UNIVERSITI TEKNOLOGI MARA

SYNTHESIS, CHARACTERIZATION AND CATALYTIC PERFORMANCE OF MCM-41 AND SBA-15 FROM BOTTOM ASH OF TG BIN POWER PLANT

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ABSTRACT

Coal electric generator produced huge amount of waste which can be categorized into bottom and fly ash. In this research, bottom ash was chose instead of fly ash because very limited research was reported. Bottom ash known to have high percentage of silica has been utilized to produce mesoporous which are MCM-41 and SBA-15. The prepared mesoporous silica was then functionalized with 3-aminopropyltriethoxy-silane (APTES) and the catalytic performance of functionalized mesoporous silica was determined in the oxidation reaction of cyclohexene. The percent yield of silica extracted from bottom ash via alkali fusion found to be 23% and used in synthesis of MCM-41 and SBA-15. As comparison, TEOS was used as silica source. APTES is needed to increase the hydrophobicity of framework MCM-41 and SBA-15 thus enhanced their catalytic performance. Nitrogen adsorption-desorption of P MCM-41, P SBA-15 and BA SBA-15 shows type IV isotherm while BA MCM-41 exhibit type III isotherm. Calcined BA MCM-41, NH_2 BA MCM-41, calcined BA MCM-41, NH_2 BA MCM-41, calcined P SBA-15 and NH_2 P SBA-15 characterized using XRD revealed the well ordered (100), (110) and (200) peak which confirmed hexagonal structure. FTIR spectra showed that the organosilane group still retained in the pore channels even after functionalized with APTES.FESEM micrograph shows that all purely synthesis MCM-41 and SBA-15 exhibit worm-like morphology whilst the bottom ash synthesized MCM-41 and SBA-15 have agglomerated images. Then, catalytic performance of catalysts was tested in liquid phase of cyclohexene oxidation using tert-butylhydroperoxide as oxidant. The catalysts found to be active by giving the results of conversion were 84.05%, 62.49%, 57.38% and 43.74% for NH_2 P MCM-41, NH_2 P SBA-15, NH_2 BA SBA-15 and NH_2 BA MCM-41, respectively. 2-cyclohexen-1-one was found as major product whilst 2-cyclohexen-1-ol as minor product. This study was presented the ability of bottom ash supernatant to replace TEOS as silica source. The best catalytic activity is shown by NH_2 P MCM-41 in oxidation reaction of cyclohexene.

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CHAPTER ONE
INTRODUCTION

1.1 Research Background

The rising price of raw materials and consciousness towards environmental issues increased the demand of waste materials in industry. Recycling and reutilization of waste materials were effective routes to develop sustainable, environment-friendly, and cost effective industrial processes (Chiang, Ghyselbrecht, Santos, Meesschaert, & Martens, 2012). One of the industries which produce abundance of waste is coal-electrical generator. This residue was classified as bottom ash and fly ash conferring to the zone where it is recovered from (Benavidez, Grasselli, & Quaranta, 2003; Chandrasekar, You, Ahn, & Ahn, 2008). Almost 80% of ash trapped in the gas flow and discovered as fly ash while the remaining 20% is a granular, dark and porous material which found at the bottom of furnace known as bottom ash (Benavidez et al., 2003). In recycling systems, fly ash has a high recycling rate and can be use in various parts while 79% of bottom ash thrown and only 21% was used in asphalt or in construction. Researchers had considerable interest in conversion of coal bottom ash and fly ash into zeolites or mesoporous silica materials as MCM-41, MCM-48 and SBA-15 by change the silica source with silica extracted from these waste (Chiang et al., 2012; Park, Youn, Yang, & Ahn, 2012). The different combinations of activation solution, ash sources, pressures, reaction time and temperature can obtained various types of porous materials (Chiang et al., 2012).

Mesoporous MCM-41 has relatively large and tunable pore size (14-100Å), long range and high surface area of ordered pore structure which give advanced advantages over zeolites (Huang, Huang, Xiao, & Eić, 2007). Santa Barbara Amorphous-15 (SBA-15) which have similar properties as MCM-41 can be used as catalyst for wide applications due to hydrothermally stable, highly ordered mesopores, thick wall, large surface area and greater pore volume (Rahmat et al., 2010). SBA-15 and its relative SBA-16 mesoporous silica were prepared in a highly acidic medium by using poly (ethylene oxide)-poly (propylene oxide)-poly (ethylene oxide) (EO_n-PO_m-EO_n) type triblock copolymers. They demonstrated enhanced hydrothermal stability over M41S silica due to their larger uniform channels ranging from 5 to 30