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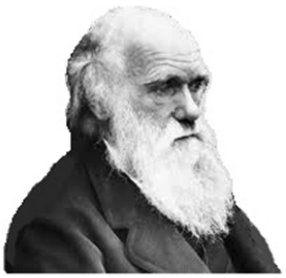

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Synthesis and spectral analysis of (E)-1-(2,4-dihydroxy phenyl)-3-(4-hydroxyphenyl) prop-2-en-1-one

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ABSTRACT

Multifunction chalcone: (E)-1-(2,4-dihydroxy phenyl)-3-(4-hydroxyphenyl)prop-2-en-1-one (THCH) was synthesized by reacting a 0.01 mol 2,4-dihydroxy acetophenone, and a 0.01 mol 4-hydroxybenzaldehyde using 10 ml of 60% aq. sodium hydroxide as a catalyst and 25 ml of ethanol as a solvent at reflux temperature for 3h. The THCH was crystallized using a water-methanol system. The structure of THCH was supported by UV-Vis, FTIR, ^{1}HNMR, ^{13}CNMR, and mass spectroscopic techniques.

Keywords: Chalcones, spectroscopic techniques

1. INTRODUCTION

Worldwide, chalcones and their derivatives are focused on the synthesis and biodynamic activities. They are found useful for the treatment of viral disorders, cardiovascular diseases, parasitic infections, pain, gastritis, and stomach cancer, food additives and cosmetic formulation ingredients, artificial sweeteners, scintillators, polymerization catalysts, fluorescent whitening agents, organic brightening agents, stabilizers against heat, visible light, ultraviolet light and aging [1-6].

In the modern era, traditional engineering materials like metals are constantly replaced by new polymeric materials with the advent of new technologies. Polymeric materials possess good to excellent physicochemical properties like strength-to-weight ratio, resistance to

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corrosion, low cost, low density, ease of availability, ability to form intricate shapes, durability, processability, transparency, electrical and thermal resistance, etc. Polymeric materials find their industrial applications in every aspect of life, from medicine to food, packaging to computers.

Bisphenols are useful for synthetic high-performance polymers like polyethers, polyesters, and thermosetting epoxy resins for various industrial applications. Epoxy resins are well-known for their excellent thermo-mechanical and electrical properties, high adhesion to many substrates, and good heat and chemical resistances [7]

Recently we have synthesized some diols containing chalcone moieties and used them for the synthesis of epoxy resins and their characterization [8-12]. We aimed to synthesize multifunctional chalcone for high-performance polymers. In the present work, we have synthesized (E)-1-(2,4-dihydroxy phenyl)-3-(4-hydroxyphenyl)prop-2-en-1-one and supported its structure by spectral techniques.

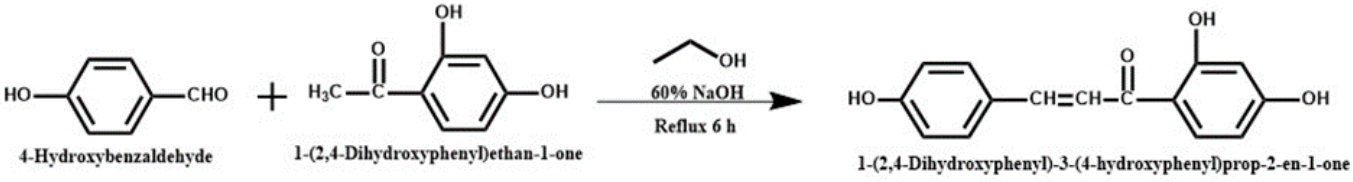
2. MATERIALS AND METHODS

2. 1. Materials

All the chemicals and solvents used were of LR grade and were used as received. 4-Hydroxybenzaldehyde, 2,4-dihydroxy acetophenone, Hydrochloric acid (37%), and sodium hydroxide were supplied by RENKEM and supplied by Sisco Pvt. Ltd. Mumbai

a. Synthesis of (E)-1-(2,4-dihydroxyphenyl)-3-(4-hydroxyphenyl)prop-2-en-1-one

A 100 ml round-bottomed flask equipped with a condenser was placed in a water bath. In this flask, a 0.01 mol 2,4-dihydroxy acetophenone, and a 0.01 mol 4-hydroxybenzaldehyde were dissolved in 25 ml ethanol and then 10 ml 60% aq. NaOH was added as a catalyst. The reaction mixture was brought to reflux and continued for 3 h and cooled to room temperature. The reaction mass was acidified to pH 1 using 1M aq. HCl. The separated product was filtered and washed well with distilled water till the filtrate was found neutral and dried in an oven at 50 °C. The product was crystallized three times from a methanol-water system. (E)-1-(2,4-dihydroxy phenyl)-3-(4-hydroxyphenyl)prop-2-en-1-one is designated as THCH. The yield and mp of THCH are 60% and 190 °C, respectively. The reaction scheme is as under.



Scheme 1. Synthesis of (E)-1-(2,4-dihydroxy phenyl)-3-(4-hydroxyphenyl)prop-2-en-1-one

b. Characterization methods

The ultraviolet-visible (UV-Vis.) spectrum of THCH in THF was scanned on a Shimadzu UV1700 over the wavelength range from 250-500 nm. The FTIR spectrum was scanned on a Shimadzu 1S-IR affinity FTIR spectrometer over the frequency range from 4000-600 cm^{-1}.

The ^{1}H NMR and ^{13}CNMR spectra were scanned on a 400 MHz Bruker AVANCE II spectrometer by using DMSOd6 as a solvent and TMS as an internal standard. The mass spectrum was recorded on a Shimadzu GCMS-QP-2010 spectrometer.

3. RESULTS AND DISCUSSION

The structure of THCH was supported by various spectroscopic techniques. UV-Vis, FTIR, ^{1}HNMR, ^{13}CNMR, and mass spectra.

UV-Vis spectrum of THCH is shown in Figure 1. THCH showed two absorption peaks at 261 and 368 nm, and are assigned as π-π* and n-π* transitions due to the presence of double bonds and lone pairs of electrons on oxygen atoms.

Figure 2 represents the IR (KBr) spectrum of THCH. The IR stretching and bending vibration frequencies are assigned as follows: 3399.96 and 3261.05 (O-H str.), 2948.35 (asym. C-H str.), 1631.04 (C=O str.), 1605.42 (C=C str.), 1438.88(C-H def.), 1026.36 (O-H def.) 1167.28 (C-O str.), 829.068 (C-H oopd).

A 400 MHz ^{1}HNMR spectrum in DMSOd6 is presented in Figure 3. The chemical shifts (ppm) due to different types of protons, their multiplicities, and their coupling constants (J) are assigned as follows: 6.430-6.409 [d,1H (o), J=8.4], 6.865-6.845[d,4H(c), J=8], 6.299 [s,2H(l)], 7.759 [s, 4H (d)], 8.184-8.162 [d,1H (p,f,g), J=8.8], 10.150 [s,1H (a)], 10.698-10.653 [d,1H (n), J=18], 13.614-13.528 [d, 1H (j), J=34.4].

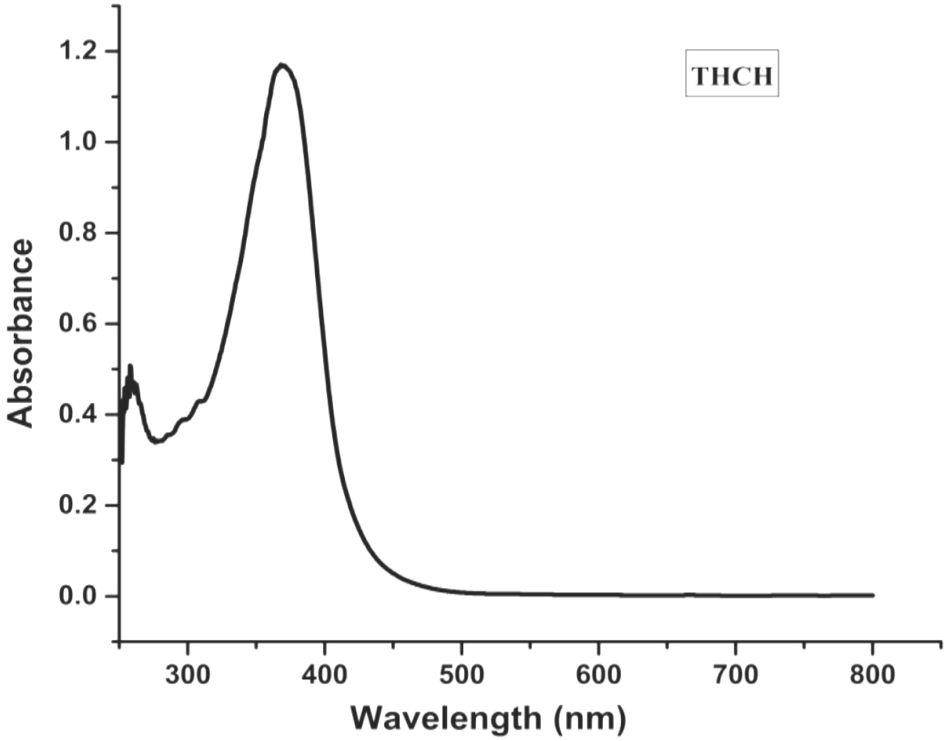


Figure 1. UV-Vis spectrum of 10^{-5} % THCH in THF.

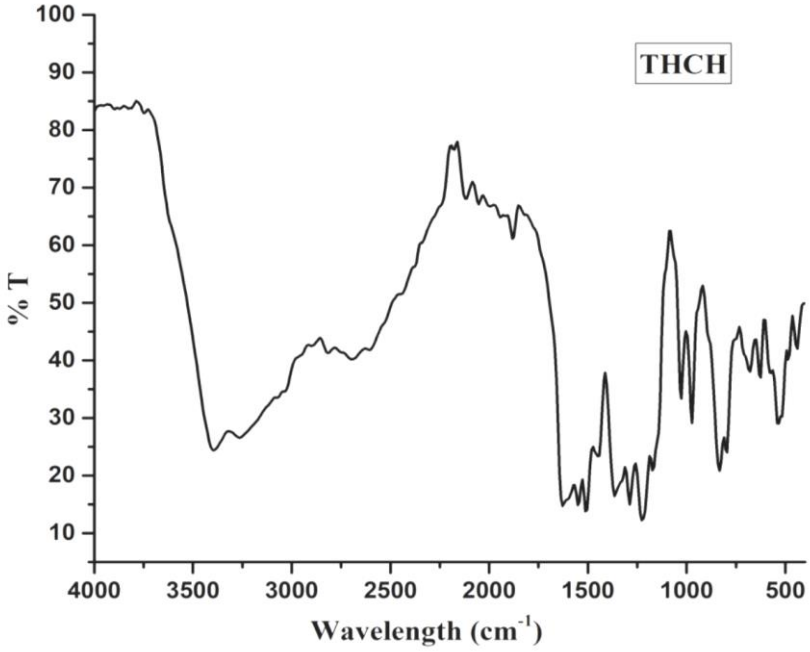


Figure 2. FTIR (KBr) spectrum of THCH.

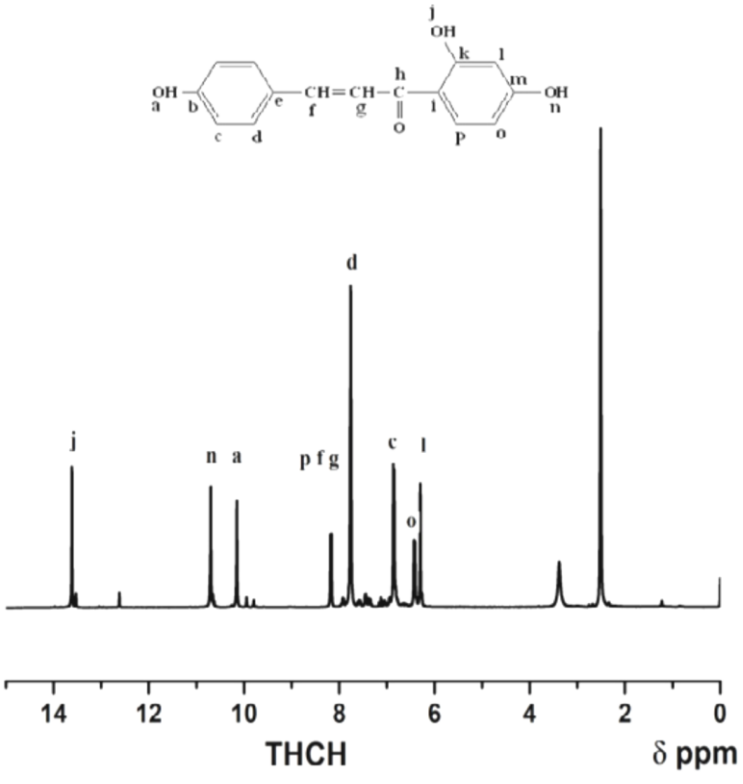


Figure 3. [1]HNMR (400 Mz) spectrum of THCH in DMSOd6.

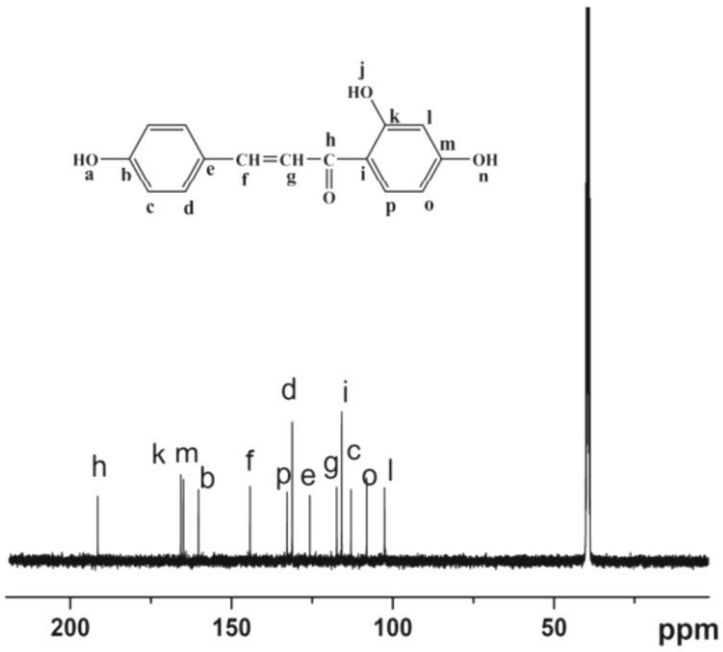


Figure 4. ^{13}C NMR (400 MHz) spectrum of THCH in DMSOd6.

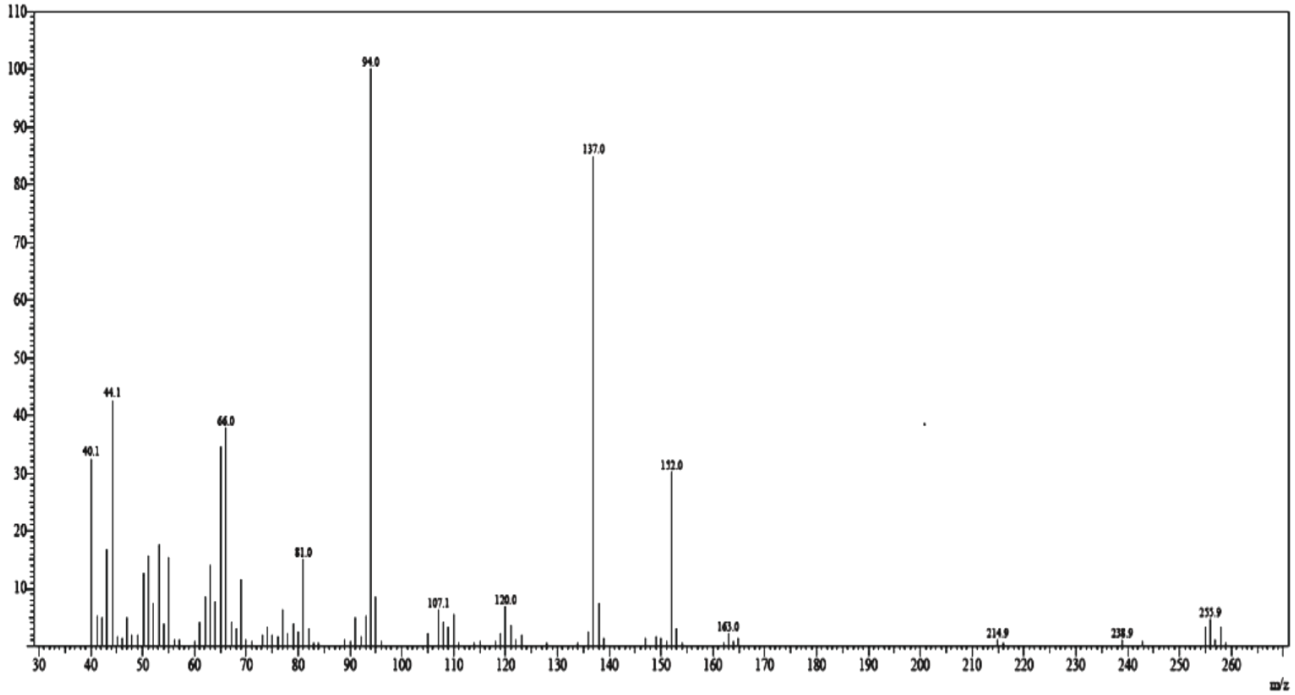


Figure 5. The mass spectrum of THCH.

Figure 4 shows the 400 MHz ^{13}C NMR spectrum in DMSO d6. The chemical shifts (ppm) of different types of carbon atoms are assigned as follows: 102.56(l), 108.07(o), 112.97(c), 115.82(i), 117.38(g), 125.72(e), 131.19(d), 132.81(p), 144.24(f), 160.26(b), 164.93(m), 165.76(k), 191.51(h).

The mass spectrum is depicted in Figure 5 and important mass fragments (m/z) are listed as follows: 258 (M^++2), 256(M^+), 255 (M^+-1), 239, 215, 163, 154, 139, 138, 121, 107, 95, 94 (BP), 81, 65, 64, 43, and 41.

IR spectral inspection provided expected stretching and bending vibrations, while ^{1}HNMR and ^{13}CNMR spectral analysis revealed the expected number of protons and carbon atoms in the compound. The mass spectral analysis furnished the expected molecular mass of THCH. Thus, spectroscopic techniques supported the structure of the compound under investigation.

4. CONCLUSIONS

A bioactive and industrially important (E)-1-(2,4-dihydroxy phenyl)-3-(4-hydroxyphenyl) propanone was synthesized and its structure was supported by various spectroscopic techniques.

Acknowledgments

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