PREPARASI KATALIS NANOPARTIKEL $Fe_3O_4/SiO_2/N\text{-}SO_3H$ UNTUK SINTESIS TURUNAN IMIDAZO[1,2-*a*]PIRIDIN

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INTISARI

Sintesis nanopartikel $Fe_3O_4/SiO_2/N\text{-}SO_3H$ dan uji aktivitasnya sebagai katalis asam dalam sintesis senyawa turunan imidazo[1,2-*a*]piridin telah dilakukan. Penelitian diawali dengan mensintesis Fe_3O_4 dengan metode sono-kopresipitasi. Fungsionaliasi dilakukan dengan berturut-turut mereaksikan tetraetil ortosilikat (TEOS) dan 3-kloropropil trimetoksisilan (CPTMS). Reaksi substitusi nukleofilik dengan dietilenetriamina (DETA) menghasilkan nanopartikel $Fe_3O_4/SiO_2/NH_2$. Sulfonasi dilakukan dengan menggunakan 1,3-propana sulton untuk menghasilkan gugus asam sulfonat. Nanopartikel $Fe_3O_4/SiO_2/N\text{-}SO_3H$ dikarakterisasi dengan menggunakan *X-Ray diffractometer* (XRD), *fourier transform infrared spectrometer* (FTIR), *scanning electron microscope-energy dispersive X-ray mapping* (SEM-EDX *mapping*), *transimission electron microscope* (TEM), *vibrating sample magnetometer* (VSM) serta dilakukan penentuan angka keasaman katalis dengan *temperature programmed desorption of ammonia* (NH_3-TPD). Uji aktivitas katalitik nanopartikel $Fe_3O_4/SiO_2/N\text{-}SO_3H$ sebagai katalis heterogen dilakukan dengan mensintesis senyawa turunan imidazo[1,2-*a*]piridin melalui reaksi multikomponen berbahan dasar fenilglioksal, anilin dan 2-aminopiridin. Parameter yang dioptimasi meliputi variasi jenis pelarut, jumlah katalis, waktu reaksi, dan temperatur. Senyawa produk reaksi multikomponen dikarakterisasi dengan spektrometer ^{1}H-NMR, ^{13}C-NMR, dan FTIR-ATR.

Hasil penelitian menunjukkan bahwa nanopartikel $Fe_3O_4/SiO_2/N\text{-}SO_3H$ memiliki sifat magnetik dengan momen magnetik sebesar 32,8 emu/g sehingga dapat dipisahkan dari medium cair menggunakan magnet eksternal. Nanopartikel $Fe_3O_4/SiO_2/N\text{-}SO_3H$ memiliki angka total keasaman sebesar 6,29 mmol/g. Kondisi reaksi optimum diperoleh saat reaksi multikomponen dilakukan dalam pelarut DMSO dengan menggunakan 50 mg katalis pada 80 °C selama 6 jam. Elusidasi struktur menggunakan ^{1}H-NMR, ^{13}C-NMR, dan FTIR menunjukkan bahwa produk reaksi multikomponen adalah *N*,2-difenil imidazo[1,2-*a*]piridin-3-amina.

Katakunci: fenilglioksal, imidazo[1,2-*a*]piridin, nanopartikel $Fe_3O_4/SiO_2/N\text{-}SO_3H$.

PREPARATION OF NANOPARTICLES CATALYST $Fe_3O_4/SiO_2/N\text{-}SO_3H$ FOR SYNTHESIS OF IMIDAZO[1,2-a]PYRIDINE DERIVATIVE

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ABSTRACT

Synthesis of $Fe_3O_4/SiO_2/N\text{-}SO_3H$ nanoparticles and tests of their activity as an acid catalyst in the synthesis of imidazo[1,2-*a*]pyridine derivative compounds have been conducted. The research began by synthesizing Fe_3O_4 by the sono-coprecipitation method. Functionalization was carried out by successively reacting tetraethyl orthosilicate (TEOS) and 3-chloropropyl trimethoxysilane (CPTMS). Nucleophilic substitution reactions with diethylene triamine (DETA) produce $Fe_3O_4/SiO_2/NH_2$ nanoparticles. Sulfonation is carried out by using 1,3-propane sulton to produce a sulfonic acid group. $Fe_3O_4/SiO_2/N\text{-}SO_3H$ nanoparticles were characterized using X-Ray diffractometer (XRD), fourier transform infrared spectrometer (FTIR), scanning electron microscope-energy dispersive X-ray mapping (SEM-EDX mapping), transimission electron microscope (TEM), vibrating sample magnetometer (VSM) and determination of the acidity rate of the catalyst by temperature programmed desorption of ammonia (NH_3-TPD). The catalytic activity of $Fe_3O_4/SiO_2/N\text{-}SO_3H$ nanoparticles as heterogeneous catalyst was carried out by synthesizing imidazo[1,2-*a*]pyridine derivative compounds through multicomponent reactions based on phenylglioxal, aniline and 2-aminopyridine. The optimized parameters include the variation of solvent type, number of catalysts, reaction time, and temperature. Multicomponent reaction product compounds are characterized by 1H-NMR, 13C-NMR, and FTIR-ATR spectrometers.

The results showed that $Fe_3O_4/SiO_2/N\text{-}SO_3H$ nanoparticles have magnetic properties with a magnetic moment of 32.8 emu/g so that can be separated from the liquid medium using external magnets. $Fe_3O_4/SiO_2/N\text{-}SO_3H$ nanoparticles have a total acidity of 6.29 mmol/g. Optimal reaction conditions are obtained when a multicomponent reaction is performed in a DMSO solvent using 50 mg of a catalyst at 80 ◦C for 6 hours. The structure elucidation using ^{1}H-NMR, ^{13}C-NMR, and FTIR showed that the multicomponent reaction product was *N*,2-diphenyl imidazo[1,2-*a*]pyridine-3-amine.

Keywords: phenylglyoxal, imidazo[1,2-*a*]pyridine, nanoparticles $Fe_3O_4/SiO_2/N\text{-}SO_3H$