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SYNTHESIS, CHARACTERIZATION AND OPTICAL PROPERTIES OF THIO-1,3,4-OXADIAZOL-2-YL DERIVATIVES

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ABSTRACT: A novel compound of thio-1,3,4-oxadiazol-2-yl derivatives is designed and synthesized by a reaction of (S)-2-amino-2-phenylacetic acid with ethanol-derived ethyl (S)-2-amino-2-phenylacetate. The ethyl (S)-2-amino-2-phenylacetate reacts with Boc anhydride and hydrazine to produce ethyl (S)-2-((tert-butoxycarbonyl)amino)-2-phenylacetate. The intermediate 5-alkyl amino-1,3,4-oxadiazole-2-thiols have been isolated as stable compounds. The chemical structure of synthesized compounds was established based on spectral data of 1HNMR, 13CNMR, and IR. The mass of the novel compounds was established with the help of the LCMS test. By the powder XRD, the crystalline nature of the samples was established. The photoluminescence spectrum demonstrates the optical property of the compound and displays the absorption range between 360 to 362 nm.

KEYWORDS: (S)-2-amino-2-phenylacetic acid, Crystallization, optical activity.

INTRODUCTION:

The peptides are the important constituents of living organisms possessing diverse biological functions. However, the poor metabolic stability in *vivo*, low oral bioavailability, and hydrolysis by proteases limited its applications. The decoration of the peptide backbone with various amide-surrogates such as urea, thiourea, carbamate, and heterocycles have been found to improve the biological potency of the naturally occurring peptides, thus making them suitable candidates for various applications.[i-iii] In particular, the insertion of heterocycles such as tetrazole, triazole, thiazole, and isoxazoline in the place of amide bond is of considerable interest in designing the peptidomimetics due to the added pharmacophoric values of those aromatic nuclei.[iv-v] The compounds possessing 1,3,4-oxadiazoles belong to a group of heterocycles that have been attracting attention for the last two decades due to their wide range of biological interactions and gained a special interest in drug discovery.[vi-vii] Many of them exhibit antibacterial, anticonvulsant, anticancer activities and are used to fight infections involving AIDS.[viii-x] This heterocycle can also serve as a surrogate of amide and esters.[xi]In particular, S-linked 1,3,4-oxadiazoles have been endowed with a wide spectrum of pharmacological activities such as antiviral.[xii] They are also applied in agriculture as herbicides, fungicides, and insecticides.[xiii] The 1,3,4-oxadiazoles exhibiting photochemical, photophysical, electrochemical property, also have thermal and electroluminescent

properties.[xiv-xvii] In the electronic field the 1,3,4-oxadiazoles are used to produce organic light-emitting diodes (OLED), optical brighteners, and laser diodes. It is also a promising material for the PLED application.[xviii-xix] The 1,3,4-oxadiazoles derivatives exhibit the crystal structure.[xx-xxiii] based on earlier studies, an attempt is made to prepare and evaluate thio-1,3,4-oxadiazole derivatives for optical properties and the results are discussed.

EXPERIMENTAL

Synthesis of ethyl(S)-2-amino-2-phenylacetate (2):

In a round bottom flask, 100 mL of ethanol was taken. About 10gm (66mmol) of phenyl glycine was added and stirred well. Then con. H2SO4 7 mL (0.2eq, 13 mmol) was added slowly. The temperature of the solution is maintained below 35°C. After complete addition, the reaction mixture was refluxed for 16 h. The mixture was monitored by TLC (mobile phase Ethyl acetate: hexane: 8:2). The reaction mixture was concentrated, washed with water, and dried with Na2SO4 to an obtained oily residue. This ester was directly used for the second stage without carrying for any further purification.

It was obtained as colourless liquid. The yield was 66%. (LCMS: 95.2% purity). B.pt.104-106°C, IR (KBr, cm^{-1}):v_{max}1740 (C=O), 1537-1481(C=C), 3342 (amide -NH_2), 1259-1043 (C-O, C-N), 982-726 (C-H). ^{1}HNMR (400 MHz, DMSO-d_6, ppm): δ 1.321-1.338 (t, 3H, *J*=6.8Hz, -CH_3), 4.894-4.914 (t, 1H,*J*=8 MHz, -CH), 3.964-4.142 (m, 2H, -CH_2), 7.233-7.314(m, 5H, Ar-CH), 8.563-8.578 (d, 2H,*J*=6Hz, -NH_2). ^{13}CNMR (100 MHz, DMSO-d_6, ppm): δ 15.16, 64.70, 128.32, 129.78, 128.63, 136.20, 176.40, 63.82. For $C_{10}H_{13}NO_2$, Calculated: C- 67.02%, H-7.31%, N-7.82%, O-17.85%. Found: C-67.14%, H-7.26%, N-7.64%, O-17.96%. LCMS [M+1] $^+$: m/z 180.6.

Synthesis of ethyl(S)-2-((tert-butoxy carbonyl)amino)-2-phenylacetate(3):

The appropriate phenylglycine ethyl ester 8g (44mmol) and 1.4 mL (10.6 mmol) of triethylamine was stirred for about 20 min. Then 3.28 g (0.3eq, 13mmol) of di-tert-butyl dicarbonate was added and left agitating at room temperature for 24 h. The white precipitate of triethylamine was filtered off and the solution was evaporated on the rotary evaporator. The white crude product was dried in air and crystallized from isopropanol.

It was obtained as white solid with 52% yield, (LCMS: 95.7% purity), m.pt.112-114°C, IR (KBr, cm^{-1}):v_{max}1682(C=O), 1526-1493 (C=C), 3346 (amide -NH), 1262-1036 (C-OC-N), 994-713 (C-H). ^{1}HNMR (400 MHz, DMSO-d_6, ppm): δ 1.408(s, 9H, -CH_3), 7.257-7.326(m, 5H, Ar-CH), 5.527-5.545(d, 1H,*J*=7.2MHz, -CH), 8.324-8.340 (d, 1H, *J*=6.4Hz, -NH), 4.142-4.379 (m, 2H, -CH_2), 1.328-1.346 (t, 3H, *J*=7.2Hz, -CH_3). ^{13}CNMR (100 MHz, DMSO-d_6, ppm): δ 28.56, 79.58, 170.36, 62.74, 129.45, 128.98, 127.38, 136.82, 62.82, 176.32, 2.82, 16.72. For $C_{15}H_{21}NO_4$, Calculated: C-64.62%, H-7.42%, N-5.22%, O-22.74%.Found: C-64.50%, H-7.58%, N-5.01%, O-22.91%. LCMS [M+1] $^+$: m/z 280.7.

Synthesis of tert-butyl(2-hydrazineyl-2-oxo-1-phenyl)ethyl)carbamate(4):

The compound N-protected phenylglycine ethyl ester (3) 6.4gm (24 mmol) was dissolved in 20 mL of ethanol and then 3.6 mL of 98% hydrazine hydrate (76mmol) was dropped in. It was stirred for 24 h and concentrated under reduced pressure. The oily residue was crystallized by trituration with 10 mL of hexane. The crude product was filtered off and recrystallized from a mixture of hexane–ethanol (2:1, v/v).

It was obtained as white solid in 67% of yield, (LCMS: 95.3% purity), m.pt.96-98°C, IR (KBr, cm^{-1}):v_{max}1623(C=O), 1593-1518 (C=C), 3340 (amide -NH), 1309-1024 (C-O, C-N),1000-675 (C-H). ^{1}HNMR (400 MHz, DMSO-d_6, ppm): δ 1.380 (s, 9H, -CH_3), 2.508-2.526 (d, 1H, *J*=7.2Hz, -CH), 5.123-5.145 (d, 1H,*J*=8.8MHz, -CH), 7.227-7.338 (m, 5H, Ar-CH), 4.261-4.276 (d, 3H, J=6Hz, -NH), 9.351-9.368(t, 1H,*J*=6.8Hz, -NH). ^{13}CNMR (100 MHz,

DMSO-d_6, ppm): δ 28.62, 78.85, 169.88, 56.77, 128.65, 127.48, 127.93, 139.41, 19.88. For $C_{13}H_{19}N_3O_3$Calculated: C-58.85%, H-7.22%, N-15.84%, O-18.09%. Found: C-58.57%, H-7.43%, N-15.68%, O-18.33%. LCMS $[M+1]^+$: m/z.266.30.

Synthesis oftert-butyl (S)-(5-mercapto-1,3,4-oxadiazol-2-yl)(phenyl)methylcarbamate (5)

To a stirred solution of tert-butyl(2-hydrazineyl-2-oxo-1-phenyl)ethyl)carbamate (4) (5g, 18.8 mmol), in ethanol (30 mL), KOH 2.1 g (37.6 mmol) were added and stirred for 30min. Then $CS_2$1.75g (22.56 mmol) was added and stirred for 1h at room temperature. After completion of the reaction, concentrated to the residue, acidify with 1.5N HCl, solid was thrown out, filtered, and dried.

It was obtained as white solid with 67% of yield (LCMS: 95.3% purity), m.pt.104-106°C, IR (KBr, cm^{-1}):v_{max}1681(C=O), 1529-1518 (C=C), 3350 (amide -NH), 1016-1245 (C-O C-N), 991-704 (C-H), 637 (C-S). ^{1}HNMR (400 MHz, DMSO-d_6, ppm): δ 1.410 (s, 9H, -CH_3), 8.198-8.216(d, 1H,*J*=7.2Hz -NH), 6.156-6.175(d, 1H,*J*=7.6Hz -CH), 7.298-7.434 (m, 5H, ArH), 12.908(s, 1H, -SH).^{13}CNMR (100 MHz, DMSO-d_6, ppm): δ 28.46, 79.23, 155.82, 59.23, 126.68, 128.26, 127.03, 141.96, 163.32. For $C_{14}H_{17}N_3O_3S$, Calculated: C-54.70 %, H-5.58%, N-13.67%, O-15.62%, S-10.43%.Found: C-54.64 %, H-5.46%, N-13.75%, O-15.67%, S-10.48%. LCMS $[M+1]^+$: m/z 308.8.

Preparation of 1,3,4-oxadiazol derivatives (6a-e)

The one equivalent (100mg) weighed compound-**(5)** was dissolved in DMF (2mL), K_2CO_3 (2eq) was added and the reaction mixture was stirred for 15 min. Then alkyl/aryl halide (1eq) solution was added dropwise and stirred for 16 h at room temperature. After the completion of the reaction (monitor by TLC), ice water was added and extracted withethyl acetate. The organic layer was filtered to remove potassium carbonate. The filtrate was concentrated and diluted with Ethyl acetate (2x 2mL) washed with water and brine solution, dried, and concentrated. The crude was purified by column chromatography (gradient elution of 30-40% of ethyl acetate in hexane) to get desired product **(6a-e).**All the reaction schemes were shown in Fig.1. The yield and melting point of these samples are presented in Table.1

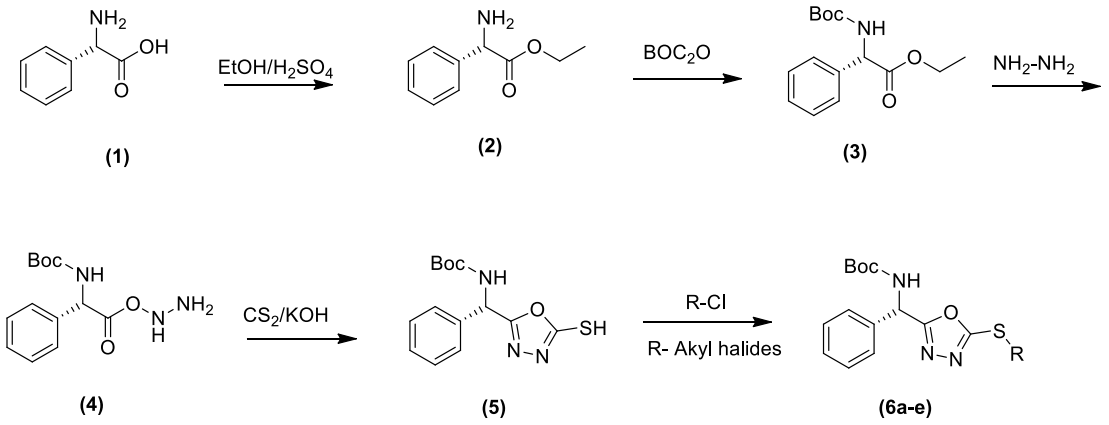
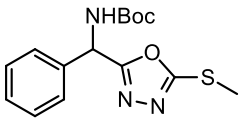
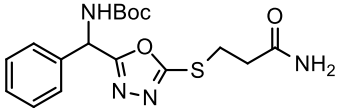
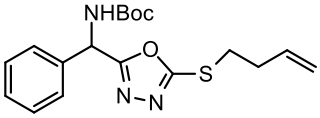
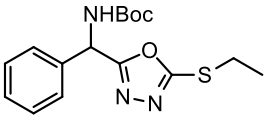
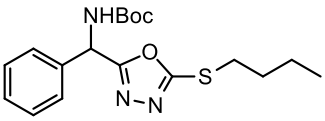


Fig.1. The scheme of reaction

Table.1 List of Alkyl halides used in the reaction

Compound	Alkyl halides	Products	Yield %	m.pt. °C
6a	Methyl iodide		93	110 - 112
6b	3-bromo propyle amine		97	127 - 129
6c	4-bromo butene		94	55 - 56
6d	Ethyl iodide		95	90 - 92
6e	Iodo butane		96	65 - 67

Synthesis of tert-butyl((5-(methylthio)-1,3,4-oxadiazol-2-yl)(phenyl)methyl) carbamate (6a).

The methyl iodide which is an alkyl halide was used to get the title compound. The reaction results as a white crystalline solid of 93% yield. (LCMS: 95.1% purity), m.pt.110-112°C, IR (KBr, cm^{-1}):v_{max}1686 (C=O), 1570-1481 (C=C), 3365 (amide -NH), 1251-1046 (C-O, C-N), 631(C-S) 972-705 (C-H).^{1}HNMR (400 MHz, DMSO-d_6, ppm): δ 1.400 (s, 9H, -CH_3), 8.258-8.272(d, 1H,*J*=5.6Hz -NH), 6.056-6.077(d, 1H,*J*=8.4Hz -CH), 7.349-7.434 (m, 5H, ArH), 2.508(s, 3H, -CH_3).^{13}CNMR (100 MHz, DMSO-d_6, ppm): δ 14.72, 28.56,51.00,79.48, 128.00, 128.74, 129.06, 137.42, 155.42,164.38.For $C_{15}H_{19}N_3O_3S$. Calculated:C-56.06%, H-5.96%, N-13.07%, O-14.93%, S-9.98%. Found: C-56.12%, H-5.90%, N-13.05%, O-14.97%, S-9.96%. LCMS $[M+1]^+$: m/z.322.8.

Synthesis of tert-butyl((5-((3-amino-3-oxopropyl)thio)-1,3,4-oxadiazol-2-yl)(phenyl)methyl) carbamate(6b).

The 3-chloro propanamide was added that reacts to gives the compound 6(b). It was obtained as white solid with a 97% yield. (LCMS: 95.3% purity), m.pt.127-128°C, IR (KBr, cm^{-1}):v_{max}1683 (C=O), 1570-1475 (C=C), 3365 (amide -NH), 1295-1016 (C-O, C-N), 621(C-S) 982-698 (C-H). ^{1}HNMR (400 MHz, DMSO-d_6, ppm): δ 1.380(s, 9H, -CH_3), 6.945-6.958(d, 1H, *J*=5.2Hz, -NH), 6.063-6.078(d, 1H,*J*=6Hz -CH), 7.348-7.416 (m, 5H, ArH), 5.755 (s, 2H, -NH_2), 3.316-3.337 (t, 2H,*J*=8.4Hz, -CH_2), 2.508-2.530(t, 2H,*J*=8.8Hz, -CH_2).^{13}CNMR (100

MHz, DMSO-d_6, ppm): δ 28.32, 28.56, 34.76, 50.99,79.56, 127.99, 128.74, 129.06, 137.32, 155.38, 164.72,172.17. For $C_{17}H_{22}N_4O_4S$, Calculated: C-53.95%, H-5.86%, N-14.80, O-16.92%, S-8.47%. Found: C-53.91%, H-5.80%, N-14.84%, O-16.96%, S-8.49%. LCMS $[M+1]^+$: m/z.379.8.

Synthesis of tert-butyl((5-(but-3-en-1-ylthio)-1,3,4-oxadiazol-2-yl)(phenyl)methyl) carbamate(6c).

The compound 6(c) was obtained by the addition of 4-bromo-1-butene. It was obtained as a white solid with a 94% yield, (LCMS: 95.6% purity), m.pt.55-56°C, IR (KBr, cm^{-1}):v_{max}1688 (C=O), 1577-1475 (C=C), 3380 (amide -NH), 1287-1015 (C-O, C-N), 630(C-S) 973-697 (C-H).^{1}HNMR (400 MHz, DMSO-d_6, ppm): δ 1.402 (s, 9H, -CH_3), 8.250-8.268 (d, 1H,*J*=7.2Hz, -NH), 6.070-6.088 (d, 1H, *J*=7.2Hz, -CH), 7.349-7.442 (m, 5H, ArH), 3.270-3.294(t, 2H,*J*=9.6Hz,-CH_2), 2.460-2.508(m, 2H, -CH_2), 5.759-5.825(m, 1H, -CH), 5.063-5.084(d, 2H,*J*=8.4Hz,-CH_2).^{13}CNMR (100 MHz, DMSO-d_6, ppm): δ 2.82, 28.56,31.68,33.44, 51.00,79.49, 117.58, 128.73, 128.00,129.04, 136.08, 137.31,155.38, 164.46.For $C_{18}H_{23}N_3O_3S$, Calculated: C-59.49%, H-6.58%, N-11.79%, O-13.43%, S-8.71%. Found: C-59.42%, H-6.63%, N-11.72%, O-13.45%, S-8.78%. LCMS $[M+1]^+$: m/z.362.9.

Synthesis of tert-butyl((5-(ethylthio)-1,3,4-oxadiazol-2-yl)(phenyl)methyl)carbamate(6d).

The addition of ethyl iodide yields the compound 6(d), white crystalline solid, with a 95% yield. (LCMS: 93% purity), m.pt.90-92°C, IR (KBr, cm^{-1}):v_{max}1683 (C=O), 1574-1475 (C=C), 3375 (amide -NH), 1289-1016 (C-O, C-N), 629(C-S) 966-697 (C-H).^{1}HNMR (400 MHz, DMSO-d_6, ppm): δ 1.400(s, 9H, -CH_3), 8.246-8.262(d, 1H,*J*=6.4HZ, -NH), 6.065-6.085(d, 1H, *J*=8Hz, -CH), 7.332-7.439(m, 5H, ArH), 3.315-3.327 (m, 2H, -CH_2), 1.340-1.362(t, 3H,*J*=8.8Hz -CH_3).^{13}CNMR (100 MHz, DMSO-d_6, ppm): δ 15.24, 27.06,28.56, 51.01, 79.49, 128.01, 128.73, 129.05, 137.32, 155.37, 164.49.For $C_{16}H_{21}N_3O_3S$, Calculated: 57.29%, H-6.32%, N-12.53%, O-14.31%, S-9.56%. Found: 57.29%, H-6.38%, N-12.46%, O-14.26%, S-9.58%. LCMS $[M+1]^+$: m/z.336.8.

Synthesis of tert-butyl((5-(butylthio)-1,3,4-oxadiazol-2-yl)(phenyl)methyl)carbamate(6e).

The reaction of iodobutane (alkyl halide) and the compound-5 results the compound 6(e) in the form of a white solid. Its yield percentage is 96%. (LCMS: 95.4% purity), m.pt.65-67°C, IR (KBr, cm^{-1}):v_{max}1685 (C=O), 1577-1475 (C=C), 3361 (amide -NH), 1286-1020 (C-O, C-N), 633(C-S) 969-702 (C-H).^{1}HNMR (400 MHz, DMSO-d_6, ppm): δ 1.403(s, 9H, -CH_3), 8.252-8.259 (d, 1H, *J*=2.8Hz, -NH), 6.058-6.063 (d, 1H, *J*=2Hz, -CH), 7.350-7.439 (m, 5H, ArH), 3.317-3.336 (t,2H, J=7.6Hz, -CH_2), 1.654-1.692 (m, 2H, -CH_2), 1.350-1.403 (m, 2H, -CH_2), 0.864-0.891(t, 3H, J=10.8Hz, -CH_3).^{13}CNMR (100 MHz, DMSO-d_6, ppm): δ 13.76, 21.45,28.55, 31.46, 32.15,51.00, 79.47, 128.00,128.72, 129.03, 137.31, 155.38, 164.60.For $C_{18}H_{25}N_3O_3S$, Calculated: C-59.48%, H-6.93%, N-11.57%, O-13.20%, S-8.82%. Found: C-59.54%, H-6.87%, N-11.52%, O-13.21%, S-8.86%. LCMS $[M+1]^+$: m/z 365.0.

RESULT AND DISCUSSION

The functional groups present in all the compounds were identified by FTIR spectra of these compounds. The compounds 6g and 6h show an additional peak due to C-F functional group in the range 1007-1055 cm^{-1}. The result of FTIR confirms the formation of the synthesized compounds. In^{1}HNMR, all the compounds display the almost same chemical shift values. When comparing the ^{1}HNMR spectrum of the compounds 6c and 6e, it is observed that the triplet at δ (0.864-0.891 ppm) is changed as a doublet at δ (5.063-5.084 ppm). It is due to the double-bound present at C-3 and C-4 in substituted butene.The same result was also obtained in ^{13}CNMR the chemical shift value of the compounds 6e and 6c at C-3 changes from 21.45-136.08 ppm and C-4 changes from 13.76-117.58 ppm. The structure of these compounds was

confirmed from the ^{1}HNMR and ^{13}CNMR. The melting point of the samples was studied and the results were given in Table.1. It shows that almost all the compounds except 6(c) and 6(e) have high melting points. The compounds 6(c) and 6(e) clearly explain the butane has high melting point than butene.The results of LCMS analysis establish the formation of the products. The percentage of the elements present in the products was obtained from elemental analysis. These values agree with theoretically calculated values. Hence the formation of the products is also confirmed from this analysis.

POWDER XRD STUDIES

The powder XRD pattern is shown in fig.2. From the graph, it is observed that the peaks are sharp and intense. This shows that the sample is pure and crystalline in nature.

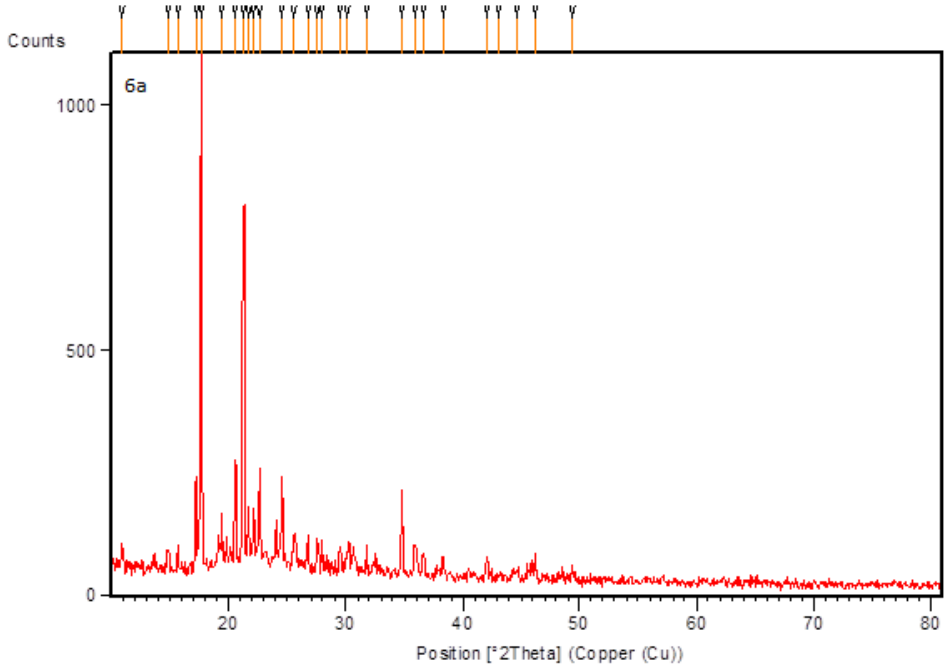


Fig. 2.Powder X-ray diffraction pattern oftert-butyl((5-(methylthio)-1,3,4-oxadiazol-2-yl)(phenyl)methyl) carbamate (6a)

The crystalline size is calculated from the Debye-scherrer formula

$$D = \frac{K\lambda}{\beta \text{Cose}\theta} \quad \text{where } k = 0.9$$

$$D = \frac{0.9\lambda}{\beta \text{Cose}\theta}$$

$\lambda \rightarrow$ wavelength 1.546 A°

$\beta \rightarrow$ Full width half (0.1476deg = 0.002576 rad)

$\theta \rightarrow$ Angle of diffraction(17.6814/2 = 8.8407deg = 0.1543 rad)

D = 0.9 *X* 0.154/0.002576 *X* Cos(0.1543)

D = 54.4702 nm.

The powder XRD pattern is shown in fig.3. From the graph, it is observed that the peaks are sharp and intense. This shows that the sample is pure and crystalline in nature.

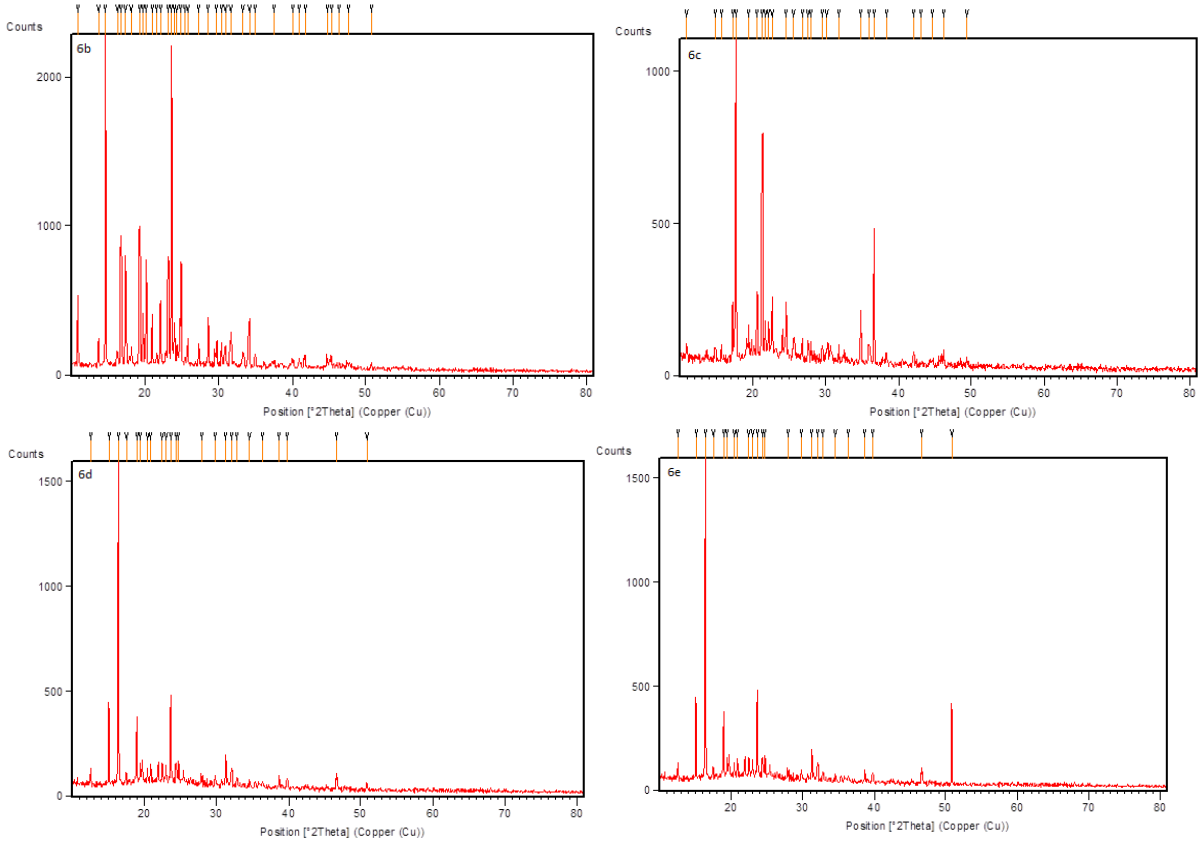


Fig. 3. Powder X-ray diffraction pattern ofcompound 6b, 6c, 6d and 6e

The crystalline size of the samples are calculated and presented in Table.2

Table.2 Crystalline size of the compounds

S.No	Compound name	Size (nm)
1	6a	54.4702
2	6b	54.3391
3	6c	54.3471
4	6d	54.3748
5	6e	54.3463

From the table, it is observed that the crystalline size is decreasing due to the incorporation of additional compounds.

PHOTOLUMINESCENCE

Photoluminescence (PL) examines material for wide applications in the field of medical, biochemical, and chemical research. In PL spectroscopy, generally, a beam of light excites the electrons in the molecule of given materials and causes them to emit light in a longer wavelength than the observed radiation. The figures Fig.7-11 show the PL spectra of the samples. These spectra give the absorption wavelength at around 360 nm which means

the emission of blue radiation. The absorption peak is due to the band-to-band electronic transition in a material. The result predicts the use of the materials as a color filter.

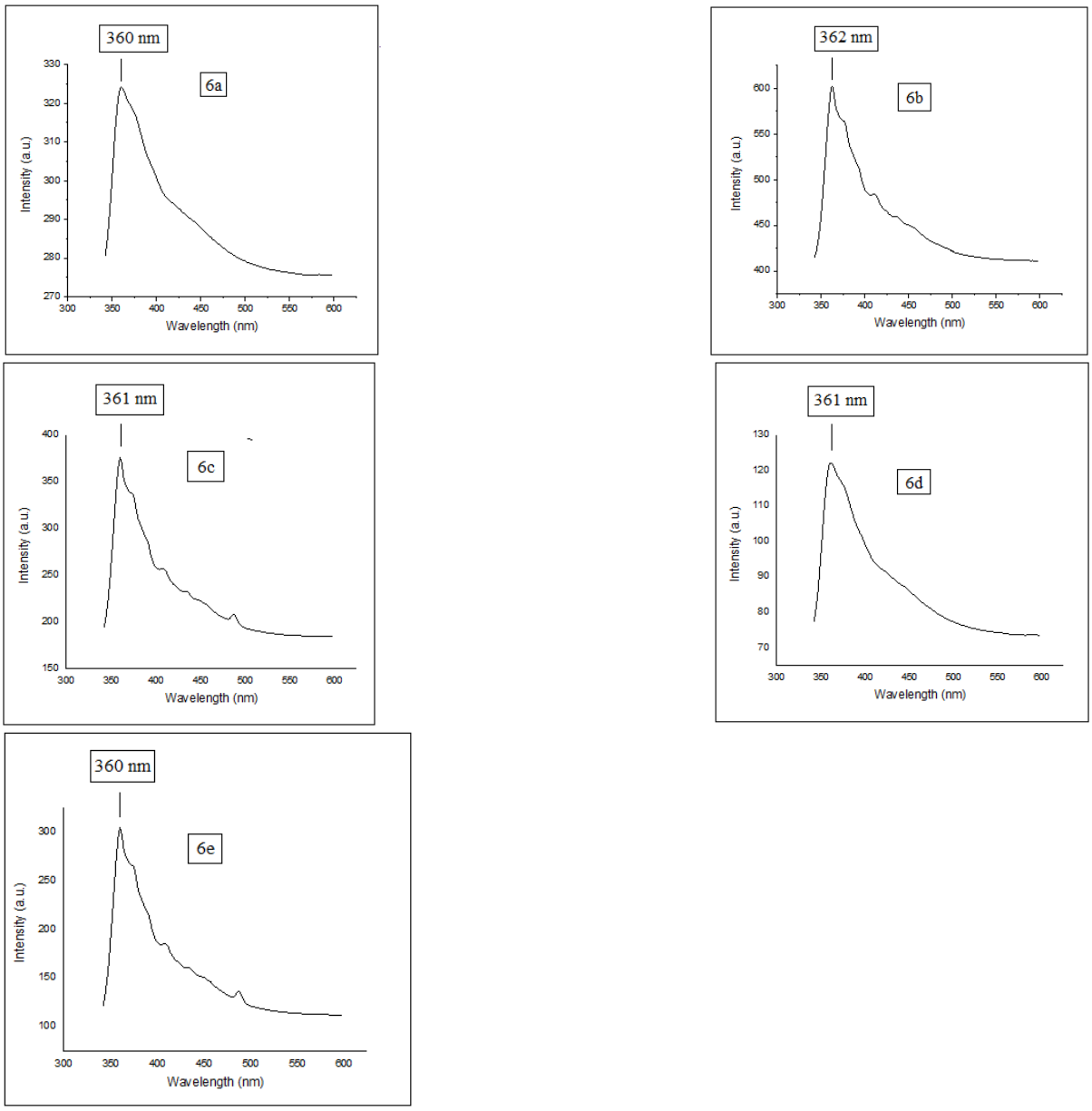


Fig.4. Photoluminescence spectrum ofcompound 6a, 6b, 6c, 6d and 6e

CONCLUSION:

The synthesis of derivatives of thiol-1,2,3-oxadiazole (6a-e) was carried out. The functional groups present in the samples were studied from the FTIR spectra and thus it confirms the synthesis of the compounds. The proton and carbon positions of these were obtained through 1HNMR and 13CNMR spectra respectively. The LCMS study indicates the good yield of all the compounds. The melting point of the samples was studied and shows that almost all the compounds except 6(c) and 6(e) have high melting points. The compounds 6(c) and 6(e) clearly explain the butane has high melting point than butene. The crystalline nature of samples 6 (a to e) was confirmed by the powder X-ray diffraction studies. These samples good optical nature as studied from PL study.

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