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ISETC2025

2ND INTERNATIONAL SCIENCE,
ENGINEERING AND TECHNOLOGY
COLLOQUIUM



THE POWER OF SCIENTIFIC INNOVATION

INTELLIGENT SOLUTIONS FOR A
SUSTAINABLE WORLD

ORGANIZED BY:
FACULTY OF APPLIED SCIENCES
UNIVERSITI TEKNOLOGI MARA
PERAK BRANCH, TAPAH CAMPUS
MALAYSIA

CO-ORGANIZED BY:



**2nd International Science, Engineering and Technology Colloquium
(ISETC 2025)**

Faculty of Applied Sciences,
Universiti Teknologi MARA,
Perak Branch, Tapah Campus,
35400 Tapah Road, Perak, Malaysia.

Colloquium date: 9th July 2025

Publication date: 9th September 2025

Proceedings of Extended Abstracts

EDITORS

Dr. Emi Norzehan Mohamad Mahbob

Dr. Saiyidah Nafisah Hashim

Ms. Lili Widarti Zainuddin

Ms. Nurul Nazwa Mohammad

Ms. Rozaina Saleh

e-ISBN: 978-629-97630-5-5

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Perak Branch, Tapah Campus, 35400 Tapah Road, Perak, Malaysia.

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PREFACE

Welcome to the 2nd International Science, Engineering and Technology Colloquium 2025 (ISETC 2025)!

It is with great pleasure that we present this year's colloquium, a distinguished gathering of scholars, researchers, and professionals dedicated to the advancement of applied sciences. Held virtually on 9th July 2025, ISETC 2025 serves as a platform to highlight cutting-edge research, foster meaningful networking, encourage collaborations, and explore innovative solutions to global challenges.

This year's theme, "The Power of Scientific Innovation: Intelligent Solutions for a Sustainable World" highlights our shared goal to solve global challenges through applied science. It celebrates innovation as a key to building a more sustainable and resilient future. We're proud to feature keynote speakers and presenters who bring fresh insights, bold ideas, and breakthroughs shaping tomorrow's world.

We would like to extend our heartfelt appreciation to the Faculty of Applied Sciences, Universiti Teknologi MARA, Perak Branch, Tapah Campus, for their outstanding support and assistance throughout the planning and execution of the event. Their dedication has been instrumental to the success of this colloquium. We would like to sincerely thank all participants and researchers for their valuable contributions. We also appreciate the reviewers for their careful evaluations and helpful feedback. Your hard work and support mean a lot to us.

As editors, we are proud to present this collection of proceedings. It is an honour to bring together the interesting and meaningful work of our speakers and authors. We hope the ideas shared will lead to new discoveries, encourage more research, and help solve global challenges through science and innovation.

Warm regards,

Dr. Emi Norzehan Mohamad Mahbob

Dr. Saiyidah Nafisah Hashim

Ms. Lili Widarti Zainuddin

Ms. Nurul Nazwa Mohammad

Ms. Rozaina Saleh

ISETC 2025 Proceedings Editors

PREPARATION OF Fe-MODIFIED RICE HUSK CATALYST FOR OLEIC ACID ESTERIFICATION

Aina Zulaikha Muzafar Shah¹, Nurul Jannah Abd Rahman², Farah Wahida Harun^{1*}

¹*Faculty of Science and Technology, Universiti Sains Islam Malaysia, 71800 Nilai, Negeri Sembilan, Malaysia*

²*Tamhidi Center, Universiti Sains Islam Malaysia, 71800 Nilai, Negeri Sembilan, Malaysia*

**Corresponding author: farahw@usim.edu.my*

Abstract: This work focuses on the preparation and characterization of a sustainable and cost-effective catalyst derived from rice husk for the esterification of oleic acid, a crucial step in biodiesel production. An agro-waste solid acid catalyst was prepared by modifying rice husk char with a structure-directing agent and various concentrations of Fe³⁺ ions. The prepared catalysts, namely RH, 0.02 M Fe-RH and 0.06 M Fe-RH were characterized using XRD, FESEM, EDX, and N₂ adsorption-desorption isotherm analysis. The effects of Fe³⁺ impregnation on the catalytic performance were investigated using the esterification of oleic acid and methanol as the probe reaction. The catalysts produced exhibit a mesoporous structure, providing a large surface area and uniform pore size distribution, which enhance its catalytic activity. The findings revealed that the 0.06 M Fe-RH catalyst demonstrated markedly improved catalytic performance over unmodified RHA, achieving the highest oleic acid conversion rate of 75.01 ± 0.65%. This work demonstrates the potential of utilizing rice husk as a sustainable precursor for the synthesis of highly efficient biodiesel catalysts, contributing to both waste management and the development of a more sustainable energy future.

Keywords: Esterification, rice husk, catalyst, oleic acid

INTRODUCTION

Esterification is one of the important steps in biodiesel production, particularly when a high free fatty acid (FFA) content of feedstocks is used. Waste cooking oils and non-edible oils are among the common feedstocks for biodiesel that contain high FFA and thus require an esterification step to reduce the FFA levels. This is to prevent soap formation, which can inhibit transesterification efficiency, consequently improving the overall yield and quality of biodiesel (Yu & Williams, 2022). Therefore, the development of efficient and sustainable catalysts for esterification is essential to utilize low-cost, high-FFA feedstocks effectively and reduce production costs.

Although significant progress has been achieved in developing efficient bio-derived catalysts for biodiesel production, reducing production costs remains a major challenge. One way to address this issue is by identifying cost-effective raw materials for catalyst synthesis, particularly silica precursors, which are crucial in the preparation of advanced catalytic materials. Based on Suyanta et al. (2023), costly silica precursors like sodium silicate and tetraethyl orthosilicate (TEOS) are commonly used in the synthesis of mesoporous materials. However, researchers have explored alternative sources of silica precursors to reduce production costs. These have included iron ore tailing (Deng et al., 2020), waste from the sugar industry (Chindaprasirt & Rattanasak, 2020), granite and silicon carbide sludge (Lin et al., 2021), bentonite (Perez et al., 2022), and rice husk (RH). RH is a natural substance that contains high concentrations of SiO₂, ranging from 92 to 95% (Jongpradist et al., 2018). Mesoporous materials prepared from rice husk exhibit properties including porosity, crystal structure, and hydrothermal durability. These properties are similar to those of materials synthesized from TEOS, a commercial silicate (Cazula et al., 2021).

This study aims to characterize the physicochemical properties of rice husk catalysts impregnated with varying amounts of Fe³⁺ and to evaluate their catalytic performance in the esterification of oleic acid with methanol. The scope of this study includes the synthesis of Fe-RH catalyst from rice husk through a series of steps involving rice husk pre-treatment, silica extraction, mesoporous formation, and Fe³⁺ impregnation via the incipient wetness method. The catalyst was characterized using XRD, EDX, and N₂ adsorption-desorption analysis, and its performance was evaluated through the esterification of oleic acid, with product acidity used to determine conversion efficiency.

MATERIALS AND METHODS

Preparation of Catalyst

Rice husk (RH) char was prepared following the method adapted from Suyanta et al. (2023) and Abbas et al. (2020) with slight modifications. The rice husk was washed and dried in an oven at 120°C for 12 hours. Then, 100 g of the dried husk was refluxed with 3 M HCl at 80°C for 3 hours. The husk was then dried for 12 hours at 120°C, and subsequently, calcined for 6 hours at 650°C. A solution of NaOH was mixed with calcined RH and heated to 80°C. Subsequently, cetyltrimethylammonium bromide (CTAB) as a structure-directing agent and sodium silicate solutions were added and stirred. The pH was adjusted to 11 by adding 1 M H₂SO₄, and the mixture was incubated at 100°C in the oven for 96 hours. After cooling, filtering and rinsing with distilled water, the sample was oven-dried at 105°C for 24 hours. Finally, the CTAB template was removed by calcining at 450°C for 5 hours. To prepare the Fe-modified catalyst, 5 g of the dried catalyst was submerged in a 500 ml liquid that had been acidified with HNO₃ to a pH of 2, containing two different Fe(NO₃)₃·9H₂O concentrations (0.02 M and 0.06 M). After 8 hours of magnetic agitation at 40°C, the mixture was cleaned with distilled water and heated for 3 hours at 110°C and then calcined in the air for 8 hours at 500°C. The final fine faded orange powder samples were obtained and labelled as 0.02 M Fe-RH and 0.06 M Fe-RH. All produced materials were characterized with XRD, N₂ adsorption-desorption analysis and EDX.

Catalytic Activity

The esterification of oleic acid (OA) with methanol (MeOH) was used to examine the activities of the catalysts produced from RH. A 250 mL two-necked round-bottom flask was fitted with a coiled condenser with a heating mantle. The procedure involved sequentially adding a 1:12 ratio of OA:MeOH into the flask. Then 4 wt.% of catalysts to oleic acid were added after the temperature reached 60°C and was maintained for 4 hours. Following the reaction, it was filtered to separate the liquid product from the heterogeneous catalyst. Then, the filtered mixture obtained after the esterification process was heated to 60°C-70°C to remove the excess methanol.

RESULTS AND DISCUSSION

The XRD patterns for RH and Fe-RH samples are presented in Figure 1. RH powder exhibited a broad diffraction hump at $2\theta = 20 - 30^\circ$, typical of amorphous silica in the mesoporous framework (Andas et al., 2020). This result suggests the high surface area and uniform pore structure, which are ideal for reactant accessibility in catalytic processes. 0.02 M Fe-RH and 0.06 M Fe-RH retained the broad amorphous silica feature with slightly reduced intensity, indicating the successful incorporation of Fe species into the silica framework without significant structural damage. The emergence of sharp diffraction peaks, possibly attributable to crystalline Fe₂O₃ can also be seen for the iron modified samples. These outcomes are aligned with a study conducted by Suyanta et al. (2023), which reported that the impregnation of Fe³⁺ ions into the mesopore structure leads to the incorporation of Fe species into the silica framework, resulting in the formation of acid sites that enhance catalytic activity.

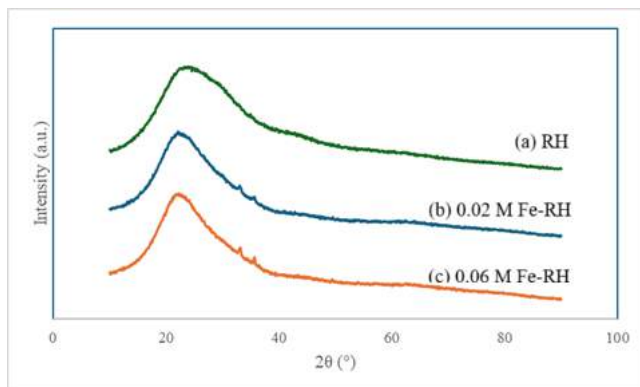


Figure 1. The X-ray diffractogram of RH and Fe-RH samples

Figure 2 illustrates the N₂ adsorption-desorption isotherms for the powder samples of RH and iron modified RH. All materials exhibited an IV isotherm type with a hysteresis loop, indicating that they are typical mesoporous materials (Suyanta et al., 2023). The textural properties of the samples are summarized in Table 1. The RH exhibited the highest BET surface area, indicating a high surface availability for catalytic activity. Upon iron impregnation, a reduction in surface area was observed for both 0.02 M Fe-RH and 0.06 M Fe-RH, suggesting partial pore blockage or coverage of the internal surface by iron species, a phenomenon also reported by Zhang et al. (2024). This is further supported by the XRD patterns, which showed that while the characteristic of amorphous silica in the mesoporous framework was retained, its intensity decreased slightly in Fe-modified samples, indicating successful incorporation of Fe species without significant structural collapse. Despite this decrease, the total pore volume increased slightly from 0.558 cm³/g (RH) to 0.566 cm³/g (0.02 M Fe-RH) and 0.570 cm³/g (0.06 M Fe-RH), possibly due to the development of additional mesopores or slight pore widening (Guo et al., 2021). The average pore size decreased from 10.498 nm (RH) to 10.077 nm (0.02 M Fe-RH) and 10.057 nm (0.06 M Fe-RH), which supports the assumption of structural modification after iron loading. These results indicate that iron loading alters the textural properties of RH, which may influence its performance in catalytic applications.

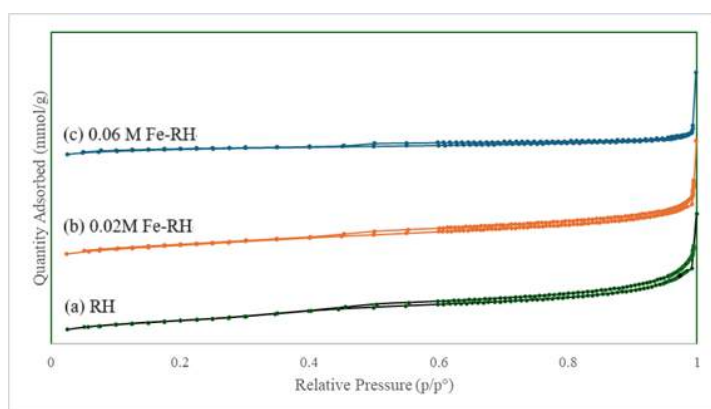


Figure 2. The N₂ adsorption-desorption isotherms of RH and Fe-RH samples

Table 1. The porosity and EDX analysis of RH and Fe-RH samples

Catalyst	BET (m ² g ⁻¹) ^a	Pore Volume (cm ³ g ⁻¹) ^b	Pore Size (nm) ^c	Fe (wt.%) ^d
RH	301.149	0.558	10.498	Not detected
0.02 M Fe-RH	250.182	0.566	10.077	0.21
0.06 M Fe-RH	251.892	0.570	10.057	5.08

^aBET surface area; ^bTotal pore volume taken from BJH adsorption; ^cBJH adsorption average pore size; ^dFe content from EDX analysis

The catalytic performance of RH and Fe-RH catalysts with different Fe concentrations in the esterification of oleic acid with methanol is summarized in Table 2. RH catalyst exhibited a moderate conversion of 36.06 ± 4.75%, primarily due to its mesoporous structure, which provides a high surface area but lacks active acid sites. The incorporation of Fe into the RH framework enhanced the catalytic activity, as observed in 0.02 M Fe-RH, which achieved a conversion of 48.30 ± 4.18%. The highest performance was recorded for 0.06 M Fe-RH, with a conversion of 75.01 ± 0.65%. This improvement is attributed to the increased availability of Fe-based active sites, facilitating the esterification reaction (Enguilo Gonzaga et al., 2021). The high conversion observed for Fe-MCM-41 (0.06 M) aligns with its structural properties, as confirmed by XRD, N₂ adsorption-desorption and EDX analyses. Incorporation of iron species into a catalyst creates active sites within the material. The Fe sites exhibit Lewis's acidity, which contributes to a strong affinity for electron pairs (Pirouzmand et al., 2017). This Lewis acidity is crucial for catalyzing the esterification reaction. By promoting the forward reaction, the Fe catalyst helps to shift the equilibrium towards the formation of methyl oleate, resulting in a high conversion of oleic acid.

Table 2. Esterification of oleic acid with methanol

Catalyst	Conversion (%)
RH	36.06 ± 4.75
0.02 M Fe-RH	48.30 ± 4.18
0.06 M Fe-RH	75.01 ± 0.65

CONCLUSIONS

It can be concluded that rice husk, a silica-rich agricultural waste, can be effectively converted into a mesoporous solid acid catalyst suitable for the esterification of oleic acid as a key step in biodiesel production. The incorporation of Fe³⁺ ions into the RH framework successfully enhanced its catalytic activity, with the 0.06 M Fe-RH catalyst demonstrating the highest conversion efficiency at 75.01 ± 0.65%. This improved performance is attributed to the formation of active acid sites within the mesoporous silica structure, which facilitates better interaction with reactants. This finding supports the potential of RH-derived mesoporous materials as sustainable, low-cost alternatives to commercial catalysts (e.g., TEOS-based materials). The process is environmentally friendly and cost-effective, aligning with green chemistry and circular economy principles. It is recommended to optimize the esterification reaction conditions to further improve oleic acid conversion while also evaluating the catalyst's reusability, long-term stability, and performance with various high-FFA feedstocks to support its industrial applicability.

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Surat kami : 700-KPK (PRP.UP.1/20/1)
Tarikh : 20 Januari 2023



Prof. Madya Dr. Nur Hisham Ibrahim
Rektor
Universiti Teknologi MARA
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Kelulusan daripada pihak tuan dalam perkara ini amat dihargai.

Sekian, terima kasih.

"BERKHIDMAT UNTUK NEGARA"

Saya yang menjalankan amanah,

Setuju.

27.1.2023

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Timbalan Ketua Pustakawan

PROF. MADYA DR. NUR HISHAM IBRAHIM
REKTOR
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