Sintesis senyawa berbasis spiropiperidin menggunakan nanokatalis Fe3o4 bernilai ekonomis = Synthesis of spiropiperidine-based compounds using economically of Fe3o4 nanocatalyst

Annisa Indah Reza, author

Abstrak

ABSTRAK
Senyawa turunan spiropiperidin berhasil disintesis meggunakan -diketon asam barbiturat/dimedon, formaldehid serta turunan anilin melalui kondensasi Mannich. Dua reaksi dinilai menghasilkan senyawa target senyawa 1 dan senyawa 3 , sementara satu reaksi lain menghasilkan dihidropiridin senyawa 2 . Hasil dikonfirmasi menggunakan karakterisasi FTIR, UV-Vis dan LC-MS. Sintesis senyawa menggunakan nanokatalis Fe3O4 dari proses dekomposisi termal karat besi dengan asam palmitat dalam pelarut paraffin. Paraffin sebagai pelarut memiliki titik didih tinggi dan dapat menggantikan pelarut umum seperti n-oktadekana. Penggunaan karat dan parrafin meningkatkan nilai ekonomis katalis yang dihasilkan. Karakterisasi FTIR pada katalis menunjukkan keberadaan asam palmitat sebagai capping agent mengelilingi Fe3O4. Data XRD memberikan ukuran rata-rata kristal 42,758 nm, sementara gambar TEM mengkonfimasi bentuk kristal berupa kubus dan unsur penyusunnya. Karakterisasi PSA memberikan indeks polidispersitas 0,391 dan Z-average 48,94. Dua senyawa hasil sintesis dioptimasi dalam suhu, waktu reaksi dan jumlah katalis berat yang berbeda. Hasil optimasi reaksi untuk senyawa 1 15-phenyl-2,4,10,12,15-pentaazadispiro[5.1.5.3]-hexadecane-1,3,5,9,11,13-hexa-one memberikan kondisi optimum pada suhu 50 oC, 4 jam reaksi dan 7,5 katalis dengan rendemen 69,30 . Sementara senyawa 2 10-4 chlorophenyl -9,10-dihydropyrido[2,3-d;6,5-d rsquo;]-dypyrimidine-2,4,6,8 1H,3H,5H,7H -tetra-one optimum pada suhu 50 oC, 4 jam dan 5 katalis dengan rendemen 72,34 . Studi penggunaan ulang katalis dilakukan pada senyawa 1 dan memberikan kecendrungan penurunan hasil rendemen dalam lima kali pengulangan.
<hr>
ABSTRACT
Spiropiperidine derivatives have been successfully synthesized using diketone such as barbituric acid dimedone, formaldehyde and aniline derivatives through Mannich condensation. Two reactions considered produced the targeted compounds compound 1 and compound 3 , while the other reaction produced dihydropyridine compound 2 . These results were confirmed using FTIR, UV Vis and LC MS. These compounds were synthesized using heterogenic Fe3O4 catalyst which was obtained from rust capped palmitic acid in paraffin solvent. Paraffin was chosen as solvent due to its high boiling point which was suitable to replace common solvents n octadecane. The use of rust and paraffin would increase catalyst rsquo s economic value. FTIR characterization of catalyst showed the presence of palmitic acid as capping agent surrounding Fe3O4. XRD data gave average crystal size of 42.758 nm, while the TEM image confirmed the crystal structure as cube and its constituent elements. PSA result gave polydispersity index of 0.391 and Z average 48,94. Two compounds were optimized under various of temperatures, times and amount of catalyst weight . Optimization condition for compound 1 15 phenyl 2,4,10,12,15 pentaazadispiro 5.1.5.3 hexadecane 1,3,5,9,11,13 hexa one came as 50 oC, 4 hours of time reaction and 7.5 catalyst with 69.30 of yield. Meanwhile compound 2 10 4 chlorophenyl 9,10 dihydropyrido 2,3 d 6,5 d 39 dypyrimidine

2,4,6,8 1H, 3H, 5H, 7H tetra one at 50 oC, 4 hours of time reaction and 5 catalyst yielded 72,34 . The study of reusable catalyst was conducted on compound 1 and resulted the yield rsquo s descending rsquo s tendency in each five times of repetitions.