

SINTESIS 3-BENZYLIDENISOINDOLIN-1-ON BIOACTIF DARI BENZILIDENFTALIDA MELALUI REAKSI ONE-POT MENGGUNAKAN RADIASI ULTRASONIK

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INTISARI

Telah dilakukan sintesis 3-benzildene-1-isoindolin secara one-pot dari benzildenftalida dengan metode sonikasi. Penelitian ini bertujuan untuk mempelajari pengaruh metode sintetik, efek sterik amina primer dan nukleofilisitas amina primer terhadap sintesis 3-benzilideneisoindolin-1-on.

Penelitian ini melibatkan sintesis reaksi one-pot yang dilakukan menggunakan radiasi ultrasonik pada suhu 50 °C. Reaksi dimulai dengan adisi nukleofilik amina primer ke 3-benzilidenftalida untuk menghasilkan zat antara 3-hidroksiisoindolin-1-on. Reaksi kemudian diikuti dengan penambahan larutan HCl 6M menyebabkan reaksi β -eliminasi dari 3-hidroksiisoindolin-1-on menjadi 3-benzilideneisoindolin-1-on. Produk dikarakterisasi dengan spectrometer $^1\text{H-NMR}$, $^{13}\text{C-NMR}$, FTIR dan CHNS analyzer.

Metode sonikasi menghasilkan 2-benzil-3-benzilideneisoindolin-1-on dengan persen hasil 75% dalam waktu 90 menit, sedangkan pemanasan konvensional membutuhkan 22 jam untuk persen hasil yang sama. Efek sterik dipelajari dengan menggunakan 2-metoksibenzilamina, 3-metoksibenzilamina dan 4-metoksibenzilamina. Pada 2-metoksibenzilamina dan 4-metoksibenzilamina keduanya didapatkan hasil 88%, sedangkan 3-metoksibenzilamina didapatkan hasil yang sedikit lebih rendah yaitu 82%. Efek nukleofilisitas dipelajari dengan menggunakan 4-metilbenzilamina, 4-fluorobenzilamina, furfuralamina, 2-tiofenemetilamina, dan butilamin. Persen hasil yang didapatkan untuk substrat masing-masing adalah 95%, 96%, 86%, 94%, dan 97%. Gugus amina yang sangat nukleofilik, seperti butilamina dan 2-tiofenemetilamina, menghasilkan hasil yang lebih tinggi, sedangkan substrat dengan nukleofilisitas yang lebih rendah, seperti furfuralamina menunjukkan hasil yang sedikit lebih rendah.



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ABSTRACT

The one-pot synthesis of 3-benzylideneisoindolin-1-ones from benzylidenephthalide and primary amines under ultrasound irradiation has been conducted. The objectives of the research were to study the effect of synthetic method, the steric effect of primary amines and the nucleophilicity of primary amines on the synthesis of 3-benzylideneisoindolin-1-ones.

The research involved a one-pot stepwise synthesis which was carried out under ultrasound irradiation at a temperature of 50 °C. The reactions began with the nucleophilic addition of primary amines to 3-benzylidenephthalide to generate the corresponding 3-hydroxyisoindolin-1-one intermediates within 30 min. This was followed by an acidic treatment with aqueous solution of HCl 6M which promoted the β -elimination reaction of the 3-hydroxyisoindolin-1-ones. The corresponding 3-benzylideneisoindolin-1-ones were generated within 1h of sonication. The products were elucidated with $^1\text{H-NMR}$, $^{13}\text{C-NMR}$, FTIR and CHNS analyzer.

The sonication method for the synthesis of 2-benzyl-3-benzylideneisoindolin-1-one gave a yield of 75% within 90 min, while the conventional heating took 22h for the same yield. The steric effect was investigated by using 2-methoxybenzylamine, 3-methoxybenzylamine, and 4-methoxybenzylamine. Both 2-methoxybenzylamine and 4-methoxybenzylamine produced the corresponding products in 88%, while 3-methoxybenzylamine gave a yield of 82%. The nucleophilicity effect of different substrates of 4-methylbenzylamine, 4-fluorobenzylamine, furfurylamine, 2-thiophenemethylamine and butylamine, were investigated. The yields for the substrates were 95%, 96%, 86%, 94%, and 97% respectively. Highly nucleophilic primary amines, such as butylamine and 2-thiophenemethylamine, resulted in higher yields, while amines with lower nucleophilicity like furfurylamine gave lower yields.

Keywords: one-pot synthesis, sonication, 3-benzylideneisoindolin-1-one, nucleophilicity, and steric effect.