

SINTESIS NITROBENZENA MENGGUNAKAN NANOKATALIS BERBASIS SILIKA: PENGARUH BERAT NANOKATALIS SILIKA TERSULFATASI TERHADAP PRODUK NITROBENZENA

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INTISARI

Sintesis nitrobenzena menggunakan katalis asam padat telah dilakukan. Tujuan penelitian ini yaitu untuk mempelajari material $\text{H}_2\text{SO}_4/\text{SiO}_2$ sebagai katalis asam padat dalam sintesis nitrobenzena, dan mempelajari pengaruh variasi berat katalis silika tersulfatasi dalam sintesis nitrobenzena. SiO_2 dipreparasi dengan menggunakan metode *sol-gel* dari prekursor TEOS. Gel yang terbentuk kemudian direfluks dengan metanol, kemudian dikalsinasi pada suhu 600 °C. SiO_2 berukuran 200 *mesh* diimbaskan dengan H_2SO_4 98% dengan metode pengadukan selama 1 jam. Katalis $\text{H}_2\text{SO}_4/\text{SiO}_2$ 33% (b/b) yang dihasilkan dipisahkan dengan sentrifugasi dan dikeringkan. Kemudian, katalis dikalsinasi pada suhu 600 °C. Katalis dikarakterisasi menggunakan spektrofotometer *Fourier Transform Infrared* (FTIR), *X-Ray Diffraction* (XRD), *Surface Area Analyzer* (SAA) dan. Katalis $\text{H}_2\text{SO}_4/\text{SiO}_2$ yang telah dikarakterisasi, digunakan sebagai katalis asam padat dalam sintesis nitrobenzena. Variasi berat katalis yang digunakan yaitu 0,5; 1; dan 1,5 gr. Sintesis nitrobenzena dilakukan dengan perbandingan 1:3 benzena dan asam nitrat didalam reaktor *batch microwave* pada suhu 60 °C selama 5 jam. Produk cair nitrobenzena yang dihasilkan, dianalisis menggunakan GC-MS agar diketahui selektifitas dari katalis.

Hasil karakterisasi menunjukkan bahwa sintesis katalis $\text{H}_2\text{SO}_4/\text{SiO}_2$ telah berhasil dilakukan. Hal ini ditunjukkan dengan adanya puncak-puncak tertentu pada FTIR. Nilai 2 θ pada hasil XRD juga menunjukkan bahwa katalis $\text{H}_2\text{SO}_4/\text{SiO}_2$ telah berhasil disintesis. Dari penelitian ini, reaktor *batch microwave* memiliki efisiensi yang tinggi untuk sintesis nitrobenzena. Pada uji aktivitas dan selektifitas, penggunaan katalis $\text{H}_2\text{SO}_4/\text{SiO}_2$ sebanyak 1 gr menghasilkan nitrobenzena yang optimum yaitu sebanyak $2,02 \pm 0,024$ mL dengan rata-rata persentase konversi benzena 40,33%.

Kata kunci: Katalis asam padat, microwave, nitrasi, silika tersulfatasi.

***SYNTHESIS OF NITROBENZENE USING SILICA-BASED NANOCALYST:
THE EFFECT OF WEIGHT OF SULFATED SILICA NANOCATALYST ON
NITROBENZENE PRODUCTS***

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ABSTRACT

The synthesis of nitrobenzene using solid acid catalyst has been successfully synthesized. This research aims to study $\text{H}_2\text{SO}_4/\text{SiO}_2$ material as a solid acid catalyst in nitrobenzene synthesis, and the effect of weight variation sulfated silica catalyst in nitrobenzene synthesis. SiO_2 was prepared using the sol-gel method from the TEOS precursor. The gel formed was then refluxed with methanol, then calcined at 600 °C. SiO_2 with 200 mesh size was impregnated with 98% H_2SO_4 with a stirring method for 1 hour. The resulting 33% (w/w) $\text{H}_2\text{SO}_4/\text{SiO}_2$ catalyst was separated by centrifugation and dried. Then, the catalyst was calcined at a temperature of 600 °C. The catalyst was characterized using a Fourier Transform Infrared (FTIR) spectrophotometer, X-Ray Diffraction (XRD), and Surface Area Analyzer (SAA). The characterized $\text{H}_2\text{SO}_4/\text{SiO}_2$ catalyst was used as a solid acid catalyst to synthesize nitrobenzene. The variation in the weight of the catalyst used is 0.5; 1; and 1.5 gr. The synthesis of nitrobenzene was carried out in a 1:3 ratio of benzene and nitric acid in a microwave batch reactor at 60 °C for 5 hours. The resulting liquid nitrobenzene was analyzed using GC-MS to determine the selectivity of the catalyst.

The characterization results show that the synthesis of $\text{H}_2\text{SO}_4/\text{SiO}_2$ catalyst has been successfully carried out. This is indicated by the presence of certain peaks in the FTIR. The value of 2θ on the XRD results also indicates that the $\text{H}_2\text{SO}_4/\text{SiO}_2$ catalyst has been successfully synthesized. From this research, batch microwave reactor has high efficiency for nitrobenzene synthesis. On the activity and selectivity testing, the use of $\text{H}_2\text{SO}_4/\text{SiO}_2$ catalyst as much as 1 g produced the optimum nitrobenzene as much as 2.02 ± 0.024 mL with an average percentage conversion of 40.33% benzene.

Keywords: Microwave, nitration, solid acid catalyst, sulfated silica.