

## **IMOBILISASI Ni(II) DAN Co(II) PADA SILIKA MESOPORI TERFUNGSIONALISASI AMINA SEBAGAI KATALIS DALAM REAKSI ASETILASI BENZIL ALKOHOL**

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### **INTISARI**

Sintesis material silika mesopori terfungsionalisasi amina dan terimobilisasi Ni(II) dan Co(II) telah dilakukan. Tujuan dari penelitian ini adalah sintesis silika mesopori berbasis abu sekam padi terfungsionalisasi amina dan terimobilisasi Ni(II) dan Co(II) serta penggunaannya sebagai katalis dalam reaksi asetilasi benzil alkohol. Sintesis material katalis diawali dengan preparasi silika mesopori melalui metode sol-gel dengan prekursor natrium silikat serta penambahan PEG-40 sebagai pencetak pori. Selanjutnya, fungsionalisasi amina dilakukan dengan menggunakan (3-aminopropil)trietoksilasilan (APTES) sebagai sumber amina melalui metode sonikasi. Imobilisasi Ni(II) dan Co(II) dilakukan dengan sonikasi dengan garam  $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  dan  $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  sebagai sumber ion nikel dan ion kobalt. Material hasil sintesis dikarakterisasi menggunakan difraksi sinar X (XRD), mikroskop pemindai elektron-spektrometer dispersi energi sinar X (SEM-EDX), spektrofotometer sinar inframerah (FTIR), *Thermogravimetric Analyzer* (TGA), dan *Surface Area Analyzer* (SAA). Pengujian aktivitas katalitik material dilakukan pada reaksi asetilasi benzil alkohol dengan asetat anhidrida. Nilai %konversi benzil alkohol ditentukan dengan kromatografi gas (GC).

Hasil penelitian menunjukkan bahwa silika mesopori terfungsionalisasi amina terimobilisasi Ni(II) dan Co(II) ( $\text{SiO}_2/\text{APTES}/\text{Ni}(\text{II})$  dan  $\text{SiO}_2/\text{APTES}/\text{Co}(\text{II})$ ) berhasil disintesis berdasarkan hasil karakterisasi dari puncak khas pada difraktogram XRD dengan didukung oleh hasil dari spektra FT-IR. Material hasil sintesis memiliki diameter pori rata-rata 12,83 dan 13,09 nm. Hasil studi komputasi menunjukkan bahwa silika memiliki karakter amorf dengan panjang ikatan Si-O sebesar 1,62 Å dan sudut ikat Si-O-Si dan O-Si-O masing-masing sebesar 129,09-157,24° dan 102,14-111,14°. Fungsionalisasi APTES pada silika memberikan perubahan pada panjang dan sudut ikat hasil interaksi antara silika dan APTES. Hasil uji aktivitas katalisis menunjukkan bahwa material  $\text{SiO}_2/\text{APTES}/\text{Ni}(\text{II})$  dan  $\text{SiO}_2/\text{APTES}/\text{Co}(\text{II})$  masing-masing mampu mengkonversi 98,34% dan 92,97% benzil alkohol dalam reaksi asetilasi benzil alkohol dengan asetat anhidrida sebagai agen pengasetilasi pada suhu reaksi 45 °C dan waktu reaksi 5 jam. Penggunaan katalis  $\text{SiO}_2/\text{APTES}/\text{Ni}(\text{II})$  sebanyak 3 kali pengulangan didapatkan %konversi benzil alkohol yang tidak berubah secara signifikan (98,34%; 95,20%; dan 90,61%) serta struktur yang stabil.

Kata kunci: katalis,  $\text{SiO}_2/\text{APTES}/\text{Ni}(\text{II})$ ,  $\text{SiO}_2/\text{APTES}/\text{Co}(\text{II})$ , asetilasi, benzil asetat

## **IMMOBILIZATION OF Ni(II) AND Co(II) ON AMINE-FUNCTIONALIZED MESOPOROUS SILICA AS CATALYST FOR BENZYL ALCOHOL ACETYLATION REACTION**

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### **ABSTRACT**

Synthesis of amine functionalized and immobilized Ni(II) and Co(II) mesoporous silica materials has been carried out. The aim of this research is to synthesize mesoporous silica based on rice husk ash functionalized amine and immobilized Ni(II) and Co(II) and its use as a catalyst in the acetylation reaction of benzyl alcohol. The synthesis of the catalyst material was started with the preparation of mesoporous silica using the sol-gel method with sodium silicate precursors and the addition of PEG-40 as a template. Furthermore, amine functionalization was carried out using (3-aminopropyl)triethoxysilane (APTES) as an amine source through sonication method and finally Ni(II) and Co(II) was immobilized by sonication method with Ni(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O and Co(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O salts as sources of nickel and cobalt ions. The synthesized material was characterized using X-Ray Diffractometer (XRD), Scanning Electron Microscope-Energy Dispersive X-Ray Spectrometer (SEM-EDX), FT-IR Spectrophotometer, Thermogravimetric Analyzer (TGA), and Surface Area Analyzer (SAA). The catalytic activity of the material was investigated in the acetylation reaction of benzyl alcohol with acetic anhydride. The %conversion value of benzyl alcohol was determined by Gas Chromatography (GC).

Results showed that amine-functionalized and Ni(II) and Co(II)-immobilized mesoporous silica (SiO<sub>2</sub>/APTES/Ni(II) and SiO<sub>2</sub>/APTES/Co(II)) were successfully synthesized based on the characterization results of the typical peaks on the XRD diffractogram and supported by the result of FT-IR spectra. The synthesized materials have an average pore diameter of 12.83 and 13.09 nm. Computational study results exhibited an amorphous silica with a Si-O bond length of 1.62 Å and Si-O-Si and O-Si-O bond angles of 129.09-157.24 and 102.14-111.14°, respectively. APTES functionalization on silica changes the length and bond angle resulting from the interaction between silica and APTES. The catalytic activity test results showed that SiO<sub>2</sub>/APTES/Ni(II) and SiO<sub>2</sub>/APTES/Co(II) materials were able to convert 98.34% and 92.97%, respectively, of benzyl alcohol in the acetylation reaction of benzyl alcohol with acetic anhydride as an acetylating agent at 45 °C for 5 hours. The use of SiO<sub>2</sub>/APTES/Ni(II) catalyst for 3 repetitions obtained %conversion values with insignificant reduction (98.34%, 95.20%, and 90.61%) and the structure of catalyst remained stable.

Keyword: catalyst, SiO<sub>2</sub>/APTES/Ni(II), SiO<sub>2</sub>/APTES/Co(II), acetylation, benzyl acetate