SINTESIS 3-BENZILIDENFTALIDA TERINDUKSI CAHAYA DAN APLIKASINYA TERHADAP SINTESIS 2-(3-(1*H*-INDOL-3-IL)ETIL)-3-BENZIL-3-HIDROKSIISOINDOLIN-1-ON

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INTISARI

Sintesis senyawa 3-benzilidenftalida dan turunan 3-hidroksiisoindolin-1-on terinduksi cahaya telah dilakukan. Penelitian ini bertujuan untuk mensintesis 3-benzilidenftalida dengan melakukan optimasi reaksi yaitu variasi pelarut, jumlah alkuna, dan katalis, serta mensintesis 2-(3-(1*H*-indol-3-il)etil)-3-benzil-3-hidroksiisoindolin-1-on dari 3-benzilidenftalida dan triptamina.

Sintesis 3-benzilidenftalida dilakukan dengan mereaksikan asam 2-iodobenzoat dan fenilasetilena dengan katalis CuI melalui reaksi Sonogashira dan heterosiklisasi terinduksi cahaya LED biru selama 24 jam. Reaksi Sonogashira dioptimasi dengan memvariasikan pelarut (DMF:MeCN (1:1), DMSO, *i*-PrOH), jumlah alkuna (1,3; 2; 3 ekuivalen) serta jumlah katalis (10%, 20%, 100% mol). Tahap selanjutnya adalah sintesis turunan 3-hidroksiisoindolin-1-on, yakni 2-(3-(1*H*-indol-3-il)etil)-3-benzil-3-hidroksiisoindolin-1-on melalui reaksi adisi nukleofilik terinduksi cahaya antara amina primer (triptamina) terhadap 3-benzilidenftalida. Produk dikarakterisasi dengan spektrometer FTIR, GC-MS, ^{1}H-NMR, dan ^{13}C-NMR.

Kondisi optimum sintesis 3-benzilidenftalida diperoleh ketika reaksi Sonogashira dilakukan menggunakan DMSO sebagai pelarut, katalis CuI sebesar 100% mol dan 3 ekuivalen alkuna terminal. Dengan melakukan reaksi Sonogashira pada kondisi optimum tersebut, 3-benzilidenftalida dapat diperoleh dengan rendemen 67%. Senyawa 2-(3-(1*H*-indol-3-il)etil)-3-benzil-3-hidroksiisoindolin-1-on diperoleh sebagai padatan berwarna coklat muda dengan titik leleh sebesar 158-159 °C dan rendemen 68%.

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

PHOTO-INDUCED-SYNTHESIS OF 3-BENZYLIDENEPHTHALIDE AND ITS APPLICATION TOWARDS THE SYNTHESIS OF 2-(3-(1*H*-INDOLE-3-YL)ETHYL)-3-BENZYL-3-HYDROXYISOINDOLIN-1-ONE

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ABSTRACT

The photo-induced-synthesis of 3-benzylidenephthalide and 3-hydroxyisoindolin-1-one derivative had been done. The aims of this research were to optimize the synthesis of 3-benzylidenephthalide by varying solvent, number of alkyne and catalyst, as well as to synthesize 2-(3-(1*H*-indole-3-yl)ethyl)-3-benzyl-3-hydroxyisoindolin-1-one from 3-benzylidenephthalide and tryptamine.

The synthesis was started by reacting 2-iodobenzoic acid and phenylacetylene in the presence of CuI as catalyst *via* photo-induced-Sonogashira reaction for 24 h under blue LED irradiation. The Sonogashira reaction was studied by varying solvent (DMF:MeCN (1:1), DMSO, *i*-PrOH), number of alkyne (1.3, 2, 3 equivalent), and amount of catalyst (10%, 20%, 100% mol). The next step was the preparation of 3-hydroxyisoindolin-1-one derivative, *i.e.* 2-(3-(1*H*-indole-3-yl)ethyl)-3-benzyl-3-hydroxyisoindolin-1-one *via* photo-induced-nucleophilic addition between 3-benzylidenephthalide and tryptamine. The products were characterized by FTIR, GC-MS, ^{1}H-NMR, dan ^{13}C-NMR spectrometers.

The optimum condition for 3-benzylidenephthalide synthesis was obtained when the Sonogashira reaction was carried out in DMSO as solvent, 100% mol of CuI and 3 equivalent of terminal alkyne. By performing the reaction under the optimum conditions, 3-benzylidenephthalide was obtained in 67% yield. In addition, 2-(3-(1*H*-indole-3-yl)ethyl)-3-benzyl-3-hydroxyisoindolin-1-one was afforded as light brown solid with the melting point of 158-159 °C in 68% yield.

Keywords: photo-induced-reaction, 3-benzylidenepthalide, Sonogashira reaction, 3-hydroxyisoindolin-1-one, nucleophilic additon.