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SINTESIS 3-BENZIL-2-BUTIL-3-HIDROKSIISOINDOLIN-1-ON MELALUI REAKSI TERINDUKSI CAHAYA
NABILA AZZAHRA, Dr. M. Idham Darussalam Mardjan, S.Si., M.Sc.;Dr. Tri Joko Raharjo, M.Si.
Universitas Gadjah Mada, 2018 | Diunduh dari http://etd.repository.ugm.ac.id/

SINTESIS 3-BENZIL-2-BUTIL-3-HIDROKSIISOINDOLIN-1-ON MELALUI REAKSI TERINDUKSI CAHAYA

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INTISARI

Sintesis 3-benzil-2-butil-3-hidroksiisoindolin-1-on melalui reaksi terinduksi cahaya telah dilakukan. Penelitian ini bertujuan untuk mempelajari pengaruh perlakuan terhadap sintesis 3-benzilidenftalida dan melakukan optimasi sintesis 3-benzil-2-butil-3-hidroksiisoindolin-1-on dengan memvariasikan jenis pelarut, jumlah amina dan perlakuan sintesis.

Penelitian ini diawali dengan sintesis 3-benzilidenftalida dari asam 2-iodobenzoat dan fenil asetilena melalui reaksi Sonogashira katalis CuI dan garam $NaHCO_3$ selama 24 jam. Reaksi dilakukan dengan tiga variasi perlakuan yaitu pemanasan pada 50 ^oC, kondisi gelap pada temperatur ruang dan radiasi sinar LED biru pada temperatur ruang. Produk 3-benzilidenftalida direaksikan dengan butil amina melalui reaksi adisi nukleofilik untuk mensintesis 3-benzil-2-butil-3-hidroksiisoindolin-1-on. Reaksi adisi nukleofilik dioptimasi dengan memvariasikan pelarut (DMSO dan *iso*-propanol), jumlah amina (1,3; 2; 3 ekuivalen), dan perlakuan (pemanasan 50 ^oC, gelap, radiasi LED biru). Produk dimurnikan dengan kromatografi kolom lalu dilakukan elusidasi struktur dengan spektrometer FTIR, GC-MS, ^1H-NMR dan ^{13}C-NMR.

Hasil penelitian menunjukkan bahwa perlakuan radiasi LED biru paling optimum untuk sintesis 3-benzilidenftalida dengan hasil sintesis sebesar 64%. Sedangkan, kondisi optimum sintesis 3-hidroksiisoindolin-1-on diperoleh dengan menggunakan *iso*-propanol sebagai pelarut, 2 ekuivalen amina dan radiasi LED biru sebagai perlakuan sintesis. Dengan melakukan reaksi pada kondisi optimum, produk 3-benzil-2-butil-3-hidroksiisoindolin-1-on diperoleh dengan hasil sebesar 71% sebagai padatan kuning dengan titik leleh 134-136 ^oC.

Kata Kunci : 3-benzilidenftalida, 3-hidroksiisoindolin-1-on, adisi nukleofilik, reaksi Sonogashira, reaksi terinduksi cahaya.

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SYNTHESIS OF 3-BENZYL-2-BUTYL-3-HYDROXYISOINDOLIN-1-ONE VIA PHOTO-INDUCED-REACTION

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ABSTRACT

Synthesis of 3-benzyl-2-butyl-3-hydroxyisoindolin-1-one via Photo-induced-Reaction has been conducted. The objectives of this research were to determine the optimum synthetic treatment of 3-benzylidenephthalide and to optimize the reaction conditions to synthesize 3-hydroxyisoindolin-1-one derivative including type of solvent, amount of amine and treatments.

This research was initiated by synthesizing 3-benzylidenephthalide from 2-iodobenzoic acid and phenylacetylene via Sonogashira coupling reaction using CuI as catalyst and $NaHCO_3$ as base for 24 hours. The reaction was performed using three different treatments, including heating at 50 ℃, dark condition and blue LED light radiation at room temperature. Preparation of 3-benzyl-2-butyl-3-hydroxyisoindol-1-one via nucleophilic addition between 3-benzylidenephthalide and butylamine. Nucleophilic addition reaction was optimized using various of solvents (DMSO and *iso*-propanol), amount of amine (1,3; 2; 3 equivalent) and treatments (heating at 50 ℃, dark condition and blue LED light radiation at room temperature). The products were finally purified using column chromatography, then elucidated using spectrometer FTIR, GC-MS, ^{1}H-NMR and ^{13}C-NMR.

The results showed that blue LED light radiation was the optimum treatment to synthesize 3-benzylidenephthalide with the yield of 64%. In addition, the optimum conditions to generate 3-hydroxyisoindolin-1-on were obtained using *iso*-propanol as solvent, 2 equivalent of amine and blue LED light radiation as synthesis treatment. By performing the reaction under the optimum conditions, 3-benzyl-2-butyl-3-hydroxyisoindol-1-one was afforded as yellow solid with the melting point of 134-136 ^{o}C in yield of 71%.

Key words: 3-benzylidenephthalide, 3-hydroxyisoindolin-1-one, photo-induced-reaction, nucleophilic addition, Sonogashira reaction.