

PREPARASI, KARAKTERISASI, DAN APLIKASI NANOKATALIS ZEOLIT ALAM TERSULFATASI UNTUK SINTESIS NITROBENZENA

Aldino Javier Saviola
19/439166/PA/18989

INTISARI

Material katalis nanozeolit dan nanozeolit tersulfatasi telah berhasil dipreparasi, dikarakterisasi, serta diaplikasikan sebagai pengganti peran katalis asam sulfat dalam sintesis nitrobenzena. Zeolit alam dipreparasi menjadi serbuk berdimensi nanometer kemudian diimbangkan dengan asam sulfat dengan variasi konsentrasi 0, 1, 2, 3, dan 4 M. Katalis dikalsinasi pada suhu 500 °C dengan dialiri gas N₂. Semua material katalis dikarakterisasi menggunakan instrumen FTIR dan XRF serta diuji keasamannya dengan NH₃-TPD. Katalis nanozeolit dan nanozeolit tersulfatasi dengan keasaman terbaik selanjutnya dikarakterisasi menggunakan instrumen XRD, SEM-EDX, TEM, SAA, dan TGA. Sintesis nitrobenzena dilakukan sebanyak *triplo running* dalam reaktor *batch microwave* menggunakan katalis nanozeolit dan nanozeolit tersulfatasi dengan keasaman terbaik untuk menguji aktivitas dan *reusability* katalis. Produk hasil reaksi dikarakterisasi dengan instrumen GC-MS untuk menentukan selektivitas katalis.

Hasil penelitian menunjukkan bahwa nanozeolit memiliki nilai keasaman sebesar 1,742 mmol/g dan nilai rasio Si/Al sebesar 4,67. Variasi konsentrasi 2 M menghasilkan nanozeolit tersulfatasi dengan nilai keasaman terbaik yaitu sebesar 1,625 mmol/g dan nilai rasio Si/Al sebesar 6,44. Nanozeolit tersulfatasi H₂SO₄ 2 M memiliki struktur kristalin dengan nilai derajat kristalinitas sebesar 59,52%, kandungan sulfur sebesar 0,63%, ukuran partikel rata-rata sebesar 44,37 nm, luas permukaan sebesar 115,392 m²/g, volume dan diameter pori masing-masing sebesar 0,112 cm³/g dan 3,888 nm, dan perubahan massa sebesar 8,79%. Volume produk yang dihasilkan menggunakan katalis nanozeolit dan nanozeolit tersulfatasi H₂SO₄ 2 M berturut-turut adalah 7,167 ± 0,624 mL dan 10,167 ± 0,236 mL. Kedua nanokatalis bersifat selektif terhadap reaksi nitrasi benzena dengan nilai selektivitas sebesar 100%. Rata-rata konversi benzena yang dihasilkan untuk masing-masing nanokatalis sebesar 41,53% dan 58,92%. Katalis nanozeolit tersulfatasi H₂SO₄ 2 M memiliki *reusability* yang lebih baik untuk tiga kali siklus reaksi.

Kata kunci: katalis asam padat, nanozeolit tersulfatasi, nitrobenzena, reaktor *batch microwave*.

PREPARATION, CHARACTERIZATIONS, AND APPLICATION OF SULFATED NATURAL ZEOLITE NANOCATALYST FOR NITROBENZENE SYNTHESIS

Aldino Javier Saviola
19/439166/PA/18989

ABSTRACT

Nano zeolite and sulfated nano zeolite catalyst materials have been successfully prepared, characterized, and applied as a replacement for the role of a sulfuric acid catalyst in the synthesis of nitrobenzene. Natural zeolites were prepared into nanometer-sized powders and then impregnated with sulfuric acid with varying concentrations of 0, 1, 2, 3, and 4 M. The catalysts were calcined at 500 °C with N₂ gas. All catalyst materials were characterized using FTIR and XRF instruments and tested for acidity with NH₃-TPD. The nano zeolite and sulfated nano zeolite catalysts with the best acidity were further characterized using XRD, SEM-EDX, TEM, SAA, and TGA instruments. The synthesis of nitrobenzene was carried out in three runs in a batch microwave reactor by using nano zeolite and sulfated nano zeolite catalysts with the best acidity to test the activity and reusability of the catalysts. The reaction products were characterized by GC-MS instrument to determine the selectivity of the catalysts.

The results showed that nano zeolite has an acidity value of 1.742 mmol/g and a Si/Al ratio value of 4.67. The 2 M concentration variation resulted in sulfated nano zeolite with the best acidity value of 1.625 mmol/g and a Si/Al ratio of 6.44. The 2 M H₂SO₄-sulfated nano zeolite has a crystalline structure with a degree of crystallinity of 59.52%, a sulfur content of 0.63%, an average particle size of 44.37 nm, a surface area of 115.392 m²/g, pore volume and diameter of 0.112 cm³/g and 3.888 nm, respectively, and mass change of 8.79%. The volume of product produced using nano zeolite and 2 M H₂SO₄-sulfated nano zeolite catalysts were 7.167 ± 0.624 mL and 10.167 ± 0.236 mL, respectively. Both nanocatalysts are selective towards the nitration reaction of benzene with a selectivity value of 100%. The average benzene conversion produced for each nanocatalyst was 41.53% and 58.92%. The 2 M H₂SO₄-sulfated nano zeolite catalyst has better reusability for three reaction cycles.

Keywords: batch microwave reactor, nitrobenzene, solid acid catalyst, sulfated nano zeolite.