain ketones such as MIBK, exhibit lower electrophilicity and reduced reactivity in HAA reactions compared to aldehydes or shorter ketone, making the HAA reaction more challenging, nevertheless, SO_4-SBA-15 maintained reasonable catalytic efficiency while selectively generating higher carbon-number (C_{16}) fuel precursors.

Beyond yield-based comparison, it is important to consider the carbon number distribution of the products, as this parameter directly influences fuel properties such as energy density and suitability for aviation fuel applications. In this context, the present study demonstrates a clear advantage by selectively producing higher carbon number products (C_{16}) from 2-methylfuran and MIBK, whereas several reported systems predominantly yield lower carbon number compounds (C_{11}–C_{14}). Higher carbon number hydrocarbons are generally associated with higher gravimetric and volumetric energy densities, improved combustion characteristics, and greater compatibility with conventional jet-fuel specifications. Hydrocarbons in the C_{12}–C_{16} range are particularly desirable for sustainable aviation fuel due to their favorable balance between

energy content and cold-flow properties. The ability of SO_4–SBA-15 to promote the formation of C_{16} products from a relatively unreactive ketone feedstock highlights the effectiveness of this catalyst system in enabling carbon-chain growth toward high energy density bio-aviation fuel. This selectivity toward higher carbon numbers represents a meaningful contribution, as it demonstrates a pathway to enhance fuel quality. Consequently, when evaluated from the perspective of fuel-relevant molecular characteristics, the present results compare favorably with previously reported solid acid catalytic systems.

4. Conclusions

The SBA-15 surface functionalization was successfully carried out using two different methods, sulfonation and sulfation, to enhance its acidity for catalytic application in the HAA reaction of 2-MF and MIBK. The experimental results demonstrated that the sulfation method led to a higher total acidity of SBA-15, as evidence by acidity measurements. Under the investigated reaction conditions, the sulfated-SBA-15 catalyst exhibited higher catalytic performance, resulting in increased conversion and yield of the C_{16} bio-aviation fuel precursor compared to the sulfonated-SBA-15. These findings demonstrate that sulfation is an effective strategy for enhancing the catalytic activity of SBA-15 in HAA reactions.

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