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2 ND INTERNATIONAL SCIENCE, ENGINEERING AND TECHNOLOGY COLLOQUIUM



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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

SULFATE-BASED IONIC LIQUIDS AS SOLVENTS FOR DECOMPOSITION OF LIGNOCELLULOSE MATERIALS FROM COCONUT HUSK FIBER

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Abstract: This study investigates the use of sulfate-based ionic liquids (ILs) as solvents for decomposing lignocellulose materials derived from coconut husk fibers. Coconut husk, a commonly discarded biomass waste, has high lignin content, making its decomposition challenging using traditional methods. The objective of the research was to determine the efficiency of the decomposition process of lignocellulose from coconut husk fiber by using sulfate-based ionic liquids and assess their characteristics. Samples were subjected to two types of ILs and characterised using FTIR, NMR, and TGA analyses. FTIR analysis confirmed the presence of functional groups corresponding to sulfate and ammonium ions, with distinctive peaks representing NH stretching, C–N bonds, and S=O vibrations, indicating the successful synthesis of the ILs. NMR spectra further verified the molecular structures of diethylammonium sulfate (DEAS) and triethylammonium sulfate (TEAS), with chemical shifts consistent with the expected hydrogen and carbon environments of the ionic liquid components. TGA analysis demonstrated that both ILs possessed good thermal stability, with decomposition onset temperatures above 200°C, suitable for biomass decomposition processes. The untreated and treated coconut husk fibers were analysed via FTIR that showed reductions in lignin and hemicellulose peaks, confirming partial removal of these components. FESEM analysis revealed noticeable morphological changes on the fiber surface, from smooth and denser structure (untreated) to rough and fragmented (treated), and CHNS elemental analysis showed a reduction in post-treatment carbon content, supporting the conclusion that lignin-rich fractions were successfully removed during the decomposition process. Results showed that the optimal decomposition yields were about 75.33% for TEAS and 83.66% for DEAS at 130°C for 5 hours. These findings highlight the effectiveness of sulfate-based ionic liquids in decomposing the rigid structure of lignocellulose. The significant decomposition yields demonstrate that ILs can successfully break down biomass into simpler components, providing an eco-friendly alternative.

Keywords**:** *Ionic liquids (ILs), sulfate-based, lignocellulose, coconut husk fibers, decomposition*

INTRODUCTION

Coconut husk fiber, rich in lignocellulose, represents a significant biomass resource that remains underutilized in industry. However, concerns arise regarding the slow decomposition rate contributes to environmental pollution and places pressure on waste management systems, particularly due to the traditional decomposition methods, often require high costs and energy consumption while posing some risks. Ionic liquids (ILs), especially sulfate-based ILs, present a promising alternative due to their recyclability, thermal stability, and eco-friendliness. This study aimed to (i) synthesize sulfate-based ionic liquids for decomposition of lignocellulose from coconut husk fiber, (ii) characterize the synthesized sulfate-based ionic liquids used for decomposition of lignocellulose from coconut husk fiber, and (iii) determine the efficiency of the decomposition process of lignocellulose from coconut husk fiber by using sulfate-based ionic liquids and assess their characteristics. Some of the previous studies the limitations of traditional pretreatment methods involve high operational costs, generate hazardous byproducts, and are less effective in breaking down biomass with high lignin content like coconut husk fibers.

MATERIALS AND METHODS

The coconut husk fibers were cleaned, dried, and ground before decomposition treatment. The synthesis of the sulfate-based ionic liquids, DEAS was carried by added dropwise with 2M sulfuric acid in the presence of methanol as a co-solvent. The mixture was stirred and heated at 60°C for 1 hour. Similarly, TEAS was synthesized by reacting triethylamine with sulfuric acid, followed by heating at 70°C for 3 hours. After the reaction, both mixtures were cooled and purified through repeated washing with distilled water for DEAS and diethyl ether for TEAS, and solvents were removed using rotary evaporation. The resulting ionic liquids were stored for further characterization and application in the decomposition process. The characterization of ILs was conducted using Fourier Transform Infrared Spectroscopy (FTIR), Nuclear Magnetic Resonance (NMR), and Thermogravimetric Analysis (TGA).

Lignocellulose decomposition tests were carried out by immersing 1 g of coconut husk in 27 mL of ILs at different temperatures and durations. The resulting treatments were analyzed via FTIR that used to detect changes in chemical functional groups after treatment, FESEM was employed to observe morphological changes on the fiber surfaces, and CHNS elemental was conducted to quantify the elemental composition, specifically carbon, hydrogen, nitrogen, and sulfur content before and after decomposition.

RESULTS AND DISCUSSION

The results of synthesized diethylammonium sulfate (DEAS) and triethylammonium sulfate (TEAS) were shown in FTIR analysis confirmed the successful formation of ionic liquids by identifying key functional groups. Peaks corresponding to NH stretching (~3354 cm^{-1}), C=C stretching (~1510–1600 cm^{-1}), and S=O stretching (~1189 cm^{-1}) indicated the presence of sulfate anions and ammonium groups in both DEAS and TEAS.

TGA analysis showed that both ILs exhibited high thermal stability, with decomposition temperatures starting above 200°C. This indicates that both ionic liquids are thermally stable under the decomposition conditions used for lignocellulose processing.

The decomposition process revealed that DEAS achieved the highest decomposition yield of 83.66%, followed by TEAS with 75.33% at the optimal condition of 130°C for 5 hours (Table 1).

Table 1. Percentage decomposition (%) of treated CHF

Samples	Initial weight (g)	Final weight (g)	Percentage decomposition (%)
Treated CHF with DEAS	1.0034	0.1640	83.66
Treated CHF with TEAS	1.0402	0.2566	75.33

FTIR analysis of the treated coconut husk fibers showed slight shift from the untreated which is at 3354 cm^{-1} to 3340 cm^{-1} indicated changes in the presence of amine groups in TEAS and DEAS. Besides, the S=O stretching has dropped considerably from the actual peak of the untreated biomass (1189 cm^{-1}), which is at 1157 cm^{-1}. This would mean that the treatments might reduce the ester functionality by forming new bonds with the biomass hence reducing carbonyl absorption in this region. The aromatic C=C stretch which at around 1510–1600 cm^{-1} also has disappeared, probably because treatments may cause partial depolymerization of lignin or alter its structure in such a way as to reduce the characteristic lignin-related peaks.

FESEM images provided visual evidence of morphological disruption, where untreated CHF (Figure 1 (a)) exhibited smooth and denser, compared to the rough, uneven and irregular structure of treated CHF (Figure 1 (b) and (c)). However, the treated CHF with DEAS ionic liquid appears to be rougher and more porous than the CHF treated with TEAS ionic liquid. Thus, this finding correlated to the DEAS ionic liquid gave higher percentage of decomposition, which due to more effective delignification and dissolution of the amorphous regions.

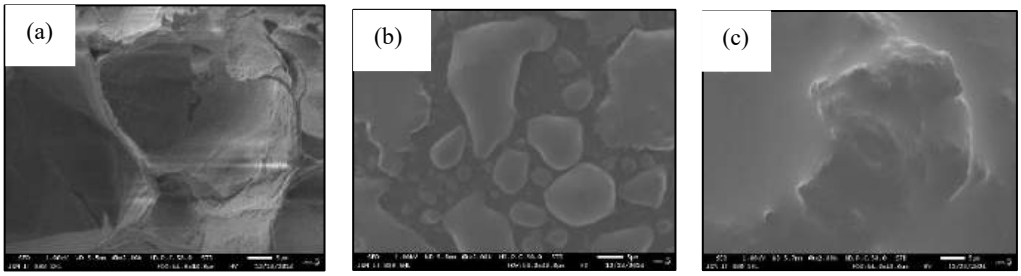


Figure 1. (a) untreated CHF, (b) treated CHF with DEAS ionic liquid, (c) treated CHF with TEAS ionic liquid

CHNS elemental analysis further supported the findings (Table 2), demonstrating a reduction in post-treatment carbon content, consistent with the removal of lignin-rich fractions. Comparatively, CHF treated with DEAS showed the lowest percentage of carbon content and gave evidence that part of the organic components, mostly hemicellulose and portions of lignin have been washed out during the treatment. Meanwhile, CHF treated with TEAS had higher carbon content that showed the elimination of the particular organic fraction without affecting the structures of cellulose and lignin. After IL pretreatment, residual IL may remain adsorbed or trapped in the porous structure of the biomass that might also contribute to the higher percentage of sulfur, hydrogen and nitrogen in the treated CHF.

Table 2. CHNS data for untreated and treated CHF

SAMPLE	C (%)	N (%)	H (%)	S (%)
Untreated CHF	44.56	0.68	5.288	0.351
CHF + DEAS	27.49	6.88	7.824	15.185
CHF + TEAS	34.14	6.39	9.947	13.51

CONCLUSIONS

Based on this study, DEAS showed higher decomposition yield and better performance compared to TEAS ionic liquid. This might be DEAS has smaller cation to facilitate better interaction with cellulose and lignin hydrogen bonds, which is crucial for disruption. Both ionic liquids are thermally stable and suitable for biomass pretreatment. DEAS may thermally degrade earlier but is still stable enough under normal pretreatment conditions.

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