Rice Husk Ash-Derived Catalyst for Diethyl Ether Production Through Ethanol Dehydration in A Large-Scale Reactor

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ABSTRACT

Ethanol dehydration to diethyl ether (DEE) was carried out in large-scale reactors using rice husk ash (RHA), a low-cost biomass catalyst derived from agricultural waste. This paper investigates the impact of temperature and liquid hourly space velocity (LHSV) to determine the optimal conditions and stability for ethanol dehydration to DEE over an RHA catalyst in a large-scale reactor (2.1 cm i.d., 30 cm length). The RHA catalyst was synthesized using a reflux process with sulfuric acid, followed by calcination at 600 °C, and characterized through X-ray Diffraction (XRD), Fourier-Transform Infrared Spectroscopy (FT-IR), Field Emission Scanning Electron Microscopy (FE-SEM), X-ray Fluorescence (XRF), Surface area and pore size analysis, NH_3 temperature-programmed desorption, and thermogravimetric analysis (TGA). The optimal conditions were determined to be 360 °C and an LHSV of 1.3 h^{-1}, which resulted in the highest DEE yield production of 99.21% and ethanol conversion exceeding 99%. The results of the stability tests, conducted over a 24-hour period, demonstrated that RHA is resilient to deactivation and suitable for long-term catalytic applications. These findings indicate the potential of RHA as a promising and sustainable catalyst for the large-scale production of DEE.

Keywords: *Rice husk ash; Catalytic dehydration; Diethyl ether; DEE; Dehydration of ethanol*

1. INTRODUCTION

Diethyl ether (DEE) is one of the most valuable chemicals, primarily used as a solvent in chemical and pharmaceutical processes. Additionally, DEE has important properties, such as a high cetane and octane number, which make it widely used as an additive for diesel and gasoline engines [1] – [6]. Generally, DEE is produced through the catalytic dehydration of ethanol using homogeneous acid catalysts, which presents challenges due to difficult separation and the corrosive nature of the catalyst [1], [2], [4]. Meanwhile, heterogeneous catalysts can help reduce production costs [1]. Consequently, the dehydration of ethanol over an acidic solid catalyst has gained attention.

In recent years, researchers have attempted to develop various solid catalysts. A 2.41% yield of DEE was obtained at 250 °C with 96% ethanol using a Raw Klaten natural zeolite (ZA) catalyst [7]. Remi et al. [8] investigated the SO_4/SiO_2 catalyst for producing DEE through the dehydration of ethanol, achieving a maximum DEE yield of 11.36% at 200 °C. Rahmanian' group [9] studied aluminum phosphate-hydroxyapatite as a catalyst for ethanol dehydration. The results showed a high DEE yield of over 75% at 340 °C and 200 bar. However, the catalyst has high production costs and a complex synthesis process, so using agricultural biomass waste for catalyst synthesis can reduce costs and aid in biomass waste disposal [10]. Lakhani and Srifa [11] focuses on the use of Ni-Re bimetallic catalysts for the conversion of biomass-derived molecules into high-value fuels and chemicals. The synergy between Ni's hydrogenation activity and ReOX's oxophilic acidity enables efficient transformations of compounds such as furfural, 5-hydroxymethylfurfural, levulinic acid, and fatty acid esters. Yuan et al. [12] studied copper-based catalysts and ruthenium complexes, with the objective of achieving this transformation under mild conditions. The direct production of ethyl acetate from bioethanol is a significant contribution to the development of green chemical processes.

The main challenge in the ethanol dehydration industry lies in the high cost of catalysts and the complexity of their preparation [13]. Conventional catalysts, including transition metal-based catalysts, often necessitate the use of rare or costly materials, resulting in increased costs and limited availability [14]. Agricultural waste provides a practical solution to this problem [15]. Rice husk ash (RHA) is a widely available agricultural waste worldwide, with every metric ton of rice producing over 150 kg of rice husk [16]. RHA is considered a low-cost catalyst and renewable materials, as it contains high silica and small amounts of alumina [17]. RHA exhibits good thermal stability, retaining its structural integrity under high temperatures, which is crucial for catalytic processes [14]. Furthermore, the high porosity and large specific surface area of RHA render it an optimal material for the production of catalysts. Chang et al. [18] developed a Cu/RHA catalyst for ethanol dehydrogenation, demonstrating high catalytic activity. Chen et al. [19] reported high catalytic activity of RHA-based catalysts in the transesterification of palm oil, achieving a biodiesel yield of 91.5%.

As mentioned earlier, Rice husk ash (RHA) is a viable industrial material due to its status as a low-cost and widely available agricultural by-product. The material's high silica content, thermal stability, high porosity, and large specific surface area provide excellent surface properties for catalytic reactions, allowing it to maintain activity under high temperatures and achieve high reaction efficiency. Additionally, RHA has gained increasing attention as a valuable material for industrial applications due to its abundance and efficient utilization of rice byproducts. In Thailand, the RHA market is projected to grow at a rate of 7.60% by 2027, following trends seen in major economies such as China, India, Japan, Australia, and South Korea. This growth is largely driven by its wide applications in sectors such as construction and agriculture, where RHA is used in concrete production and soil amendment. Beyond these uses, its unique physicochemical properties also make RHA a promising and sustainable resource for catalyst development in chemical industries [20].

The aim of the present study is to investigate biomass derived RHA as a low - cost catalyst for the catalytic dehydration of ethanol to DEE in a large-scale fixed bed reactor. This study aims to examine the effects of temperature (250 - 380 °C) and liquid hourly space velocity (LHSV 1.1-1.9 h^{-1}), as well as to investigate the stability and optimal conditions for ethanol dehydration over the RHA catalyst.

2. EXPERIMENT

2.1 SYNTHESIS OF RHA

The process of extracting silicon dioxide from rice husks (RHA) commences with the passage of the husks through a sieve with a mesh size of 1.8-2.2 microns, the objective being the elimination of impurities. Subsequently, the RHA are washed and dried in an oven set at 80°C (Memmert, D06062 Model 600). Subsequently, silica is extracted through a reflux process utilizing a 0.5 M sulfuric acid solution (H_2SO_4, 98 wt.%, Anapure Bioscientific) at a ratio of 100 g of rice husk to 1500 ml of solution, conducted at 80 °C for 2 hours. The material is washed on multiple occasions until the pH reaches 7, with the process monitored using a positive potential hydrogen ion meter (Hanna Instruments, HI 2211 pH/ORP Meter). Subsequently, the material is subjected to a second drying phase in a muffle furnace, where it is calcined at 600 °C with a heating rate of 10°C/min for 4 hours, resulting in rice husk ash rich in silicon dioxide.

2.2 RHA characterization

The crystal structure and phases present in the sample were analyzed using X-ray diffraction (XRD, Bruker D2 Phaser, Germany). The morphology and elemental composition of the RHA were examined using a field emission scanning electron microscope (FE-SEM, ZEISS Auriga, Germany). The presence and properties of SiO_2 in the RHA were identified through the use of Fourier-Transform Infrared Spectroscopy (FTIR, Bruker Vertex70, Germany) and X-ray Fluorescence (XRF, Horiba XGT5200, Japan), respectively. The specific surface area of the RHA was determined by N_2 adsorption using a surface area and pore size analyses (Quantachrome Instruments, Anton Paar, Austria). The distribution of acid sites was determined by means of NH_3 temperature-programmed desorption (NH_3-TPD, BELCAT-B, BEL Japan INC., Japan). Finally, the thermogravimetric analyzer (TGA, TGA/DSC1, Mettler Toledo, United States) was utilized to quantify the quantity of coke deposits on the catalysts.

2.3 CATALYTIC TESTS

Catalytic experiments were performed in an electrically heated fixed-bed reactor with an internal diameter of 2.1 cm and a length of 30 cm, using 3 g of catalyst (10 - 12 mesh) as shown in Figure 1. The reactions were carried out at temperatures ranging from 250 to 380 °C under atmospheric pressure. Liquid-phase ethanol (95 wt.%, Scitrader, Thailand) was introduced into a preheater system at 200°C with a variable LHSV (liquid hourly space velocity) between 1.1 and 1.9 h^{-1}. The catalyst was preheated to the reaction temperature for 1 hour before starting the reaction. The outlet gases were analyzed using FID gas chromatography with a J&W packed GC columns (Agilent 7890A, equipped with a packed column), while the liquid product was analyzed using TCD gas chromatography (Agilent 6890, equipped with an HP-5HS GC capillary column).

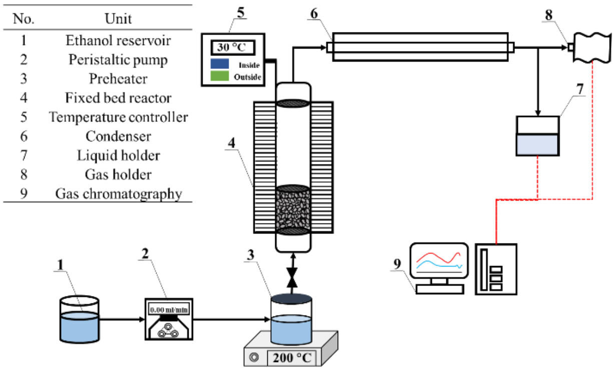


Fig. 1 Schematic diagram of reaction test system

3 DISCUSSION AND CONCLUSION

3.1 CHARACTERIZATION OF RHA

The XRD patterns obtained at a scan rate of 2.4°/min using Cu K-α radiation are shown in Figure 2 The peak observed at an angle of 22° (2θ) was confirmed as silica (silicon dioxide) [21], [22] and specifically indicated cristobalite, a polymorph of silica [23]. Additionally, the peak at 22° also indicates the presence of amorphous silica [22], [24]. Besides this, the peaks at 20.9°, 21.8°, and 26.6° also indicate silica [25], [26].

In contrast, the reference silica from a natural source exhibits sharp and intense peaks, corresponding to crystalline quartz. The absence of crystalline peaks in the RHA sample confirms that the treatment successfully produced amorphous silica, which is more reactive and suitable as a precursor for zeolite synthesis. This comparison highlights the effectiveness of RHA as a sustainable and high-potential silica source for catalyst preparation.

XRF was used to determine the chemical composition and assess the purity of silica extracted from rice husk. The XRF results for RHA, shown in Table 1, indicate that SiO_2 is the major component, with a small amount of Al_2O_3. Other components of RHA include metallic elements such as iron (Fe), chromium (Cr), potassium (K), manganese dioxide (MnO_2), tantalum (Ta) and zinc (Zn).

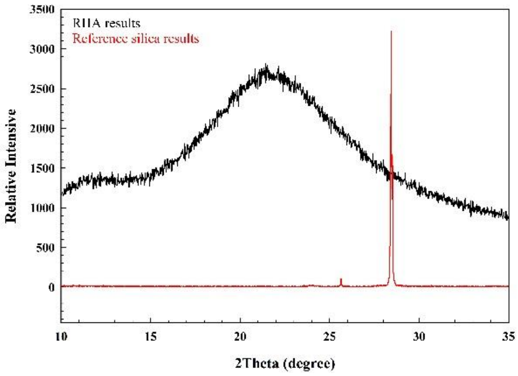


***Fig. 2** Results of the XRD patterns for RHA*

***Table 1** XRF chemical analysis results of RHA*

Element	Mass (%)	Compound	Mass (%)
Al	0.31	Al_2O_3	0.355
Si	98.23	SiO_2	99.009
P	0.20	P_2O_5	0.116
Si	0.33	SiO_3	0.204
Ca	0.80	CaO	0.272
Fe	0.04	Fe_2O_3	0.014

FTIR spectroscopy was performed to examine the functional groups in RHA, as shown in Figure 3. The spectra were recorded in the range of 400-4000 cm^{-1} with 64 scans at a resolution of 4 cm^{-1}. High-intensity transmittance peaks were observed at 802.32, 1053.05, 1641.29, 2325.97, and 3747.39 cm^{-1}. The spectral region as 1000-1260 cm^{-1} correspond to Si-O bonds from silica, as do those in the 800-1000 cm^{-1} range [27], [28]. Additionally, the bands at 1641.29 cm^{-1} and in the 3300-3600 cm^{-1} region represent the bending vibrations of hydroxyl groups and Si-OH (silanol) stretching from H_2O molecules [28] – [30]. Silanol groups enhance the acidic properties of the RHA catalyst, which are vital in ethanol dehydration. Acidic sites facilitate the protonation of ethanol molecules, leading to the loss of water molecules and the production of ethylene. Additionally, silanol groups aid in the adsorption of ethanol on the catalyst surface through hydrogen bonding [11] – [14].

NH_3-TPD profiles were obtained using a thermal conductivity detector (TCD). The sample was first heated from room temperature to 450°C at a rate of 10°C/min for 50 minutes under helium, then cooled to 100°C. It was then saturated with a 5% NH_3/He stream at 100°C for 30 minutes. Afterward, excess ammonia was removed with helium at 100°C for 15 minutes. Desorption of NH_3 was carried out by heating from 100°C to 800°C at a rate of 10°C/min. Figure 4 shows the NH_3-TPD profiles of RHA, where the small peak observed between 150 - 250°C corresponds to weak and medium-strength acid sites, and the large peak at temperatures above 600°C indicates the presence of abundant strong acid sites [31] – [33].

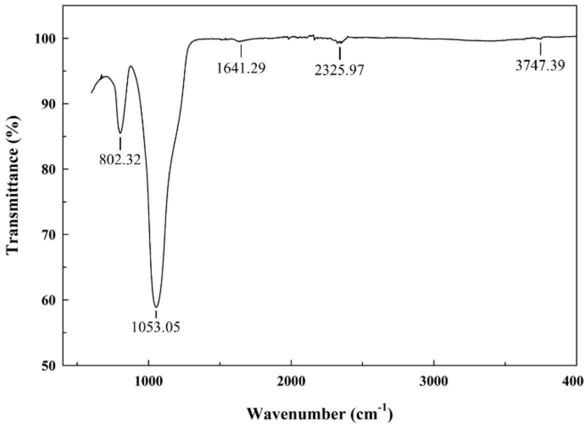


Fig. 3 *FTIR spectrum of RHA*

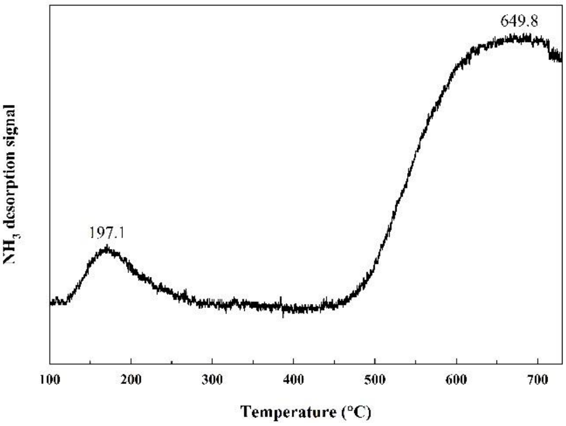


Fig. 4 *NH_3-TPD profiles of RHA*

Fig. 5 shows the RHA particles analyzed using a field emission scanning electron microscope (FE-SEM) at magnifications of 1,000X (a), 5,000X (b), 10,000X (c), and 30,000X (d), revealing their nanospherical morphology with a tendency to agglomerate [34] – [36].

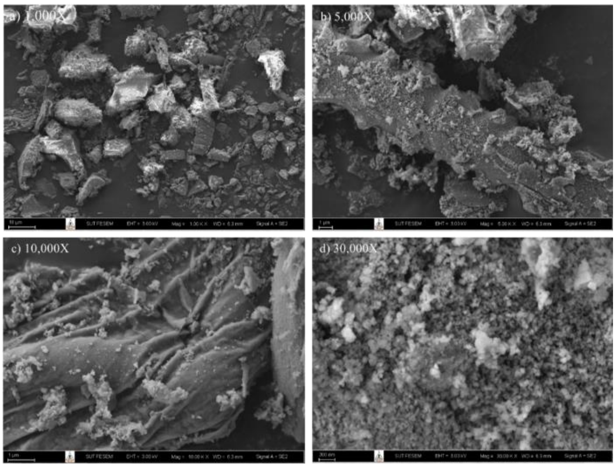


Fig. 5 *FE-SEM results of RHA at different magnifications*

Table 2 presents the results of the surface area and pore size analysis of RHA, including the BET surface area, total pore volume, and average pore diameter. The BET surface area was determined to be 208.2 m²/g, with a total pore volume of 0.332 cm³/g. An average pore diameter of 3.187 nm suggests that the material is mesoporous.

***Table 2** Results of surface area and pore size analysis of RHA.*

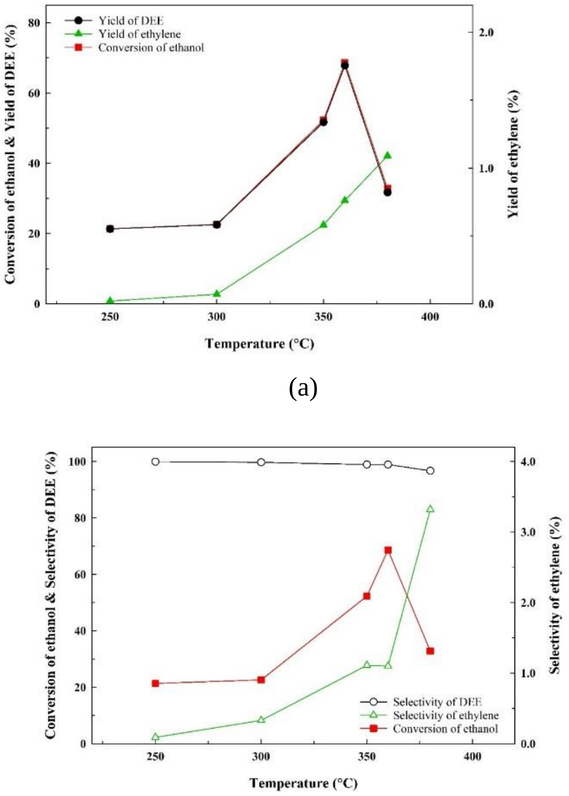
Parameter	Unit	
BET surface area	(m^2/g)	208.2
Total pore volume	(cm^3/g)	0.332
Pore diameter	(nm)	3.187

3.2 CATALYTIC PERFORMANCE OF ETHANOL DEHYDRATION

Fig. 5 shows the effect of reaction temperature on the catalytic dehydration of ethanol over RHA catalyst. As the temperature increases from 250 to 360°C, the yields and selectivity of DEE and ethylene also increase, similar to the ethanol conversion. Herein, the highest yield and selectivity of DEE were observed at 67.83% and 98.90%, respectively. As the temperature increased to 380°C, a decline in the yield and selectivity of DEE, as well as in ethanol conversion, was observed. Conversely, the yield of ethylene exhibited an increase. The formation of ethylene is favored by high temperatures through intramolecular dehydration, whereas lower temperatures encourage intermolecular dehydration, resulting in the formation of diethyl ether [2], [37].

The effect of LHSV on the catalytic dehydration of ethanol over RHA is illustrated in Fig. 6. As the LHSV increased from 1.1 to 1.3 h^{-1}, both the conversion of ethanol and the yield of DEE slightly increased. However, these values gradually decreased as LHSV continued to rise. Meanwhile, the yield and selectivity of ethylene decreased as the LHSV increased from 1.1 to 1.9 h^{-1}. In Fig. 6(b), it can be observed that the yield of diethyl ether slightly increased with the increase in LHSV, which corresponds to the decrease in ethylene yield. The basic reason is that at high space velocities, the decreased residence time of ethanol and lower interaction with active sites contribute to a reduction in conversion [38].

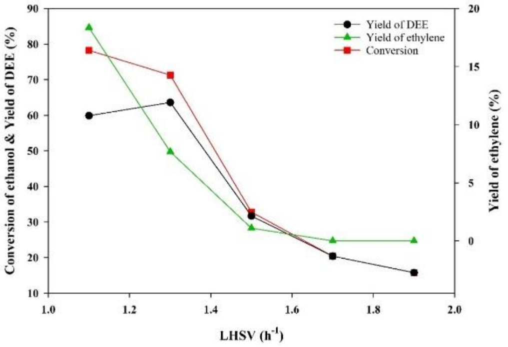
Table 3 compares this work with previous studies, showing that RHA in a pilot-scale reactor demonstrates strong efficiency for DEE production, surpassing many micro- and lab-scale catalysts. Although certain catalysts achieve marginally higher activity, the scalability and robust performance of RHA at elevated temperatures (360°C) highlights its potential for industrial application.



(a)

(b)

***Fig. 6** Conversion of ethanol and yield (a) and selectivity (b) of products (DEE and ethylene) over RHA at various temperatures with LHSV = 1.5 h^{-1}*



(a)

***Fig. 7** Conversion of ethanol, and yield (a) and selectivity (b) of products (DEE and ethylene) over RHA at various LHSV, with T = 380℃ (Cont.)*

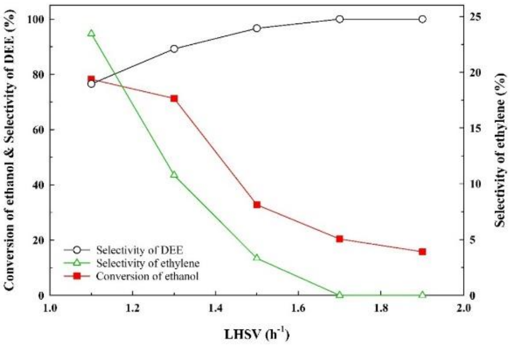


Fig. 7 Conversion of ethanol, and yield (a) and selectivity (b) of products (DEE and ethylene) over RHA at various LHSV, with T = 380 ℃

Table 3 Comparison of various catalysts and scale for DEE yield.

Catalyst	Scale	T (°C)	%yield	Ref.
HBZ Ru-HBZ Pt-HBZ	Micro-reactor	250	35.00 47.00 45.00	[39]
SO_4/SiO_2	Lab-scale	200	11.36	[8]
WO_3-MMC		250	42.63	[40]
WO_3-AC		300	22.30	
Siralox		300	65.00	[41]
SO_4/ZrO_2		225	49.85	[42]
RHA	Pilot-scale	360	99.21	This work

Fig. 8 represents the effect of time-on-stream on catalytic dehydration of ethanol over RHA. At a reaction temperature of 360°C and an LHSV of 1.3 h^{-1}, the highest DEE yield of 99.21% was achieved at 99% ethanol conversion. Over the 24-hour period, the conversion of ethanol and the yield of DEE remained relatively stable, exhibiting only minor fluctuations. This stability indicates that the RHA catalyst demonstrated robust long-term activity and resilience to deactivation under the specified operational conditions. In this study, the stability test was performed for 24-hour, which provides an initial understanding of catalyst performance. For future work, extended stability evaluations (>100 h) together with catalyst regeneration studies are recommended to further verify the long-term durability and reusability of the catalyst.

The thermal stability of RHA was examined from 20 to 1000°C at a heating rate of 10°C/min under a nitrogen atmosphere using TGA, as shown in Fig. 9, indicating that the RHA catalysts have high thermal stability. This stability is an important characteristic, indicating that RHA is capable of withstanding the thermal demands of catalytic dehydration reactions over extended periods without significant decomposition.

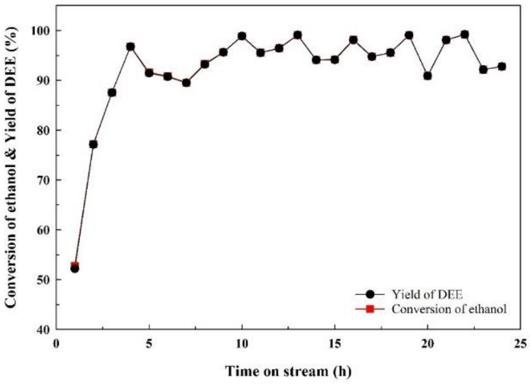


Fig. 8 Long-term 24-hour on-stream catalytic run at 360°C and LHSV = 1.3 h^{-1} in the gas phase: conversion of ethanol and yield of diethyl ether

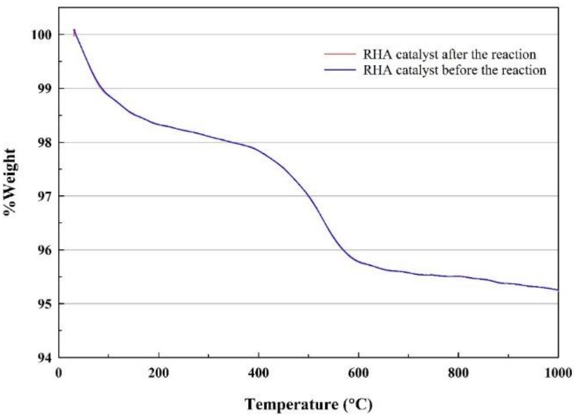


Fig. 9 TGA profiles of RHA catalysts after the 24-hour stability test

4. CONCLUSION

This study establishes rice husk ash (RHA), an abundant agricultural by-product, as an effective catalyst for the dehydration of ethanol to diethyl ether (DEE) in a pilot-scale fixed-bed reactor. In the context of optimal conditions (360°C, LHSV 1.3 h^{-1}), the process attained a DEE yield of 99.21%, accompanied by complete ethanol conversion, thereby substantiating the catalytic efficacy and stability of RHA. Moreover, the findings of this study demonstrate the potential for agricultural waste to be transformed into a high-performance catalyst, thus advancing circular economy principles, reducing reliance on costly synthetic catalysts, and lowering overall process costs.

The physicochemical analysis revealed that RHA's moderate surface area and weak-to-medium acid sites were sufficient to drive the dehydration reaction, while TGA confirmed its excellent thermal stability. These characteristics underscore RHA's aptitude for sustainable and long-term catalytic applications. However, further research is required to extend the stability testing, evaluate the regeneration of the catalyst, and assess the scalability of the process. Comprehensive techno-economic and life-cycle assessments will be essential to validate RHA's industrial feasibility.

In summary, the present work positions RHA not only as a practical catalyst for ethanol dehydration but also as a strategic pathway for waste valorization and sustainable chemical production, contributing both economic and environmental benefits to future industrial energy systems.

5. ACKNOWLEDGEMENT

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