

Photoreduction of Benzophenone in Green Chemistry Using an Alternate Solvent Ethyl Alcohol

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Abstract

Green chemistry is an approach to the synthesis, processing and use of chemicals that reduces risks to humans and the environment. A synthesis of benzopinacol from benzophenone is carried out using ethanol as a solvent. This is a free radical reaction and reduction of benzophenone occurred by sunlight via UV radiation. Benzopinacol is a catalyst of the formation of unsaturated polyesters. It is also used as an organic synthesis intermediate and as an initiator of polymerization by free radicals. Characterization of Benzopinacol was done by using spectroscopic technique like IR, NMR etc. Results positively show that solvent ethyl alcohol can be used as an alternative for photoreduction of benzophenone in case of non-availability of isopropyl alcohol.

Keywords: Photoreduction; IR; NMR; Benzopinacol; Benzophenone; Free radical

Introduction

Prevention is better than cure

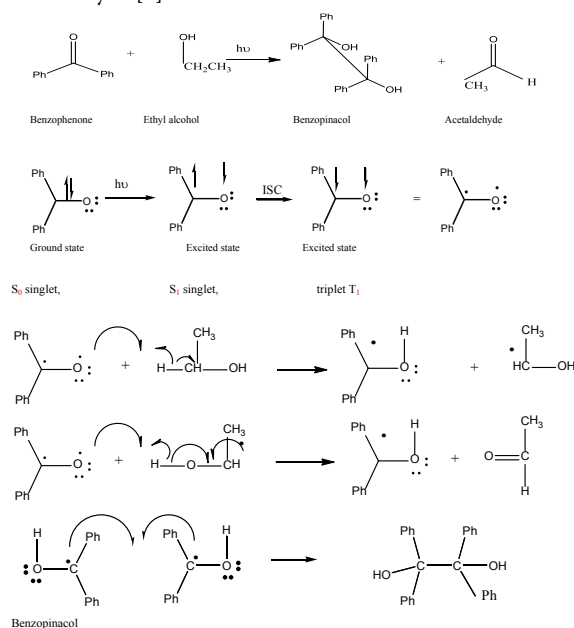
Green chemistry is the design of chemical products and processes that reduce or eliminate the use and generation of hazardous substances. It relies on a set of 12 principles that can be used to design or re-design molecules, materials and chemical transformations to be safer for human health and the environment [1,2]. The incorporation of green chemistry principles by University Grant commission New Delhi (2013-14), into the post graduate curriculum has fueled a fundamentally new approach to the teaching of chemistry. Green solvents are generally derived from renewable resources and biodegrade to innocuous, often naturally occurring product [3,4]. Ethyl alcohol is one of them. Solar energy is a renewable free source of energy that is sustainable and totally inexhaustible, unlike fossil fuels that are finite [5]. Generally, the term, photochemistry is used to describe a chemical reaction caused by absorption of ultraviolet (wavelength from 100 to 400 nm), visible light (400-750 nm) or infrared radiation (750-2500 nm). Photoreduction of benzophenone to benzopinacol is carried out by using alternate renewable source of solvent i.e., ethyl alcohol. Benzopinacol is a catalyst for the formation of unsaturated polyesters. It is also used as an organic synthesis intermediate and as an initiator of polymerization by free radicals. Bromoacetyl derivatives of benzopinacol are known as flame retardant initiators for the polymerization of unsaturated polyester (UPE) systems. Additionally, silyl and phosphorus ethers of benzopinacol as flame retardant initiators for unsaturated polyester UPE systems have been also reported [6].

Materials and Methods

The melting points determined by open capillary tube by electrical melting point apparatus and were uncorrected. FTIR spectra were recorded on Perkin Elmer (4000-400 cm^{-1}). ^1H NMR spectra were recorded on Bruker, 300 MHz, PMR Spectrophotometer using CDCl_3 as a solvent with TMS as an internal standard. The chemical shift are expressed in δ , ppm. All spectral characterizations were carried out at Central Drug Research Institute, Lucknow.

Mechanism in ethyl alcohol

Primary process is the excitation of benzophenone. Absorption of light of the appropriate wavelength around 350 nm for benzophenone generates free radicals. Ethyl alcohol acts as both solvent and reagent in this reaction. Ultra violet light is absorbed by the benzophenone and an intersystem crossing occurs to produce a diradical a molecule with two unpaired electrons. The benzophenone diradical then abstracts a hydrogen atom from the solvent and dimerises, yielding benzopinacol and acetaldehyde [7].



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Experimental

About 2 gm of benzophenone was allowed to dissolve in 5 ml of ethyl alcohol with gentle warming. After all solid was completely dissolved, the test tube was filled with ethanol to the neck and one drop of glacial acetic acid was added to prevent basic cleavage of the desired product. A cork was inserted and para film was wrapped around the mouth of the test tube and kept at sunny location to complete the reaction. After four to five days white shining crystals of benzpinacol were separate out. Chilled the test tube in an ice bath and product was collected by vacuum filtration. The crystals were dried overnight and melting point of final compound was determined by open capillary tube method. Calculate the percentage yield for product. Practical yield was found to be 40% [7-10].

Data analysis

IR: 1720 (C=O), 2945-75(=C-H str.), 2875(-C-H str.); benzophenone (Figure 1).

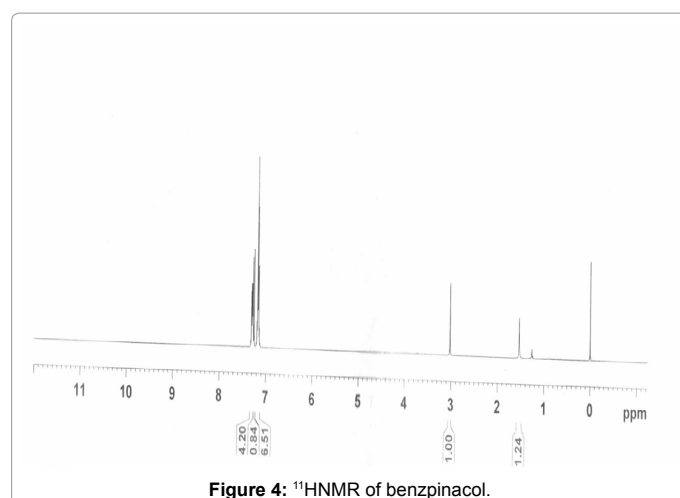
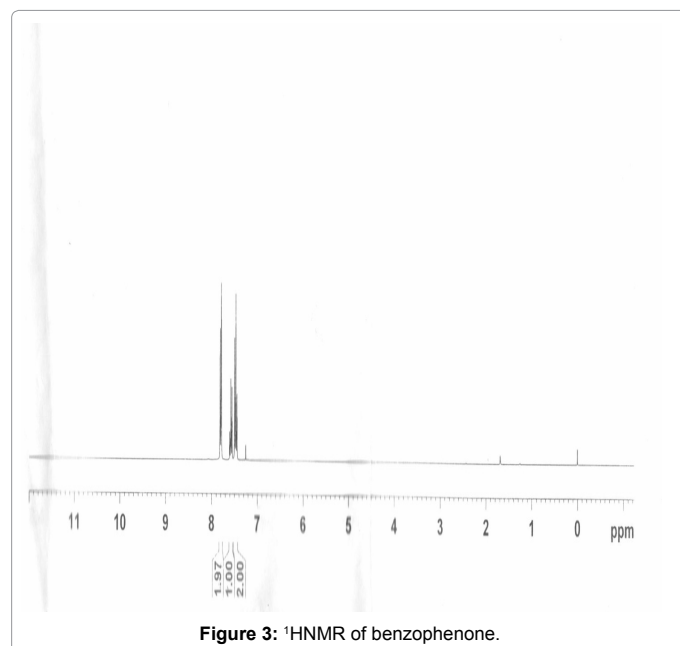
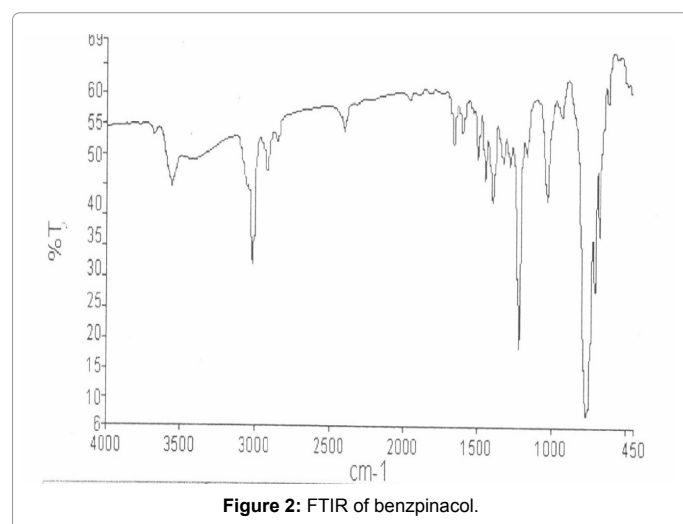
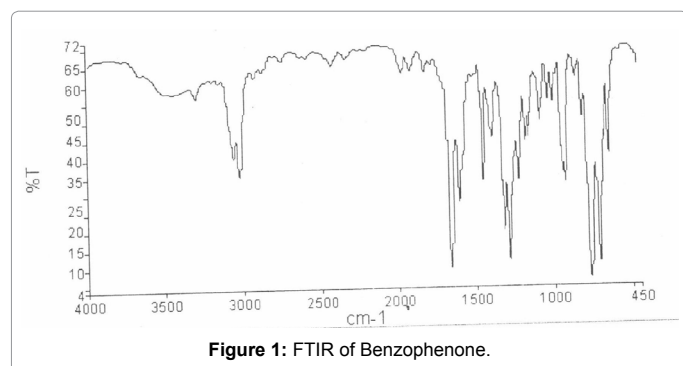
IR: 3400-3650 (O-H), 2945-75(=C-H str.), 2875(-C-H str.); benzpinacol (Figure 2).

$^1\text{H NMR}$: δ 7.0-7.5 (m 10 H, Ar-H), (Figure 3), melting point 48°C.

$^1\text{H NMR}$: δ 3.0 (s 2H, OH), 7.0-7.5 (m 20 H, Ar-H), (Figure 4), melting point 185°C.

Results and Discussion

In this experiment it is aimed to synthesise benzpinacol through



photoreduction of benzophenone catalysed by sunlight using ethyl alcohol as a solvent and reagent. Spectroscopic characterization of compound clearly indicates that benzpinacol was successfully synthesized. In IR spectra after seven days of reaction time carbonyl band at 1720 cm^{-1} disappeared and new absorption band at 3400-3650 cm^{-1} for OH appeared, shows that photoreduction of benzophenone to benzpinacol via sunlight has been completed. $^1\text{H NMR}$ spectra for benzpinacol indicates presence of singlet at δ 3.0 for OH proton Results positively show that solvent ethyl alcohol can be used as an alternative for photoreduction of benzphenone in case of non-availability of isopropyl alcohol.

Key Features of Reaction with Ethanol

According to news reports issued in March 2017 by Cleveland based industry research firm, The Fredonia Group, says global demand for green solvents derived from natural or renewable resources, will soon reach 2 billion pounds per year. In which ethanol will account for nearly half the growth in green solvents demand through 2020 and thus to remain as world's most prevalent green solvent [11].

- Solar energy is used as a source of catalyzing the reaction.
- Solvent derived from natural source have a minimal impact on the environment.
- Ethanol is cheaper solvent.

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