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RESEARCH ARTICLE

Synthesis and Spectral Characterization of Photoactive (2E, 6E) 4 -methyl- 2,6 bis (4 hydroxybenzylidene) cyclohexanone

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ABSTRACT

A novel photoactive solid, (2E, 6E) 4-methyl-2,6 bis(4-hydroxybenzylidene) cyclohexanone (MBHBC) was synthesized using 4-hydroxybenzaldehyde and 4-methyl cyclohexanone in the presence of boric acid and HCl as catalysts at 0°C. The purity of MBHBC was checked by HPLC (97.5%) and the structure was supported by FTIR, ^{1}H and ^{13}C NMR, MS and HPLC.

KEYWORDS

Photoactive bisphenol, IR, NMR, MS HPLC

INTRODUCTION

Dihydroxy compounds are the important constituents of plastics, epoxy resins and in manufacturing thermally stable polymers and polyester resins[1,2]. They are useful in manufacturing thermally stable polymers, epoxy resins, formaldehyde resins, etc. Polymers containing cinnamate, chalcone, coumarine, dibenzalacetone, and their derivatives both in main chain or side chain are used as photosensitive materials[3-5] and find their potential uses in devices for optical data storage, photo resists, and photolithographic assemblies[6-8]. To the best of our knowledge no work has been reported on (2E,6E)2,6-bis(4-hydroxy benzylidene)cyclohexanone (Scheme I). In present investigation it was thought to be of interesting to synthesize dihydroxy compound containing photosensitive group and characterized by spectral techniques.

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EXPERIMENTAL

Materials

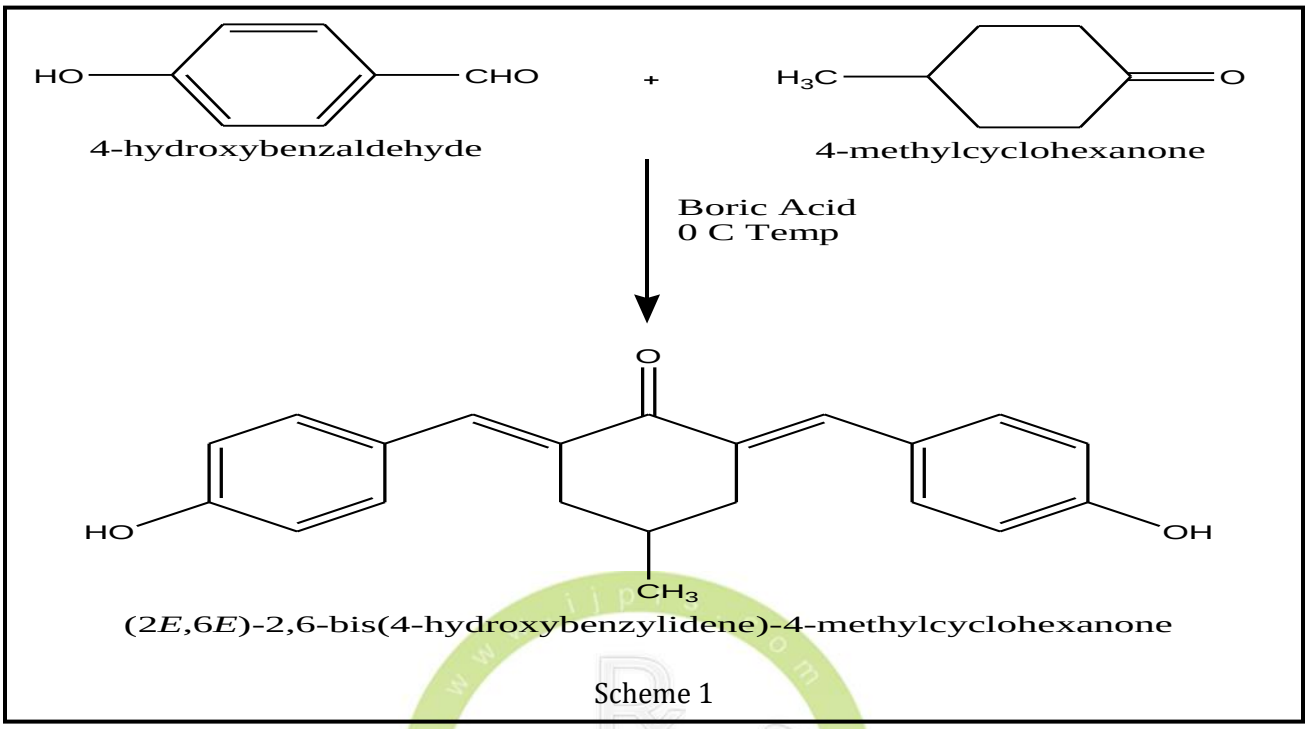
All the chemicals and solvents used were of L R grade and purified prior to their use[9]. 4-Hydroxybenzaldehyde (98%, LOBA Chemie), 4-methylcyclohexanone (99%, Spectrochem Pvt. Ltd. Mumbai), boric acid (99.5 % Allied Chemical Corporation, Vadodara) and HCl (37% Renkem) were used as received.

Preparation of photoactive diol [(2E,6E) 4-methyl-2,6-bis(4-hydroxybenzylidene)cyclo hexanone

Into a 250 ml two necks round bottom flask equipped with a mechanical stirrer and thermometer was placed in a thermostat bath. To this flask 0.1mol 4-hydroxy benzaldehyde and 0.1mol boric acid and 50ml conc. HCl were placed and stirred at 0°C for 10min. Then 0.05mol 4- methylcyclohexanone was added dropwise and the reaction mass was stirred at 0°C for 1 h with TLC monitoring. The greenish yellow product was isolated from chilled water, filtered, washed well with water until acid was completely removed and dried at 50°C.The product was repeatedly purified from dioxane-

water system. Hereafter product is designated as MBHBC. The yield of MBHBC was 80 %.

MBHBC decomposed before melting. The reaction scheme is shown as under:



Scheme 1

Measurements

Fourier transform infrared spectrum was scanned on a Shimadzu FTIR 8400 spectrometer over the frequency range from 4000-400 cm^{-1}. ^{1}H and ^{13}C NMR spectra were scanned on a Brucker Avance III 400 MHz NMR spectrometer using DMSOd6 as a solvent and TMS as an internal standard. Mass spectrum was scanned on a Shimadzu GC-MS QP 2010 spectrometer by using EI (0.7KV) detector. The ion source detector was 220^{o}C and interface temperature was 240^{o}C. Purity was checked on Shimadzu HPLC LC- 10 AT VP on diode array detector.

Results and Discussion

HPLC Analysis

High performance liquid chromatogram of MBHBC is shown in Fig.1from which it is observed that MBHBC showed 97.5% purity.

FTIR Spectral Analysis

FTIR spectrum of MBHBC is shown Fig.2. The characteristic absorption peaks (cm^{-1}) are assigned as follows: 3590 and 3358(O-H str.), 3092(=C-H str.), 1741(C=0 str.), 1674(-C=C- str.), 1467(C=C str. and C-H def.) 980 and 784(C-H oopd); and 671(C-H bend).

^{1}H NMR Spectral Analysis

^{1}HNMR spectrum of MBHBC is shown in Fig.3. The chemical shifts and types of protons are assigned as follows: 9.969 (s,2H,OH), 7.548 [s, 2H,=C-H], 7.420-7.398 (dd, 4ArH,J=8.8], 6.859-6.837(dd, 4ArH,J=8.8), 2.970-2.938(d,2H CH_2, J=12.8), 2.504[s, 2H CH_2],1.801[s, 1H CH] and 1.775(m, 3H CH_3).

^{13}CNMR Spectral Analysis

^{13}C NMR spectrum of MBHBC is presented in Fig.4. The chemical shift of different types of carbon atoms are as follows: 188.2, 158.3, 132.4, 126.4, 115.8, 38.8, 35.9, 28.8 and 21.4 ppm.

Mass Spectral Analysis

Mass spectrum of MBHBC is shown in Fig. 5. The important mass fragments are as follows: 320(M^{+}), 321(M+1), 322(M+2), 319(M-1), 303, 292, 291, 250, 249, 233, 199, 157, 146, 145, 144, 132, 131, 115, 107, 91, 77, 65 and 44 m/z.

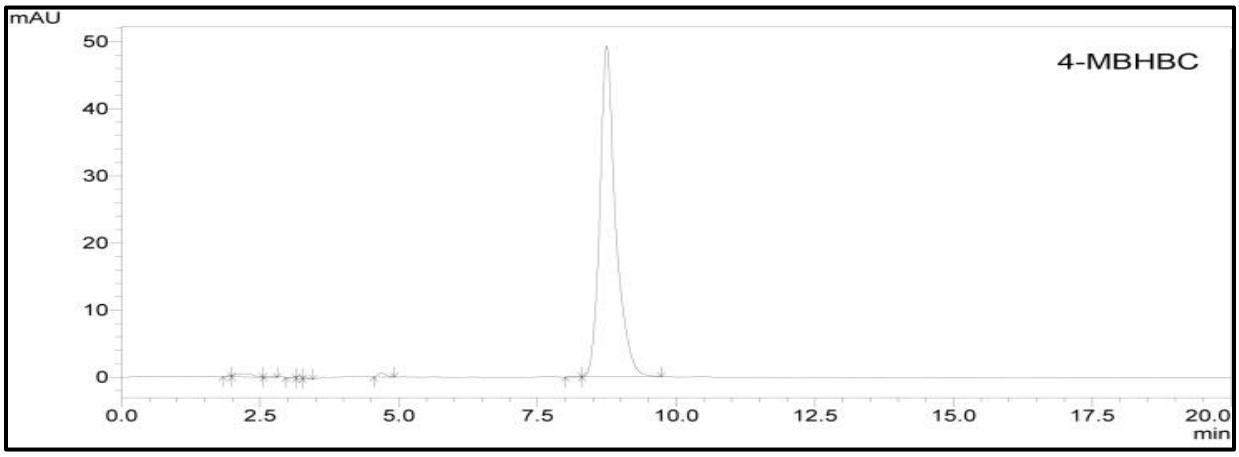


Figure 1: High performance liquid chromatogram of MBHBC

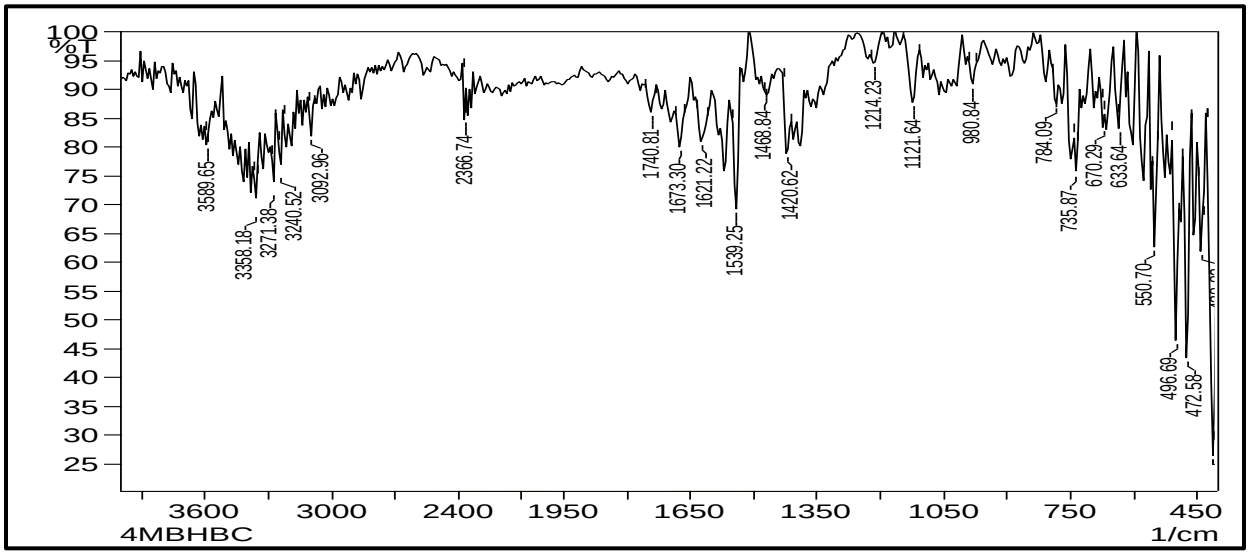


Figure 2: IR spectrum of MBHBC

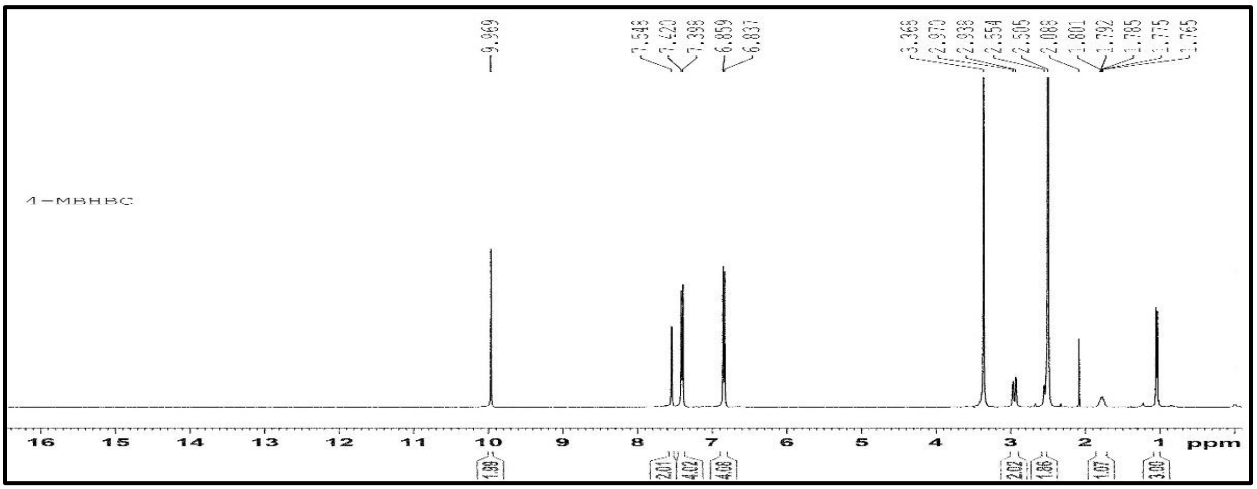


Figure 3: [1]HNMR spectrum of MBHBC

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