Pengaruh Variasi Katalis Heterogen Zeolit dan Asam Homogen dalam Sintesis Senyawa 7-Hidroksi-4-Metilkumarin menggunakan Metode Konvensional, Iradiasi Gelombang Mikro dan Ultrasonik = Synthesis of 7-Hydroxy-4-Methylcoumarin with Various Heterogenous Acidic Zeolites and Homogenous Acids using Conventional, Microwave, and Ultrasonic Irradiation Methods

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Abstrak

Senyawa 7-hidroksi-4-metilkumarin (hymecromone) merupakan senyawa heterosiklik turunan kumarin yang memiliki banyak aktivitas biologis. Hymecromone disintesis melalui reaksi kondensasi Pechmann antara resorsinol dan etil asetoasetat dengan variasi katalis asam dan metode. Katalis asam heterogen zeolit disintesis melalui reaksi kalsinasi dari NH4/MOR, NH4/Y dan NH4/USY dan katalis H-ZSM-5 didapat secara komersil lalu dikarakterisasi dengan FT-IR, NH3-TPD, XRD, SEM-EDX, dan BET. Katalis asam homogen yang digunakan adalah H3BO3 dan AlCl3 sebagai asam Lewis serta asam p-toluensulfonat monohidrat (PTSA.H2O) dan H2SO4 sebagai asam Brønsted. Reaksi dijalankan dengan metode konvensional (pengadukan), Microwave Assisted Organic Synthesis (MAOS), dan Ultrasound Assisted Organic Synthesis (UAOS) dengan variasi jumlah katalis, suhu, dan waktu reaksi. Kemudian produk dikarakterisasi dengan FT-IR, UV-Vis, NMR, dan LC/MS. Katalis heterogen H/Y memberikan yield optimum sebesar 8,06% dengan kondisi reaksi perbandingan mol reaktan 1:1, jumlah katalis 10 wt%, waktu 9 jam, suhu 80 oC menggunakan metode konvensional. Zeolit H/Y kemudian digunakan dalam studi reaksi recycle sebanyak 3 kali menggunakan metode UAOS dan menunjukkan kemampuan untuk digunakan berulang kali. Katalis homogen PTSA.H2O memberikan yield optimum sebesar 76,15% dengan kondisi reaksi perbandingan mol reaktan 1:1, konsentrasi katalis 10%, waktu 3 menit, suhu 70 oC menggunakan metode MAOS. Zeolit memberikan pilihan alternatif untuk katalis yang ramah lingkungan, mudah dipisahkan, dan dapat digunakan berulang kali.

.....7-Hydroxy-4-methyl coumarin (hymecromone) is a heterocyclic coumarin with a wide range of biological activities. In this study, hymecromone was successfully synthesized by reacting resorcinol and ethyl acetoacetate with several acidic catalysts using various methods. Acidic zeolite catalysts were synthesized by calcination of NH4/MOR, NH4/Y, and NH4/USY, while H/ZSM-5 were commercially obtained. All zeolites then were characterized by FT-IR, NH3-TPD, XRD, SEM-EDX, and BET. The homogenous catalysts used were H3BO3 and AlCl3 as Lewis acids, as well as p-toluene sulfonic acid monohydrate (PTSA.H2O) and H2SO4 as Brønsted acids. Reactions were conducted using conventional heating with stirring, Microwave-Assisted Organic Synthesis (MAOS), and Ultrasound-Assisted Organic Synthesis (UAOS) with variations in catalyst amount, temperature, and reaction times. The product was characterized using FT-IR, UV-Vis, NMR, and LC/MS. The heterogeneous catalyst H/Y afforded an optimum yield of 8.06% with the reaction conditions as follows: 1:1 (mole/mole) of reactant ratio, 10 wt% of catalyst, 9 hours of reaction time at 80oC using a conventional method. H/Y was recycled 3 times and showed good recyclability. The homogenous catalyst PTSA.H2O gave the optimum yield of 76.15% with the reaction conditions as follows: 1:1 (mole/mole) of reactant ratio, concentration catalyst of 10%, 3

minutes of reaction time at 70oC using the MAOS method. In conclusion, zeolites offer an alternative for a more environmentally friendly catalyst that is easy to separate and recyclable.