UNIVERSITI TEKNOLOGI MARA

PARAMETRIC INFLUENCE ON TEREPHTHALIC ACID SYNTHESIS THROUGH HYDROTHERMAL APPROACH

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

Faculty of Chemical Engineering

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CONFIRMATION BY PANEL OF EXAMINERS

I certify that a Panel of Examiners has met on 27 August 2018t o conduct the final examination of Mohamad Zarqani Bin Yeop in his **Master of Science** thesis entitled "Parametric Influence On Terephthalic Acid Synthesis Through Hydrothermal Approach" in accordance with Universiti Teknologi MARA Act 1976 (Akta 173). The Panel of Examiner recommends that the student be awarded the relevant degree. The Panel of Examiners was as follows:

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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.

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ABSTRACT

Terephthalic acid (TPA) is a primary chemical intermediate used to manufacture polyester. Around 70% of the terephthalate feedstocks used worldwide are produced by using the commercial Amoco process. The importance of this process is evident from the fact that almost 100% of new TPA commercial plants have adopted this method. However, this process has led to health and environment concern due to the use of bromine promoter and acetic acid solvent. So far, several process improvements for TPA production have been proposed by previous researchers and hydrothermal process is known as one of the most promising route. In the hydrothermal method, high temperature water was employed in replacement of acetic acid organic solvent as reaction medium. The advantages of hydrothermal method include environmental friendly, cheap, naturally abundant and easy to handle. Hence, the aim of the experiment is to investigate the influence of the process parameters on the TPA yields at specified conditions. In this work, TPA was synthesized under subcritical conditions (250°C, 300°C and 350°C) and supercritical condition (400°C) using *batch micro-bomb reactor* by varying parameters such as water loading, hydrogen peroxide (H_2O_2) and manganese bromide ($MnBr_2$) catalyst loading while reaction time was set at 60 minutes. The TPA yield was quantitatively analysed using high performance liquid chromatography (HPLC), meanwhile the presence of main functionality groups in the TPA product was analysed using fourier transform infrared spectroscopy (FTIR). The overall composition of the product obtained was identified and confirmed using gas chromatography-mass spectrometer (GC-MS). The optimum TPA yield of 94.56 % was achieved at 350°C with combination parameters of 2.5 mL water, 1.5 mL hydrogen peroxide and 2.0 mL manganese bromide respectively. The result shows that hydrothermal method provides an alternative route for *p*-xylene oxidation to produce TPA at comparable yield with conventional route using suitable combination of *p*-xylene, hydrogen peroxide, manganese bromide and water loadings. In addition to the batch system result, a *continuous micro-bomb reactor* system was also successfully designed, developed and commissioned for future works. The process of designing the new hydrothermal system was based on the parameters obtained from the *batch micro-bomb reactor* in this work. The pressure vessel as a main unit of the continuous system was locally fabricated and adapted from ASME BPV (2007) design code. The development of this system is essential to study in details other aspects of TPA synthesis that was not possible using the batch system.

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