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Green approach for the synthesis of chalcone (3-(4-fluorophenyl)-1-(4-methoxyphenyl) prop-2-en-1-one) using concentrated solar radiation

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ABSTRACT

The present work deals with the green solar assisted synthesis of chalcone (3-(4-fluorophenyl)-1-(4-methoxyphenyl)-prop-2-en-1-one). To check the efficacy of the process, chalcone has been synthesized by photochemical (UV radiation), thermal method (60 °C) and the conventional method. The effect of catalyst concentration and temperature on the yield of the product has been studied. The synthesized chalcone derivative was characterised using Fourier Transform Infrared (FTIR) Spectroscopy, Liquid chromatography–mass spectrometry (LCMS) elemental analysis, X-ray diffraction analysis (XRD) and thermal analysis (TG/DTA). The time required for the completion of the reaction using solar assisted method and conventional method were 10 min and 240 min (4 h), respectively. The crystallinity of material produced using concentrated solar radiation (CSR) was 14.16% higher than the conventional method. The amount of energy required to produce chalcone using CSR is 90.37% less energy than the conventional method. CSR was found to be green, simple and energy efficient novel idea for the synthesis of chalcone.

1. Introduction

Numerous techniques have been developed for the synthesis of organic materials. It is very important that any technique along with being high yield, ease of handling, uses minimum energy and that too using a renewable source of energy. To minimise the use of overall energy requirement, many people uses new technologies such as making use of microwave and UV irradiation or to utilise sonochemical methods. These methods use some kind of non-renewable source of energy and cannot be held for a long period; so, it is necessary to search for a reliable, green, and renewable source of energy.

Solar energy is available free of cost and it is a renewable source of energy. Solar energy is the unique clean, nontoxic and easily available source. The solar radiations emit a large number of ultraviolet as well as infrared radiations between the range of 280–4000 nm which serves both photochemical and thermal energy respectively. In this regards, solar energy is an able tool for the reaction and offers some advantageous. The usage of solar energy to execute the organic reactions has been reported heretofore for different photochemical reactions such as Diels-Alder reactions(Amin et al., 2015), Paterno-Buchi (Oelgemller et al., 2006), etc. Also, solar radiations used as catalyst for the synthesis of the metal oxides nanoparticles over the conventional methods have been reported (Patil et al., 2012); similarly, for the synthesis of organic materials there is need to develop the clean surrogate technology using solar energy for the conventional energy source which helps in solving the global warming issue.

Concentrated solar radiation (CSR) induced organic reaction chemistry is better as a non-conventional technique for the organic synthesis (Deshpande et al., 2015). Using CSR, A green method has been developed for the benzylic bromination (Deshpande et al., 2015). Also, the Regio- specific and Stereospecific (2p + 2p) Cycloadditions of arylethenes to 2-Substituted-1,4-naphthoquinones reported using solar method (Covell et al., 1998). Polyhydroquinoline derivatives also prepared by the solar irradiation (Mekheimer et al., 2008). The importance of CSR technique is that it is easy to achieve high temperature, can give high yields and is useful for rapid organic synthesis. That's why; we have tried to discover one of the versatile applications of solar energy on well-known Claisen-Schmidt reaction in this work. To the best of our awareness, the synthesis of chalcone using renewable energy source such as concentrated solar radiation has not been reported till date.

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Chalcone are the compounds which contain α, β unsaturated ketone functionality. It is also known as chalconoids. Chalcone are structurally simple compounds from the family of flavonoids which has a variety of biological and pharmacological applications (Joshi, 2005). It has a number of pharmacological activities such as anticancer (Valdameri et al., 2012), antileishmanial (Boeck et al., 2006). Antibacterial (Tran et al., 2012), antimicrobial (Bhuiyan et al., 2011), antimalarial (Liu et al., 2001), anti-inflammatory (Vogel et al., 2010), anti-tumor (Lee et al., 2016), anti-angiogenic (Lee et al., 2016), cytotoxic activity (Vogel et al., 2010), antioxidant (Vogel et al., 2010), antidiabetic (Hsieh et al., 2012), and antifungal (Bhuiyan et al., 2011) activities. Also, chalcone derivatives show corrosion inhibition properties(Verma et al., 2014).The number of heterocyclic compounds such as pyrazoles, pyrimidines, benzoxazepines, benzodiazepines, oxazoles and benzodiazepines has been synthesized from the intermediates of chalcone (Gelens et al., 2003). Recently it has been found that the chalcone are important naturally found with an extensively contribution in tea, spices, fruits, vegetables and soy-based foodstuff which have intense pharmacological properties (Di Carlo et al., 1999).

Previously, the synthesis of chalcone was reported using sodium hydroxide and potassium hydroxide as a catalyst which requires 12–18 h for completion of reaction has been reported (Liu et al., 2001). Also, Synthesis of the chalcone using sonochemical method has been reported (Jarag et al., 2011). As well as chalcone has been synthesized in using microwave irradiation (Hasany et al., 2012). But, such techniques have many disadvantages like long reaction time and lower energy efficiency. To overcome such problems and making economically as well as environmentally viable processes, there is a need to use renewable energy sources such as solar.

In this work, concentrated solar radiation (CSR) was used to synthesise the chalcone as well as it was synthesized by a conventional method for comparison purpose. Effect of UV light was also studied. To understand the role concentrated solar radiations, the effect of the same on reaction time, crystallite size, % crystallinity, % weight loss, the thermal stability of the product obtained has been studied. The present study is aimed at the development of environmentally benign, eco-friendly and energy efficient method for the synthesis of chalcone.

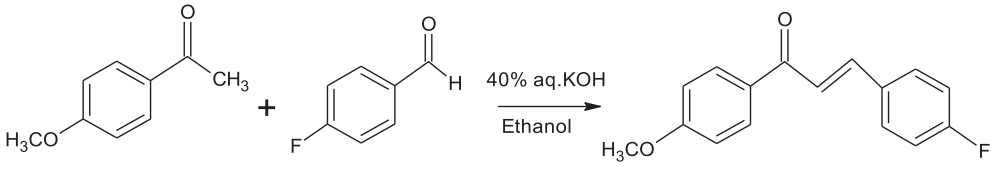
2. Materials and methods

2.1. Materials

4-Methoxyacetophenone LR (99.0%), potassium hydroxide pellets and ethanol were purchased from S d fine-chemicals Ltd. India, 4-fluorobenzaldehyde (98.0%) was obtained from Sisco research laboratory Pvt. Ltd. India and Hydrochloric acid (35–38.0%) was received from Thomas Baker India.

2.2. Reaction scheme

Synthesis of chalcone (3-(4-fluorophenyl)-1-(4-methoxyphenyl) prop-2-en-1-one) (3) by condensation of 4-methoxyacetophenone (1) with 4-fluorobenzaldehyde (2) using 40% potassium hydroxide as a catalyst is represented as follows:



Reaction Scheme

2.3. Concentrated solar radiation (CSR) set-up

Synthesis of chalcone (3-(4-fluorophenyl)-1-(4-methoxyphenyl) prop-en-1-one) was carried out by CSR, the set-up is made up of Fresnel lens as shown in Fig. 1. The round bottom flask was adjusted at the point of maximum concentration. All the experiments carried out in the month of February and March between 12:30 pm to 1:00 pm which is peak solar intensity timing. Stirring was carried out using magnetic stirrer of the mixture. The intensity of solar radiation was measured with the help of Pyranometer (Dynalab Tech. Ltd. India) i.e. solar radiation flux density (W/m^2). It also measures solar irradiation over a view of 180°.

2.4. Experimental procedures

The chalcone were synthesized by base catalysed Claisen-Schmidt condensation of acetophenone derivative and benzaldehyde derivative.

2.4.1. Synthesis of chalcone using concentrated solar radiation (CSR)

Initially, a solution of 4-methoxyacetophenone (13 mmol) in ethanol was taken in round bottom flask. Aqueous solution of potassium hydroxide (40%) was charged dropwise in this initially prepared solution of 4-methoxyacetophenone with constant stirring using a magnetic stirrer. The round bottom flask was fixed at the focal point of the Fresnel lens. After waiting for 5 min once the temperature of reaction mass reach at 58–60 °C, dropwise addition of a separately prepared solution of 4- fluoro benzaldehyde (13 mmol) in ethanol with constant stirring was accomplished. Reaction progress was monitored by thin-layer chromatography (TLC). After the completion of the reaction; the reaction mixture was quenched with ice-cold distilled water. The reaction mass was neutralised with 1 M HCl and was vacuum filtered using Buckner funnel by Whatman filter paper. The product obtained is in the form of yellowish-white- coloured powder. The obtained product was dried at 75 °C overnight and weighed.

2.4.2. Synthesis of chalcone using conventional method

Initially, a solution of 4-methoxyacetophenone (13 mmol) in ethanol was taken in round bottom flask. Forty percent potassium hydroxide was charged dropwise in the initially prepared solution