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On-water Catalyst-free Synthesis of 3-Alkenyl Oxindoles

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Abstract

3-Alkenyl oxindoles are key structural motifs in organic chemistry with a wide range of applications mainly in the pharmaceutical industry, due to excellent biological activities such as anti-inflammatory, antiviral, antibacterial, and anticancer properties. With the discovery of the importance of oxindole derivatives in the pharmaceutical industry, demand for sustainable protocols for the synthesis increased as most classical methods have their limitations and flaws. In this study on-water, catalyst-free, atom-economical green approach was developed for the synthesis of 3-alkenyl oxindole derivatives using oxindole and aldehyde as starting materials. The reaction between oxindole and benzaldehyde was conducted on water under catalyst-free conditions. Interestingly, 100% conversion was observed when the reaction was carried out for 24 hours at 100° C. As the next step, the applicability of the developed approach was proven for different aldehydes. Furaldehyde, cinnamaldehyde, and vanillin were used as the aldehydes to synthesize 3-alkenyl oxindole derivatives, and the isolated yields were obtained as 77%, 54%, and 51% respectively. The desired product formation was confirmed by comparing the Rf value of the product with that of the previously synthesized well-characterized compounds; 3-benzylideneindolin-2-one, 3-(furan-3-methylene)indolin-2-one, 3-(3-phenylallylidene)indolin-2-one, and 3-(4-hydroxy-3-methoxybenzylidene)indolin-2-one using Thin Layer Chromatography. Further characterization was done by Fourier-transform infrared (FT-IR), Ultraviolet-visible (UVVis), and Nuclear Magnetic Resonance (NMR) spectroscopic techniques. In further optimization steps, the reaction was performed using surfactants as the catalyst. By adhering to the green chemistry principles different sources of biosurfactants were used including *Acacia concinna* pods, *Sapindus emarginatus* fruit pericarp, *Dillenia retusa* fruit pericarp, and *Trigonella foenumgraecum* seeds. In order to compare the effect of biosurfactants, a synthetic surfactant, sodium dodecyl sulfate (SDS) was used as the catalyst. When the reaction was carried out for 21 hours at 100 °C using fruit pericarp of *Sapindus emarginatus* as the source of biosurfactant, 100% conversion was observed. Using water as the solvent, readily available starting materials, higher yield, and eco-friendliness and catalyst-free conditions suggest the possible use of the developed method for large-scale preparations of 3-alkenyl oxindole derivatives.

Keywords: Green protocol, Oxindole derivatives, Surfactant, On water synthesis, Catalyst-free