UNIVERSITI TEKNOLOGI MARA

# PRETREATMENT OF OIL PALM LIGNOCELLULOSIC BIOMASS USING 1-ETHYL-3-METHYLIMIDAZOLIUM ACETATE

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Thesis submitted in fulfillment
of the requirements for the degree of
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**(Chemical Engineering)**

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# AUTHOR'S DECLARATION

I declare that the work in this thesis was carried out in accordance with the regulations of Universiti Teknologi MARA. It is original and is the results of my own work, unless otherwise indicated or acknowledged as referenced work. This thesis has not been submitted to any other academic institution or non-academic institution for any degree or qualification.

I, hereby, acknowledge that I have been supplied with the Academic Rules and Regulations for Post Graduate, Universiti Teknologi MARA, regulating the conduct of my study and research.

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# ABSTRACT

Lignocellulosic biomass has a great potential to be a prospective renewable resource that can be used for the generation of bioethanol. Oil palm biomass was chosen as it is one of the most abundances residues in Malaysia. The major concern in lignocellulose conversion is overcoming biomass recalcitrance through pretreatment while still maintaining a green engineering method, an energy efficiency and an economically feasible process. Recent development showed that ionic liquids can be utilized to treat lignocellulosic biomass efficiently, however, the drawback of using pure ionic liquid is its expensive cost and also extremely viscous. Therefore, this research investigated on the use of aqueous IL during pretreatment. The ionic liquid used was 1-ethyl-3-methylimidazolium acetate [EMIM]Ac and the raw materials were Microcrystalline Cellulose (MCC) and Oil Palm Frond (OPF). The effect of [EMIM]Ac concentration [0-100%], temperature (70-99°C) and dissolution time (1-3 hours) on pretreatment of lignocellulosic biomass was analysed by Fourier Transform Infrared (FTIR), X-Ray Diffraction (XRD) and Scanning Electron Microscopy (SEM). It was found that the optimum conditions for the pretreatment is 50-100% [EMIM]Ac concentration, temperature of 99°C and dissolution time of 3 hours. Based on peak analysis and Lateral Order Index (LOI) value, Crystallinity Index (CI) value and the changes of morphology. The effect of increasing concentration, temperature and time decreased LOI and CI values indicating reduction in the degree of crystallinity and increased of amorphousity of the MCC and OPF sample. The LOI and CI values for OPF pretreated with 10% [EMIM]Ac are 1.02 and 0.45 where it decreased to 0.24 and 0.28, respectively after concentration of [EMIM]Ac was increased to 100%. The same trend was observed for effect of temperature where the LOI and CI values for OPF pretreated with 50% [EMIM]Ac at temperature of 70°C which are 0.97 and 0.46 were decreased to 0.61 and 0.38, respectively after pretreated with 50% [EMIM]Ac at temperature of 99°C. Moreover, the LOI and CI values for OPF pretreated with 50% [EMIM]Ac for 1 hours dissolution time are 1.05 and 0.48 where it decreased to 0.48 and 0.38, respectively after the dissolution time was increased to 3 hours. The findings from SEM support the FTIR and XRD results as the pretreated sample structure became cracked and disordered while the raw sample have a smooth structure. Based on the optimum condition studied, the pretreatment was also conducted by using Oil Palm Trunk (OPT) and Empty Fruit Bunch (EFB). It is proven that aqueous [EMIM]Ac starting with 50% to 90% concentration can disrupt the cellulose structure. Therefore, it is feasible to employ aqueous [EMIM]Ac in the range of 50-90% concentration in order to achieve efficient dissolution in an economically viable pretreatment.

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# CHAPTER ONE
# INTRODUCTION

## 1.1 Research Background

In recent years, the rapidly increasing of energy requirement, environmental threat and also the depletion of fossil fuels reserves have drawn significant attention among researchers to explore the lignocellulosic ethanol production as an attractive research area (Zhu *et al.*, 2013; Trinh *et al.*, 2015 and Mahmood *et al.*, 2016). Lignocellulosic materials can be represented as softwood, hardwood, agricultural and forest residues, grasses, food industry wastes and also municipal solid wastes. Cellulose, hemicellulose and lignin are the major components exist in the lignocellulosic biomaterials (Reddy, 2015 and Mahmood *et al.*, 2016). The typical content of cellulose, hemicellulose and lignin are of 35-50%, 20-35% and 5-30%, respectively, based on the plant dry weight (Lynd *et al.*, 2002). For the production of bioethanol from lignocellulosic biomass, cellulose has to be separated from lignin and hemicellulose. During pretreatment prior to hydrolysis, the fermentable sugar (glucose) produced from hydrolysis of cellulose is fermented for bioethanol.

In Malaysia, one of the most abundant resources available are the cellulose content from various parts of palm tree (*Elaeis Guineensis*). Palm residues particularly coming from palm oil industry such as palm frond and palm trunk contain high content of cellulose of 50.78% and 41.88%, respectively (Lai and Idris, 2013).

In order to fully utilize the lignocellulosic biomass by making cellulose accessible for hydrolysis, there must be an efficient pretreatment method for a deconstruction of biomass (da Costa Lopes *et al.*, 2013). Pretreatment of the lignocellulosic biomass at an earlier stage before producing bioethanol is important for practical cellulose conversion processes. There are several pretreatment methods which can be classified into 4 categories which are physical (pulverization, irradiation), chemical (acid, alkali, ionic liquid, oxidizing agents, organic solvent treatments), physicochemical (steam explosion, supercritical fluid, wet oxidation treatments) and biological (pretreatment by using microorganism in lignin degradation such as white and soft-rod fungi (Mosier *et al.*, 2005). Among all of the pretreatment methods, ionic