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Microwave Assisted Solvent-Free Green Approach to Synthesis of Oxindole Derivatives using Surface Modified Silica from Rice Husk Waste

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Abstract

Designing new synthetic schemes for synthesis of biomedical significant compounds in environmentally benign manner is an emerging challenge. Microwave assisted solvent-free synthetic methodology for drug synthesis address our future challenges by maximising atom economy and minimising by-product formation. Oxindole nucleus is a ubiquitous pharmacopore found in variety of pharmaceutical active compounds having extensive range of biological applications. There are different types of methodologies for synthesis of 3-benzylidene-indolin-2-ones by modifying oxindole nucleus with different functional groups. Major disadvantage associated with the above methods is usage of environmentally hazardous solvents. The present study introduce a novel green approach to synthesise 3-benzylidene-indolin-2-ones from naturally occurring aldehydes in solvent free conditions under microwave irradiation. (3-Aminopropyl) triethoxysilane (APTES) modified silica was used as the catalyst for the above reaction. Silica was extracted from rice husk waste collected from Polonnaruwa, Sri Lanka. Further it was activated using concentrated hydrochloric acid. APTES was immobilised on activated rice husk silica to attached amino functionality on the silica surface. Surface amino functionality of the APTES-Silica facilitate the reaction between oxindole and aldehydes. The reaction afforded targeted compounds in high yield within 12 minutes under the microwave irradiation. Percentage yield of the compounds A, B and C was 91%, 87%, 88% respectively. The silica supported catalyst was characterised by thermogravimetric analysis (TGA) and showed a similar weight loss as reported in the literature. Synthesised compounds were characterised by Fourier-Transform Infrared Spectroscopy (FTIR), Nuclear Magnetic Resonance Spectroscopy (^{1}H NMR) and melting point analysis. Melting points of the compounds A, B and C were 174^o C, 178^o C and 205^o C respectively. FTIR spectra showed that the characteristic peaks for the main functional groups present in the compounds. ^{1}H NMR spectra of the resulting compounds confirmed that the expected products were successfully synthesised. Microwave assisted synthesis is a rapid efficient and environmentally safe green method in the synthesis of 3-benzylidene-indolin-2-ones derivatives. Further silica obtained from rice husk waste can be effectively modified to use as a solid catalyst in the synthesis of biologically important compounds of medicinal interest.

Keywords: Microwave assisted, Solvent free, APTES-Silica, Oxindole, 3-Benzylidene-Indolin-2-Ones