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Synthesis and Structure Characterization of 1-(4-Bromophenyl)-3-(2-Chloro-6-Fluorophenyl) Prop-2-En-1-One Using Spectroscopic Techniques and Density Functional Theory

Virupakshi M. Bhumannavar[1], Parutagouda Shankaragouda Patil[2], Neelamma B. Gummagol[3]
[1]Hirasugar Institute of Technology, Nidasoshi-591236, Karnataka, India
[1, 3]K.L.E. Institute of Technology, Opposite Airport, Gokul, Hubballi-580030, Karnataka, India
[2]B.L.D.E.A'S, S. B. Arts and K. C. P. Science College Vijayapur-586103, Karnataka, India

Abstract: The structural confirmation of the 1-(4-Bromophenyl)-3-(2-chloro-6-fluorophenyl) prop-2-en-1-one compound is done by experimental techniques. Experimental techniques FTIR, proton NMR, UV-Visible, performed for the compound. The experimentally obtained results are compared with density functional theory obtained results. The decomposition and melting point of the compound is obtained by TGA & DTA. Density functional theory is performed for the 1-(4-Bromophenyl)-3-(2-chloro-6-fluorophenyl) prop-2-en-1-one compound B3LYP/6-311G++(d,p) basis set. Time dependent density functional theory calculated for three different methods B3LYP, Hartree-Fock and CAMB3LYP also employed for the 2C6FBC at 6-311G++(d,p) basis set.

Keywords: DFT Study, HOMO-LUMO, FTIR, ^{1}H NMR, TGA/DTA, chalcone

I. INTRODUCTION

Chalcones are found in plants and derivatives of heterocyclic rings like pyrimidines, isoxazoles, pyrazolines and cynopyridines [1]. They are synthesized by the method Claisen –Schmidt Condensation method. The chalcone have anti-inflammatory activity [2]. The chalcone have antioxidant and antibacterial [3-4]. The chalcone have microbial activity [5]. The chalcone have anti-infective [6]. Chalcone spectroscopic study is done for structure confirmation [7-9]. Infrared spectra analysis and thermal study of the chalcone are performed [10]. The HOMO-LUMO of the 2C6FBC molecule was performed by keeping B3LYP/6-311++G(d,p) point of theory [11]. The experimental and theoretical UV-Visible, FTIR spectral analysis is performed to chalcones[12-14].

II. EXPERIMENTAL

A. Synthesis

The compound 1-(4-Bromophenyl)-3-(2-chloro-6-fluorophenyl) prop-2-en-1-one (2C6FBC) be synthesized with Claisen –Schmidt Condensation method. 2–chloro-6-fluorobenzaldehyde (0.01 mol)) with 4-bromoacetophenone (0.01 mol) in methanol (60 ml) and mixture was treated with 5 ml of 30% sodium hydroxide solution and stirred at room temperature for 6 h. The precipitate obtained was poured into ice-cold water (500 ml) and left to stand for 5 h. The resulting crude solid was filtered, dried and recrystallized by slow evaporation. The following Fig.1 shows scheme of reaction.

B. Computational Study

Theoretical computations for title compound are done with GAUSSIAN 09W program. Optimized geometry of 2C6FBC molecule obtained at B3LYP with 6-311++G (d, p) basis set. Also DFT executed to with 6-311++G(d, p) basis set for 2C6FBC molecule at different levels B3LYP, HF and CAMB3LYP. FTIR and NMR are performed with same basis set. Results are visualized in Gauss View 5 software. Obtained results of title compound were compared and discussed.

C. Characterization

The 2C6FBC compound spectrum of FTIR, ^{1}H NMR and UV-Visible recorded. The analysis of thermal behavior, decaying and melting point of 2C6FBC was done by TG/DTA analysis.

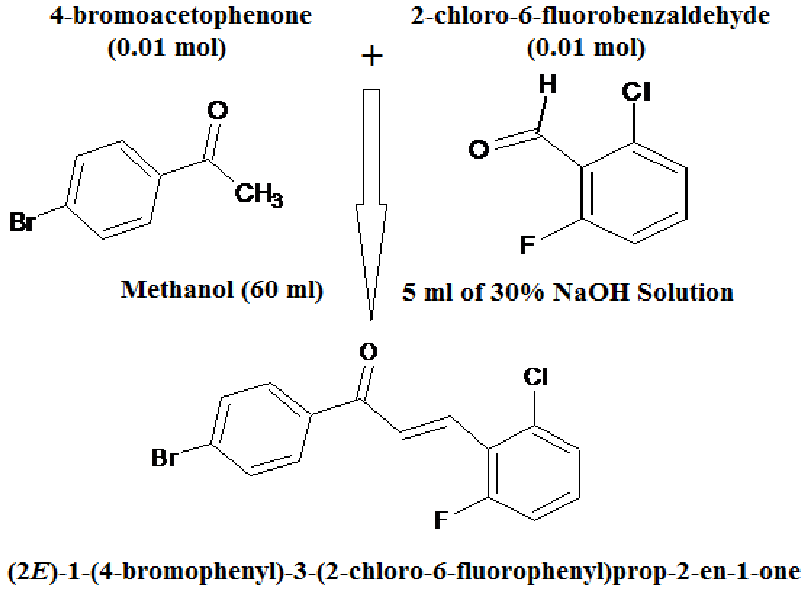

Fig.1. Synthesis scheme of 2C6FBC

III.RESULTS AND DISCUSSIONS

A. *Structure Characterization*

The optimization of geometry 2C6FBC compound carry out using DFT at B3LYP/6-311+G(d, p) level. Structure of molecule is given away in Fig. 2. From Molecule geometry bond angles, bond length and dihedral angles are obtained. The results obtained from theory and experimental XRD data are shown in Table 1. Patil et al.[15] report the XRD data of 2C6FBC.

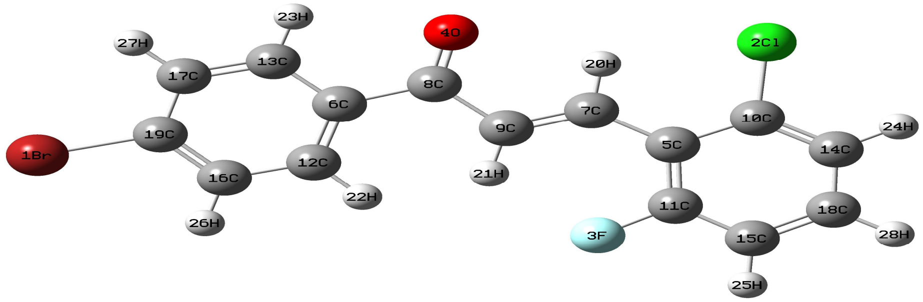

Fig. 2. Geometry optimized molecule of 2C6FBC

Table 1
Experimental (XRD data) and theoretically (DFT) determined bond length (Å), bond angles, and torsion angles (°) of 2C6FBC molecule

Bond length	DFT	XRD	Bond angle	DFT	XRD	Torsion angle	DFT	XRD
Br1– C19	1.91	1.90	C13–C6–C12	118.5	119.2	C6–C8–C9–C7	-180	-173.6
C6–C8	1.50	1.49	C16–C19–Br1	119.4	119.7	C6–C12–C16–C19	0.0	0.1
C8=O4	1.22	1.22	C17–C19–Br1	119.4	118.1	CL2–C10–C14–C18	180.0	178.8
C8—C9	1.49	1.49	C6–C8–O4	119.8	120.0	F3–C11–C15–C18	180.0	179.0
C9=C7	1.35	1.34	C5–10C–CL2	120.5	119.5	C10–C5–C11–F3	-180.0	-179.4
C7–C5	1.46	1.46	C5–C11–F3	118.8	118.2	C10–C5–C11–C15	0.0	0.0
C10–CL2	1.76	1.74	C7–C5–C11	124.5	124.3	C7–C5–C11–C15	180.0	178.4
C11—F3	1.35	1.35	C9–C7–H20	115.5	115.3	C12–C16–C19–Br1	180.0	178.5

The bond lengths of Br1– C19 is 1.91 Å (DFT) and 1.90 Å (XRD), C6–C8 is 1.50 Å (DFT) and 1.49 Å (XRD), C8=O4 is 1.22Å (DFT) and 1.22 Å (XRD), C8─C9 is 1.49 Å (DFT) and 1.49 Å (XRD), C9=C7 is 1.35 Å (DFT) and 1.34 Å (XRD), C7–C5 is 1.46Å (DFT) and 1.46 Å (XRD). The bond angles of C13–C6–C12 is 118.5 ° (DFT) and 119.2° (XRD), C16–C19–Br1 is 119.4 ° (DFT) and 119.7° (XRD), C17–C19–Br1 is 119.4 ° (DFT) and 118.1° (XRD), C6–C8–O4 is 119.8 ° (DFT) and 120° (XRD), C5–10C–CL2 is 120.5 ° (DFT) and 119.5° (XRD), C5–C11–F3 is 118.8° (DFT) and 118.2° (XRD). The torsional angles of C6–C8–C9–C7 is -180° (DFT) and -173.6° (XRD), is ° (DFT) and ° (XRD), C6–C12–C16–C19 is 0° (DFT) and 0.1° (XRD), CL2–C10–C14–C18 is 180° (DFT) and 178.8° (XRD), F3–C11–C15–C18 is 180° (DFT) and 179° (XRD). The bond length, bond angles and torsional angles obtained from DFT and XRD results are having good agreement

B. Vibrational Spectroscopy Study

The 2C6FBC compound has 78 normal modes of vibration and contains 28 atoms. Compound 2C6FBC functional groups are compared with experimentaly recorded FTIR spectra and theorotically obtained B3LYP/6-311++G(d, p) level. 2C6FBC compound bands along with their vibrational assignment are observed in infra red region (4000- 400 cm^{-1}). The spectra of compound are shown in Fig.3 their band assignments are shown in Table 2. Arromatic C─H stretching vibrational modes is 3100─3000 cm^{-1} regions. The strong unique absorption peak of carbonyl group region in between 1750-1620 cm^{-1}. Strong peak of C ═ O group vibration is observed at 1664.98 cm^{-1} (experimental), and 1636 (theoretical) and for the molecule of 2C6FBC. The theoretical and experimental C ═ C stretching found in the vicinity of 1600-1550 cm^{-1}. The theoretical and experimental C═C stretching found be 1600.58 cm^{-1} and 1596 cm^{-1} respectively. The 2C6FBC molecule has modes of vibrations lie in the range of 1550–600 cm^{-1} for experimental spectra and calculated spectra. The stretching vibration of C–B of 2C6FBC is obtained at 658.56 (expt) and 644 cm-1 (DFT). The stretching vibration of C–F of 2C6FBC is obtained at 1068.89 (expt) and 1084 cm-1 (DFT). The stretching vibration of C–Cl of a molecule is observed at 740.80cm^{-1} (expt) and 724 cm^{-1} (DFT). The C-H bending of aromatic ring is found in the range of 840 cm^{-1} – 710 cm^{-1}. The C-C bending of aromatic ring is found in the range of 1600 cm^{-1} – 1450 cm^{-1}. The details of the functional vibrational frequencies are assigned. In presumption, the results of FTIR vibrations calculated and theoretical functional groups frequencies are good agreement with each other. Hence, Vibrational spectroscopic study authenticates Functional groups and their molecular structure of 2C6FBC.

Table 2
Experimental and theoretical (DFT) FT-IR vibrational frequency bands assignments (wavenumbers in cm^{-1}) of 2C6FBC

EXPT	DFT	Assignment
3455.03	3268	ν (C-H Aromatic Ring)
2925.46	3204	ν (C-H Aromatic Ring)
2366.86		ν (C-H Aromatic Ring)
1934.11		ν (C-H Aromatic Ring)
1852.07		ν (C-H Aromatic Ring)
1664.98	1636	ν_{sym} (C=O)
1600.58	1596	ν(C=C), ν(C=C Aromatic ring)
1564.11	1516	ν C-C Aromatic Ring
1478.49	1484	ν C=C-C Aromatic ring
1456.13		δ (C-H Aromatic Ring)
1437.92	1420	δ (C-H Aromatic Ring)
1394.28		ν (Aromatic Ring)
1352.77	1356	δ(C-H, Ring) Sc
1323.33	1324, 1300	ν (C-H Aromatic Ring)
1274.85	1268	δ (C-H Aromatic Ring)
1248.07	1228	δ (C-H Aromatic Ring)
1203.07	1204	δ (C-H Aromatic Ring)
1172.77	1164	δ(C-H, C-C Ring)
1110.81	1132	δ(C-H, C-C Ring)

1068.89	1084	ν(C-F)
1026.10	1036	τ (C-H Aromatic)
1004.24	1020	γ(C-C, C-H Ring) Wa
979.40		ν(C-C-C)Sym, δ(C-C-C Ring)
924.49	932	ν(C-C-C)Sym, δ(C-C-C Ring)
856.06	860	γ(C-H Ring), τ(H-C-C-H Ring)
821.37	836	γ(Ring) Wa, τ(Ring)
782.44	796, 764	γ(C-H Ben Ring) w
740.80	724	ν (C-Cl)
705.67		δ(C-C-C Ben Ring) w
658.56	644	ν (C-Br)
638.38		γ (Aromatic Ring)
624.64		γ (Aromatic Ring)
509.44	516	γ(C-H Ben Ring) w
485.07	476	γ(C-H Ben Ring) w
459.85		γ(Ben Ring) Wa, ν(Ring)
429.69	436	γ(Ben Ring) Wa, ν (Ring)

ν-Stretching;δ-In plane bending; γ-Out of plane bending; τ-Torsional, w-weak, Wa-wagging, sym-Symmetric, Ben-Benzene

C. Proton NMR Analysis

The number of protons and molecular structure of tittle compound 2C6FBC are confirmed. The combined ^{1}H NMR spectra of experimental and theoretical are given away in Fig. 4 and their relevant chemical shifts (δ) are given in Table 3. 2C6FBC molecular structure has 9 protons. The theoretical ^{1}H NMR spectra shows that it every proton consists of separate peaks, where as the experimental spectra shows that it consists of compound peaks because of degeneracy.

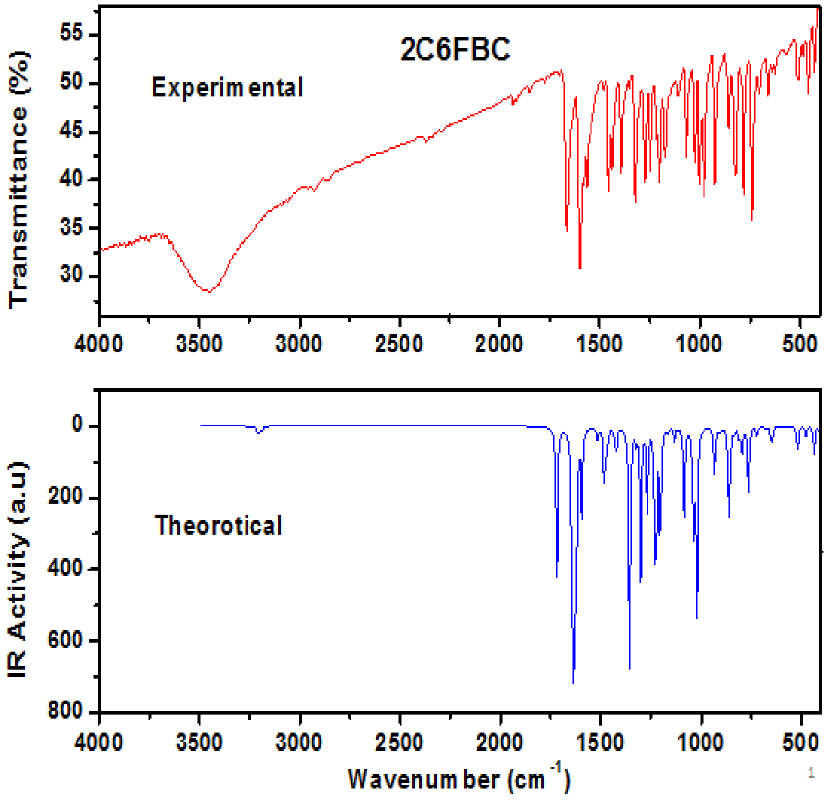

Fig. 3. Calculated and Experimental FTIR vibrational spectra of 2C6FBC

The 2C6FBC molecule will have chemical shifts δ=7 ppm to 8.5 ppm is observed. ^{1}H NMR spectral study results of experimental and theoretical values are in good correlation with their derivatives.

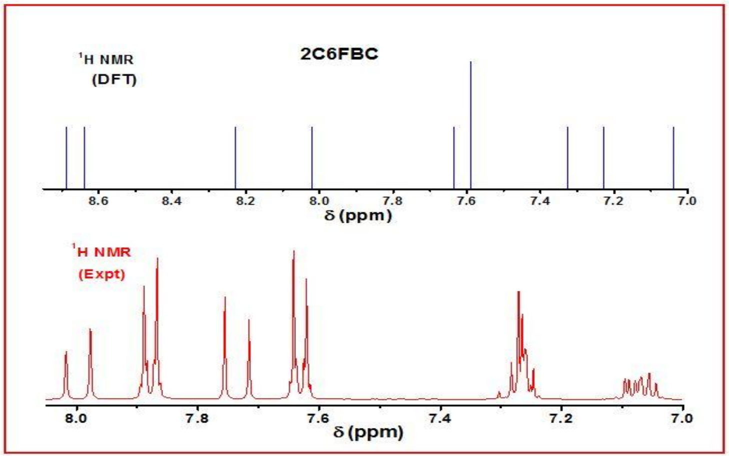

Fig. 4. The theoretical and experimental ^{1}H NMR spectra for 2C6FBC.

Table 3
The experimental and theoretical (DFT)^{1}H NMR chemical shifts (δ in ppm) of 2C6FBC.

(^{1}H) type	EXPERIMENTAL	DFT
25-H	7.044, 7.055, 7.069, 7.078, 7.089, 7.095	7.0396
28-H	7.246, 7.263	7.228
24-H	7.270, 7.283, 7.303	7.3264
27-H	7.621	7.5892
26-H	7.642	7.6346
22-H	7.716, 7.755	8.0203
21-H	7.867	8.2289
23-H	7.888	8.6374
20-H	7.977, 8.017	8.6894

D. Thermal Analysis

The Fig.5 shows Thermo gravimetric (TG) and differential thermal (DT) analysis. A weight of 11.414 mg compound 2C6FBC initially used for investigation. Thermo gravimetric (TG) curve sketch confirms that the weight loss is 1 % for a temperature 30-230 ^{0}C, due to humidity and unpredictable solvent. The major weight loss is 17.31 % at 250.81 ^{0}C because of decomposition of 2C6FBC. In TGA show that final residual mass is about 82.69 % heating up to 500 ^{0}C. There is an endothermic peak observed in DTA trace at 113.35 ^{0}C which is assigned to melting point of 2C6FBC molecule.

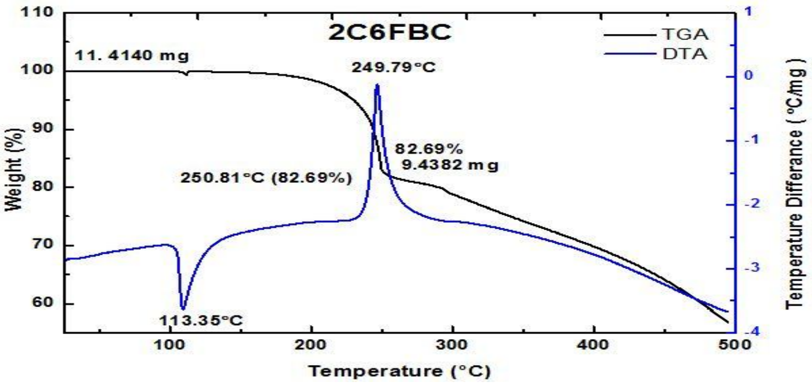

Fig. 5. TGA/DTA plots for 2C6FBC crystal

E. Ultra Violet-Visible- Absorption Spectrum

Ultra Violet-Visible-Infrared absorption spectrum was obtained for the sample 2C6FBC at the range 200nm-800nm. Two peaks in absorption spectrum corresponding to electronic transitions of π–π* and n–π* and their absorption peaks are 236 nm and 328 nm respectively. The absorption must be maximum and it will have the peak at 328 nm as shown in Fig.6. From absorption spectrum the energy gap of 2C6FBC was computed using Tauc's plot [16]. Plotting graph of $(\alpha h\nu)^{1/n}$ verses photon (hν) by taking n=0.5, intersects x-axis and the value of energy gap (E_g) was found to be 2.91 eV (indirect method) and 3 eV (direct method) as shown in Fig. 6.

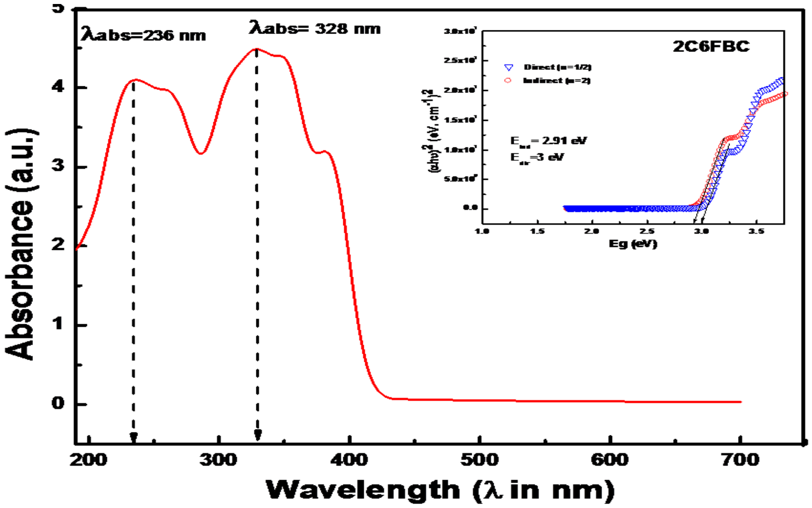

Fig. 6. The experimental absorption spectra of 2C6FBC

F. TD-DFT Linear Absorption Study

The electronic excitation, wavelength absorption and oscillator strength can be calculated for three different levels by TD-DFT method with B3LYP/6-311++G(d, p), CAM-B3LYP/6-311++G(d, p) and HF/6-311++G(d, p) basis in gas phase[17] as shown in Fig.7. The vertical absorption in UV-Vis spectrum corresponds to highest absorption peak. The theoretical absorption vales from TD-DFT Absorption spectrum at for basis set HF, CAM-B3LYP and B3LYP found to be 265.24 nm, 292.65 nm, and 331.60 nm. The absorption wavelength, excitation energy, oscillation strength and contribution of HOMO-LUMO orbital are revealed in Table 4. Excitation energy of 2C6FBC from TD-B3LYP predicted 3.74 eV.

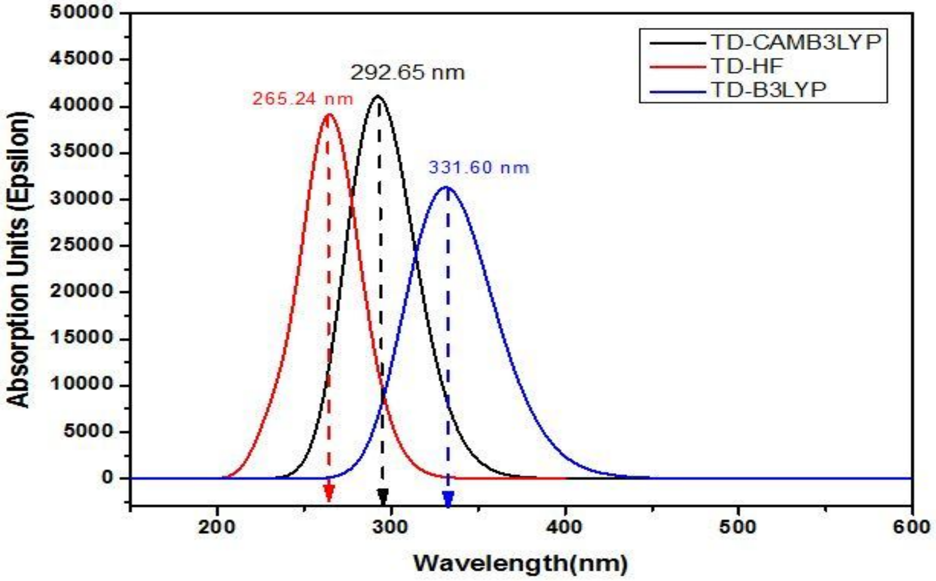

Fig. 7. Calculated absorption spectra of 2C6FBC

Table 4
The excitation energy ΔE, oscillator strengths f_0 and major contributions of HOMO-LUMO orbital's of 2C6FBC molecule

Method	Electronic transitions	λ_{Ex} (nm)	ΔE (eV)	f_0	Major contributions (in %)
Experimental	-	328	3.78	-	-
B3LYP	$S_0 \rightarrow S_1$	391.28	3.17	0.000	H-2→L (96)
	$S_0 \rightarrow S_2$	331.60	3.74	0.7665	H→L (88)
	$S_0 \rightarrow S_3$	314.02	3.95	0.0090	H-3→L (41)
CAM-B3LYP	$S_0 \rightarrow S_1$	352.91	3.51	0.000	H-3→L (87)
	$S_0 \rightarrow S_2$	292.65	4.24	1.0045	H→L (92)
	$S_0 \rightarrow S_3$	268.66	4.61	0.0243	H-2→L (71)
HF	$S_0 \rightarrow S_1$	271.29	4.57	0.000	H-5→L (60)
	$S_0 \rightarrow S_2$	265.24	4.67	0.9471	H→L (78)
	$S_0 \rightarrow S_3$	235.13	5.27	0.1718	H-1→L (44)

G. HOMO-LUMO Study

The molecular orbit (MO) consists of unoccupied molecular orbital (LUMO) and highest occupied molecular orbital (HOMO). HOMO-LUMO energy gap of compound 2C6FBC theoretically determined by B3LYP/6-311++G (d, p) is 4.14 eV are listed in Table 5. Molecular orbits are shown in Fig. 8. Transition of $\pi \rightarrow \pi^*$ orbital strongly suggest that it is system of donor and acceptor. The energy gap of HOMO-LUMO is good agreement calculated is 4.14 eV and excited energy gap 3.74 eV.

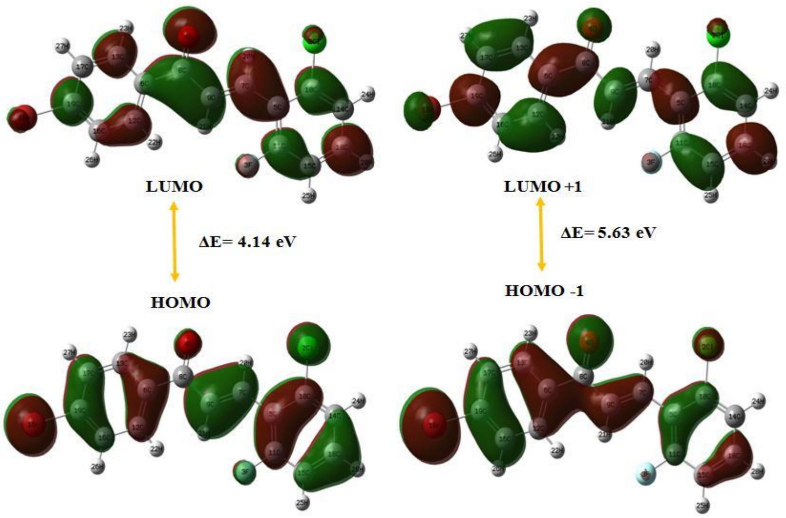

Fig. 8 HOMO-LUMO plots of 2C6FBC

Table 5
The frontier molecular orbital energies (in eV), and global chemical reactivity descriptors (in eV), chemical hardness (η), potential (μ), softness (S), electronegativity (χ), and electrophilic index (ω) of 2C6FBC molecule

E_H	E_L	E_{H-1}	E_{L+1}	ΔE_{H-L}	$\Delta E_{H-1-L+1}$	η	μ	S	χ	ω	I	A
-7.02	-2.88	-7.15	-1.51	-4.14	-5.63	2.07	-4.95	0.24	4.95	5.91	7.02	2.88

H. Global Chemical Reactivity Descriptors (GCRD)

This is the important utensils to identify the chemical stability, hardness, softness, potential, electronegativityand electrophilic index. Calculated GCRD are listed in the table 5. The value hardness must larger so that molecule will be more stable. Hardness (η) of 2C6FBC compound is found to be 2.07 eV, softness (S) is 0.24 eV, Potential (μ) is -4.95 eV, Electronegativity (χ) is 4.95 eV, Electrophilic index (ω) is 5.91 eV, Ionization energy (I) is 7.02 eV and Electron affinity (A) is 2.88 eV.

IV. CONCLUSIONS

The compound 2C6FBC are confirmed with spectroscopic experiments results of FTIR, proton NMR and UV-Visible. The density functional theory is used to obtain optimized geometry by keeping B3LYP/6-311G++(d,p) at the basis set. Theoretical results of bond length, bond angle and torsinal angle are obtained from DFT are compared with XRD results of the crystal. The results are good agreement with each other. Also confirmation of molecular structure is done with proton NMR. Experimental and theoretical (DFT) FTIR spectrum of functional group frequency results are good agreement with each other. This compound is a derivative of chalcone and will be helpful in the field medical field for the treatment of disease. Synthesized compound is a chalcone derivative will able to help in the field of nonlinear optics.

V. ACKNOWLEDGMENT

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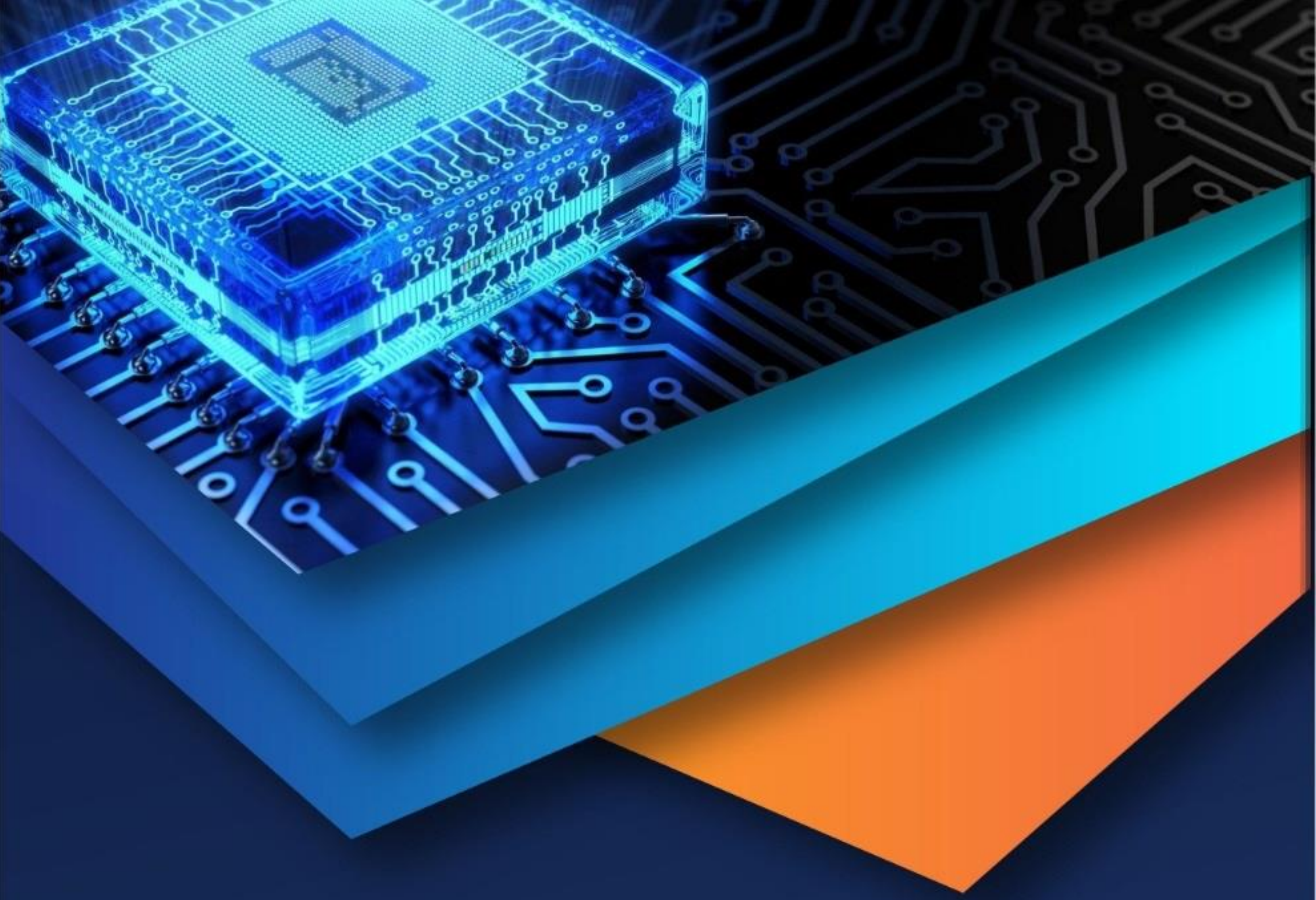

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TOGETHER WE REACH THE GOAL
IMPACT FACTOR:
7.429

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