Cyclic Acetalization of Furfural on Porous Aluminosilicate Acid Catalysts

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ABSTRACT

Porous aluminosilicate materials included microporous and mesoporous ZSM-5, hierarchical aluminosilicates, and mesoporous aluminosilicate were tested for acetalization of furfural (furan-2-carbaldehyde) with propylene glycol. The existing synthesis methods for aluminosilicate and ZSM-5 were modified to produce aluminosilicate material with hierarchical porous structure. Catalytic activity in acetalization of furfural by propylene glycol were conducted by refluxed of the mixture of furfural, propylene glycol and catalyst, using toluene as solvent and nitrobenzene as internal standard, at 106 °C for 4 h. The result showed that a combination of two structure directing agents, tetrapropylammonium hydroxide (TPAOH) and cetyltrimethylammonium bromide (CTAB) and modification of catalytic crystallization produced an active aluminosilicate framework that provides a wide access for a bulky reactants and strong acid sites to catalyze the reaction. The pore structure and the strength of the Brønsted acid sites were crucial for the high conversion of furfural to produce a cyclic acetal.

Keywords: *acetalization; furfural; propylene glycol; porous aluminosilicates*

ABSTRAK

Material aluminosilikat yang meliputi ZSM-5 mikropori dan mesopori, aluminosilikat berpori hirarkis, dan aluminosilikat mesopori diuji aktivitasnya untuk asetalisasi furfural (furan-2-karbaldehida) dengan propilena glikol. Metode sintesis material alumonisilikat berpori dan ZSM-5 dimodifikasi untuk menghasilkan material aluminosilikat dengan struktur pori hirarkis. Reaksi katalitik katalis dalam asetalisasi furfural dilakukan dengan merefluks campuran furfural, propilena glikol, dan katalis, menggunakan toluena sebagai pelarut dan nitrobenzena sebagai standar internal, pada suhu 106 °C selama 4 jam. Hasil penelitian menunjukkan bahwa kombinasi dua agen pengarah struktur, tetrapropilamonium hidroksida (TPAOH) dan setiltrimetilamonium bromida (CTAB) dan modifikasi kondisi kristalisasi menghasilkan kerangka aluminosilikat yang dapat diakses oleh reaktan dan memiliki sisi asam yang kuat untuk reaksi katalisis. Struktur pori dan sisi asam Brønsted merupakan hal yang paling berpengaruh untuk mengkonversi furfural menjadi asetal siklis.

Kata Kunci: *asetalisasi; furfural; propilena glikol; aluminosilikat berpori*

INTRODUCTION

In general, acetalization is a viable method to protect carbonyl functionalities in organic compounds [1] and important process in industry and has been widely used to produce active additive in fragrances [2]. The industrial needs of acetal compound especially to be used as additive in fragrances has prompted this study to find alternative ways for designing a simpler synthesis approach and environmentally friendly catalysts [3].

Acetals include a number of 'amber' chemicals. These materials are so-called due to their resemblance to *ambergris*, a material formed in the stomach of whales probably as a protection against intestinal damage by the 'shelly' parts of plankton. The material is occasionally found washed up on beaches, but the major source was the whaling industry. Unsurprisingly, this is now a rare and expensive material, driving the search for synthetic alternatives [4].

Furfural (furan-2-carboxaldehyde) is produced from plant residues which are rich in pentoses. This is one of important oxygen-containing heterocyclic aroma chemicals occurring in almost every type of food flavor and has a sweet caramel-like, nutty, baked bread,

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