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RESEARCH ARTICLE

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Effect of organic solvents on solvatochromic, fluorescence, and electrochemical properties of synthesized thiazolylcoumarin derivatives

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Abstract

In this present investigation, thiazolylcoumarin derivatives (**5a**–**5k**) were synthesized from thiosemicarbazide, ethyl acetoacetate, and naphthaldehyde through a multistep route. The formation of thiazolylcoumarin derivatives with bioactive scaffolds was confirmed through nuclear magnetic resonance spectroscopy. A solvatochromic study of synthesized thiazolylcoumarin derivatives was carried out using ultraviolet–visible methods for dimethylformamide (DMF), ethyl acetate, and ethanol solvents. The redox behaviour of as-synthesized thiazolylcoumarin derivatives (**5a**–**5k**) was examined in dimethyl sulphoxide by conducting an electrochemical study. Fluorescence properties of thiazolylcoumarin derivatives were studied in DMF, ethanol, and ethyl acetate to visualize the solvent effect on the emitting ability of thiazolylcoumarin derivatives.

KEYWORDS

fluorescence, solvatochromic study, solvent effect, thiazolylcoumarin derivatives, thiosemicarbazide

*Ali Bahadur and Shahid Iqbal contributed equally to this study.

1 | INTRODUCTION

Biologically active heterocyclic compounds, especially coumarin derivatives, have potential application in the medical field.[1–5] Coumarins are naturally occurring heterocycles as well as secondary metabolites.[3,6–8] Many other publications are available describing coumarin and its derivatives that show that these compounds are highly bioactive in nature.[9–13] Thiazoles are widely used in diverse chemical reactions as parent nuclei and as intermediates, as well as substituents. Thiazoles heterocycles are also used to synthesize various thiazolyl coumarin derivatives.[14–18]

Regarding thiazole-based coumarinyl scaffold bioactivity (after surveying other publications), coumarin derivatives such as thiazole and azetidine exhibit antibacterial, insecticidal, antifungal, antiproliferative, antioxidant, antitubercular, anti-inflammatory, and anticancer properties.[19–24] Therefore, these derivatives are very important in the fields of agriculture, organic chemistry, and medicine for the synthesis of fungicides, dyes, and drugs, repectively.[25–27] Coumarins have a double bond in *trans* conformation that features in the high fluorescence quantum yield, strong fluorescence, and photostability in most of the coumarin derivatives.[28–33] Coumarin derivatives have very good fluorescence properties and have potential application on a commercial scale as photoluminescent materials.[34–36]

Enhanced coumarin derivatives screening biological activity have made it necessary to study these chemical classes, as these scaffolds have substantial therapeutic capabilities.[35,37–39] From a recent review of other publications, triazoles and chromenes, heterocyclic derivatives have been often applied in biotechnology, medicine, and industry. Therefore, in this study, novel coumarin derivatives were synthesized.[40–42]

In this present investigation, thiazolylcoumarin derivatives were synthesized through a multistep route. The effect of different organic solvents [ethyl acetate, ethanol, and dimethylformamide (DMF)] on the solvatochromic, electrochemical, and fluorescence properties of synthesized thiazolylcoumarin derivatives was studied. All synthesized thiazolylcoumarin derivatives were used effectively as a tool to visualize electrochemical behaviour and fluorescence phenomena. The solvatochromic, electrochemical, and fluorescence results of synthesized thiazolylcoumarin derivatives were highly encouraging.

2 | EXPERIMENTAL

2.1 | Materials

Thiosemicarbazide (CH_5N_3S, 99%, Sigma), ethyl acetoacetate ($C_6H_{10}O_3$, 99%, Sigma), naphthaldehyde ($C_{10}H_7CHO$, 98%, Sigma), substituted aromatic aldehydes (**3a**–**3k**), sodium acetate ($C_2H_3NaO_2$, 98%, Sigma), bromine (Br_2, 98.5%, Sigma), piperidine ($C_5H_{11}N$, 99%, Sigma), chloroform ($CHCl_3$, 99%, Sigma), ethanol (C_2H_5OH, 99.8%, Sigma), DMF (C_3H_7N, 99.8%, Sigma), ethyl acetate ($C_4H_8O_2$, 99.8%, Sigma), dimethyl sulfoxide (DMSO, 98%, Sigma), and acetic acid (CH_3COOH, 98%, Sigma) were used to synthesize thiazolylcoumarin derivatives and used without further purification.

2.2 | Preparation of starting compounds

2.2.1 | Preparation of 2-acetyl-3H-benzo[f]chromen-3-one (1)

Briefly, for the synthesis of 2-acetyl-3H-benzo[f]chromen-3-one (**1**), ethylacetoacetate (100 mmol, 12.64 ml) and 2-hydroxy-1-1 naphthaldehyde (100 mmol) were mixed in piperidine (1 g, 1.12 ml) at 0°C and stirred for 0.5 h. The resulting 2-acetyl-3H-benzo[f]chromen-3-one (**1**) product was washed several times with ethanol and crystallized in H_2O to obtain the pure product (**1**) (Figure 1).

2.2.2 | Preparation of 2-(2-bromoacetyl)-3H-benzo[f]chromen-3-one (2)

Product (**1**) of the previous step was dissolved in $CHCl_3$ and Br_2 solution (1.72 g, 0.01 mol) was added dropwise. The resulting solution was refluxed for 3 h and cooled to 25°C. The final product was crystallization in $CHCl_3$:ethanol (1:2) to get pure 2-(2-bromoacetyl)-3H-benzo[f]chromen-3-one (**2**) (Figure 1).

2.2.3 | Synthesis of thiosemicarbazide derivatives (4a–4k)

Thiosemicarbazide (0.138 g, 1.0 mM) was dissolved in 25 ml absolute ethanol and substituted aldehydes (**3a**–**3k**) (1.0 mM) along with 1–2 drops of acetic acid were added to the thiosemicarbazide solution. The mixture was refluxed until the solid crystals of the product were formed. Solid products were recrystallized in ethanol to produce thiosemicarbazide derivatives (**4a**–**4k**) (Figure 1).

2.2.4 | General procedure of thiazolylcoumarin derivatives (5a–5k) synthesis

Thiazolylcoumarin derivatives (**5a**–**5k**) were synthesized by heating a mixture of 2-(2-bromoacetyl)-3H-benzo[f]chromen-3-one (**2**) (5 mol), and **4**(**a**–**k**) (6.2 mol) in 25 ml of sodium acetate/ethanol. After 5 h, the solution was cooled to get solid products (Figure 2). As-synthesized thiazolylcoumarin derivatives (**5a**–**5k**) were filtered and crystallized in ethanol (Table 1). Supplementary data contains complete characterization data.[43]

2.3 | Characterizations

Structures of the thiazolylcoumarin derivatives were determined using a Bruker DPX 300 NMR spectrometer (300 MHz) with ^{1}H-NMR and ^{13}C-NMR spectra (Figures S1 and S2). Ultraviolet (UV) light studies were performed using the UV-Genesys spectrophotometer. FTIR spectra were obtained using a Nicolet IR 100 FTIR spectrometer

FIGURE 1 Synthesis of thiosemicarbazide derivatives (**4a–4k**)

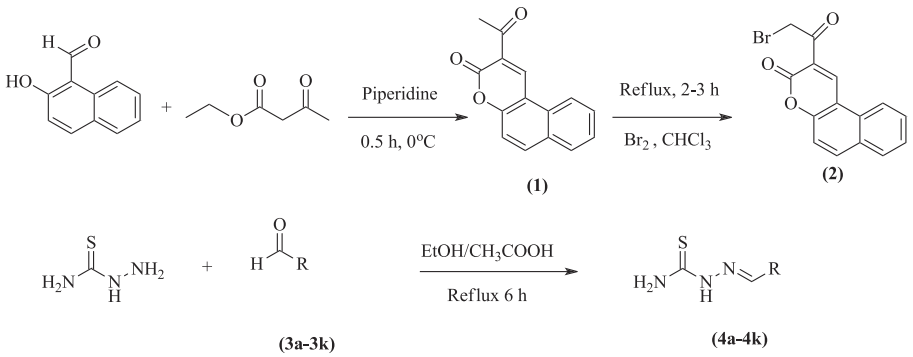
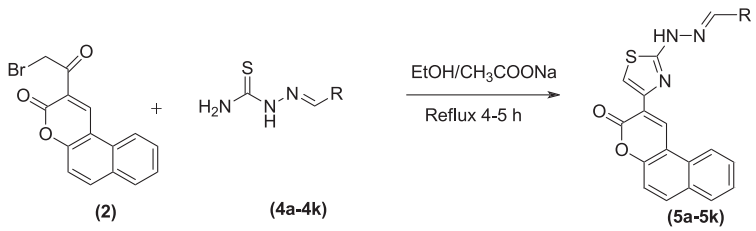


FIGURE 2 Synthesis of thiazolylcoumarin derivatives (**5a–5k**)



(Figure S3). An IME6X electrochemical workstation voltage −1.5 V to 1.5 V was used for cyclic voltammetry. A Fluoromax-4 spectrofluorometer (Horiba Scientific Japan) was used for emission spectra. An FL-980 Steady-Transient state fluorescence spectrometer (Edinburgh Instruments, UK) for time-resolved photoluminescence (TRPL) spectroscopy.

3 | RESULTS AND DISCUSSION

3.1 | Preparation of thiazolylcoumarins derivatives (5a–5k)

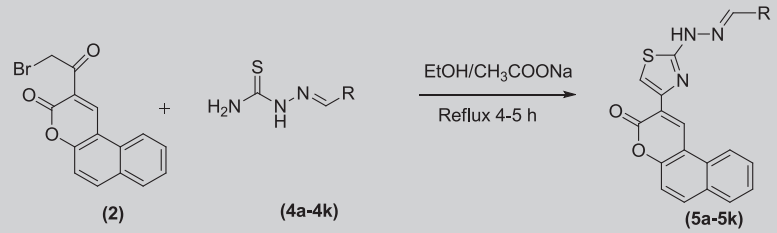
Thiazolylcoumarins derivatives (**5a–5k**) were prepared by heating a mixture of **4**(**a–k**) (6.2 mmol) and 2-(2-bromoacetyl)-3H-benzo[f] chromen-3-one (**2**) (5 mmol) in 25 ml of ethanol/sodium acetate. After heating for 5 h, the solution was cooled to obtain the final products, thiazolylcoumarin derivatives (**5a–5k**), which were then cleaned by filtering and crystallization in ethanol.[43]

3.2 | Structural confirmation of thiazolylcoumarin derivatives (5a–5k)

UV spectroscopy, FTIR spectroscopy, and NMR spectroscopy were used to confirm the structure of the thiazolylcoumarin derivatives (**5a–5k**). Figure 3 illustrates the UV–vis absorption spectra of coumarinyl derivatives (**5a–5k**); the corresponding spectral data are shown in Table 2. UV–vis spectra of thiazolylcoumarin derivatives (**5a–5k**) showed the highest absorption in the 340–392 nm range (Figure 3a–c). This absorption range may be due to the π–π^* transition of electrons in the naphthalene and benzene rings present in all the synthesized derivatives. The λ_{max} for **5c** and **5h** was 392 nm, and might be due to the π–π^* transition of bromobenzene and pyridine rings; this value was the largest for all the prepared derivatives. Many electrons are need to generate a bathochromic shift. Sulfur atoms present in the nucleus have less electronegativity relative to oxygen and, consequently, have a greater ability to provide electrons, resulting in a larger λ_{max} value. The smallest λ_{max} value for **5h** (340 nm) was due to the existence of the methylene groups between the aryl group and coumarin motif that produced a hypsochromic shift. The higher λ_{max} (362–386 nm) for **5e**, **5c-g**, and **5i,j** was due to π–π^* transitions of π electrons, and the n–π^* transition of lone pairs of electrons. There was no large difference in the λ_{max} value for any given compound with change in polarity of the solvent because the λ_{max} values for thiazolylcoumarin derivatives do not depend on the nature of the solvents. Our results suggested that the π–π^* transitions were due to B-type bands and independent of solvent, and therefore no solvatochromism phenomenon was detected.

Absorption bands due to C=N of the imine, and C=O, Ar–H, and N–H of lactone were observed from FTIR spectra of the thiazolylcoumarin derivatives (**5a–5k**). Absorption band values at 3360–3390, 2980–3170, 1730–1755, 1630–1660, 1520–1540, 1415–1450, 1280–1295, 1115–1160, 845–870, and 780–830 cm^{-1} were due to the vibration frequencies of C=C, C=S, and C–O, C=C, C=S, and C–O. For thiazolylcoumarin **5a**, the band values at 3375 cm^{-1} and 3170 cm^{-1} were due to N–H and =C–H (str.).[44] A peak at 1729 cm^{-1} revealed the presence of an ester linkage in the carbonyl of the cyclic esters. Conversely, all thiazolylcoumarin derivatives had similar stretching vibrations peaks for N–H, C=C–H, and C=O groups. Peaks at 1155 cm^{-1}, 1535, and 860 cm^{-1} were the result of C–O stretching, C=C stretching, and C=C stretching vibrations, respectively. These peaks were mainly due to the presence of

TABLE 1 As-synthesized thiazolylcoumarin derivatives (**5a**–**5k**)



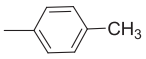
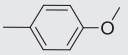
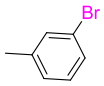
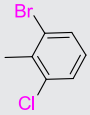
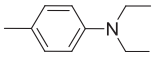
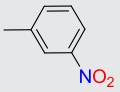
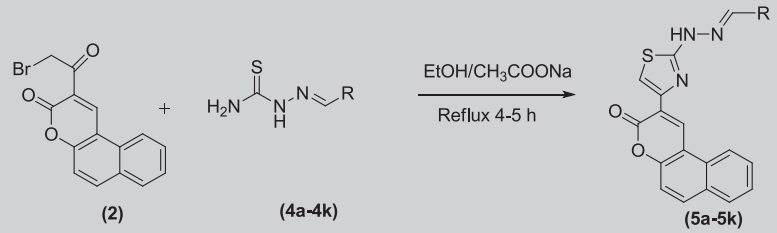
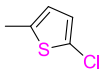
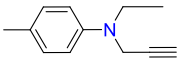
Thiazolylcoumarin derivatives	-R	Yield (%)
5a		85%
5b		83%
5c		78%
5d		78%
5e		75%
5f		73%
5g		77%
5h		76%

TABLE 1 (Continued)



Thiazolylcoumarin derivatives	-R	Yield (%)
5i		86%
5j		66%
5k		68%

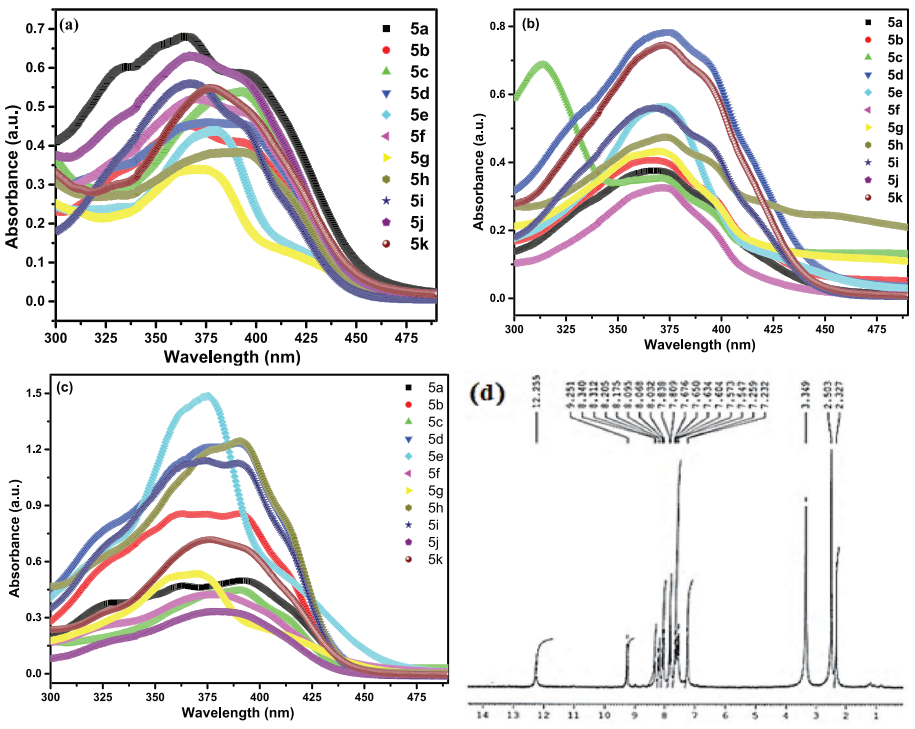


FIGURE 3 UV/vis spectra of thiazolylcoumarin (**5a**–**5k**) derivatives in (a) ethanol, (b) DMF, and (c) ethyl acetate. (d) ^{1}H-NMR of thiazolylcoumarin derivative **5a**

aromatic rings in **5a**–**5k**, whereas other peaks were the result of other functional groups and agreed with the reported values for the structures.

A singlet at 2.3 ppm for 3H on the methyl group substituted in the benzene ring was found using ^{1}H-NMR spectroscopy for thiazolylcoumarin **5a**. In addition, the N–H substituted heterocyclic

TABLE 2 λ_{max} (nm) of thiazolylcoumarin derivatives (**5a**–**5k**)

Sr. no.	Ethanol (λ_{max})	DMF (λ_{max})	Ethyl acetate (λ_{max})
5a	336	353	332
5b	351	366	363
5c	365	381	392
5d	381	344	388
5e	381	371	375
5f	372	360	379
5g	369	375	370
5h	372	376	392
5i	366	375	389
5j	373	380	375
5k	375	378	378

and phenyl rings in coumarin displayed a singlet at 12.25 ppm. A multiplet between 7.80–8.34 ppm was observed due to six aromatic protons. All thiazolylcoumarin derivatives were distinguished by the existence of coumarin H-4 at 9.251 ppm. *p*-Disubstituted benzene was suggested by a doublet at 7.562 and 7.663 ppm (Figure 3d). Aromatic carbons in **5a** were confirmed by peaks at 112.1, 114.1, 115.6, 116.9, 120.1, 121.9, 126.5, 128.9, 129.1, 129.5, 129.7, 130.5, 133.2, 134.1, 135.8, 136.4, 137.2, 144.8, and 152.7 ppm in ^{13}C-NMR spectra. Conversely, carbonyl carbon was confirmed at 169.3 ppm, methyl carbon in the benzene ring at 21.06 ppm, and imine carbon at 159.1 ppm. Similarly, other thiazolylcoumarin derivatives **5a**–**5k** were confirmed from their corresponding ^{1}H-NMR spectra (Figure 3d).

3.3 | Fluorescence studies

All the thiazolylcoumarin derivatives (**5a**–**5k**) demonstrated moderate fluorescence data, as shown in Table 3. All the prepared derivatives met the requirements for fluorescence, as the thiazolylcoumarin derivatives had no rotational or vibrational signals and were markedly conjugated. Fluorescence spectra for the thiazolylcoumarin derivatives (**5a**–**5k**) were generated in chronological order from a 385 nm source, as shown in Figure 4(a–c), at different excitation wavelengths. The fluorescence spectra demonstrated the effect of intensity at specific wavelengths when varying the excitation wavelengths. All fluorescent dyes exhibited a single emission peak only, but with different emission intensities. The **5h** dye displayed the maximum fluorescence intensity on excitation of the solution at 10^{-7} to 10^{-8} M concentrations (Figure 4) at 505 nm wavelength. In addition, ethanolic solutions showed the highest fluorescence intensity, whereas DMF and ethyl acetate exhibited smaller fluorescence intensities that might be due to the presence of thiazoles in these solvents that produced a decrease in fluorescence intensity. This higher fluorescence intensity make these compounds extremely suitable for bio-labelling and bio-imaging.

3.4 | Time-resolved study of thiazolylcoumarins derivatives (5a–5k)

A radiation emission study was carried out using TRPL spectroscopy. Absolute ethanol was used as a solvent to prepare thiazolylcoumarin derivatives (**5a**–**5k**) sample solutions for TRPL. This study used the time-correlated single-photon counting technique (TCSPT). TRPL measurements were performed at room temperature using a laser beam (305 nm) as an excitation source with 25 ps of time resolution. Thiazolylcoumarin derivative decay times have been used as a factor in TRPL measurements and photoluminescence (PL) decay kinetics is biexponential. The relative decay time of thiazolylcoumarin derivatives is shown in Figure 4(d). The thiazolylcoumarin derivatives were excited with 305 nm laser radiation at 25°C. Zero time values were measured after the laser pulse; PL from several decay time gates was recorded instantly after excitation by the laser pulse. After excitation of the thiazolylcoumarin derivatives with radiation, the time taken for the electron–hole pair to recombine was termed the radiative period. A biexponential decay function was used to fit the experimental data from the decay profile. These results showed that the excitation

TABLE 3 Fluorescence data of thiazolylcoumarin derivatives (**5a**–**5k**) in ethanol, ethyl acetate, and DMF

Dye	$\lambda_{Exitation}$ (nm)	$\lambda_{Emission}$ (nm)			Stokes' shift		
		Ethanol	DMF	Ethyl acetate	Ethanol	DMF	Ethyl acetate
5a	445	452	450	451	16	17	11
5b	445	460	490	530	18	45	75
5c	450	464	461	460	15	25	18
5d	450	461	520	560	10	60	94
5e	450	466	510	490	11	62	45
5f	436	455	456	450	14	19	22
5g	440	456	4950	485	16	58	47
5h	460	507	500	496	45	47	28
5i	440	459	495	4900	15	60	64
5j	440	453	495	500	16	50	60
5k	443	459	530	526	14	76	70

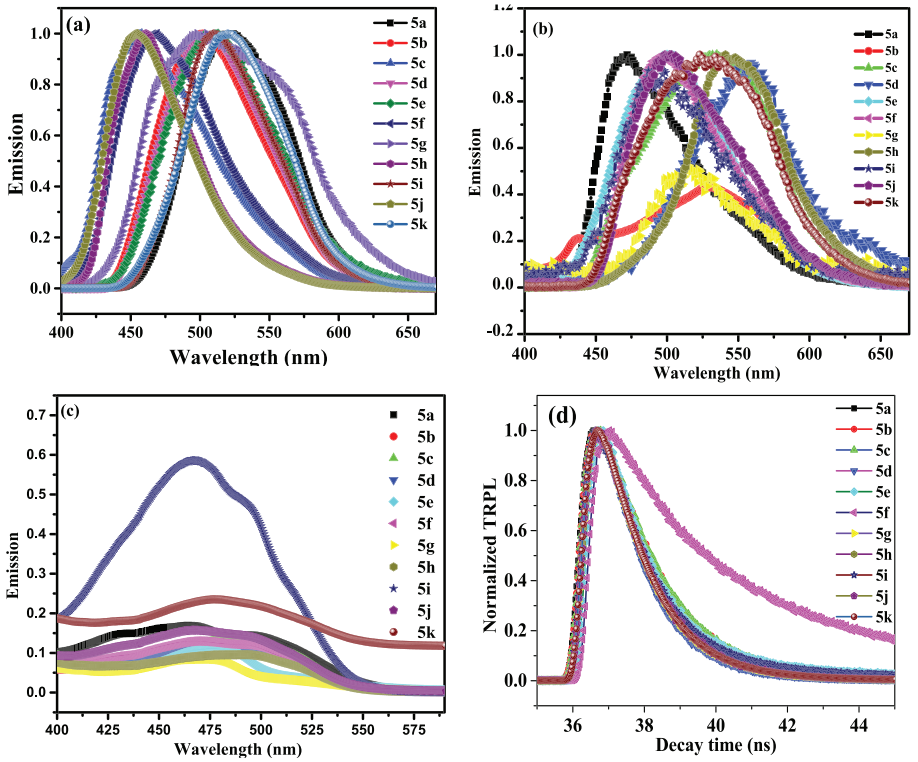


FIGURE 4 Fluorescence spectra of thiazolylcoumarin derivatives (**5a**–**5k**) in (a) ethanol, (b) ethyl acetate, and (c) DMF. (d) Combined fitted decays in TRPL plot of thiazolylcoumarin derivatives (**5a**–**5k**)

lifetime decreased to a very limited extent and appeared almost the same, except for thiazolylcoumarin derivative (**5i**), which had a relatively long lifetime compared with the other thiazolylcoumarin derivatives. Longer lifetime and a less nonexponential decay for the exciton PL was due to a reduction in the energy transference rate. Exciton lifetime was decreased for other thiazolylcoumarin derivatives and the decay curve was increasingly nonexponential as the detection was moved to higher energies, possibly due to the creation of additional discrete energy levels for the depopulation of the excited state.

3.5 | Electrochemical study of thiazolylcoumarin derivatives (5a–5k)

Electrochemical characteristics of thiazolylcoumarin derivatives (**5a**–**5k**) were investigated using cyclic voltammograms (CV) and a glassy carbon electrode, and 0.1 M $TBAPF_6$ in DMF as the electrolyte. The CV (Figure 5) were used to estimate lowest unoccupied molecular orbital (LUMO) and highest occupied molecular orbital (HOMO) energy value redox potentials (Table 4).

3.5.1 | Redox potentials ($E_{1/2}$) of thiazolylcoumarin derivatives

Thiazolylcoumarin derivatives **5c** and **5d** have Br and a chlorophenyl ring attached to thiazole and had the highest redox potential; **5i** has a thiophene ring on the chromophore and produced the lowest value (Table 4). The higher redox potential value might be due to the dual

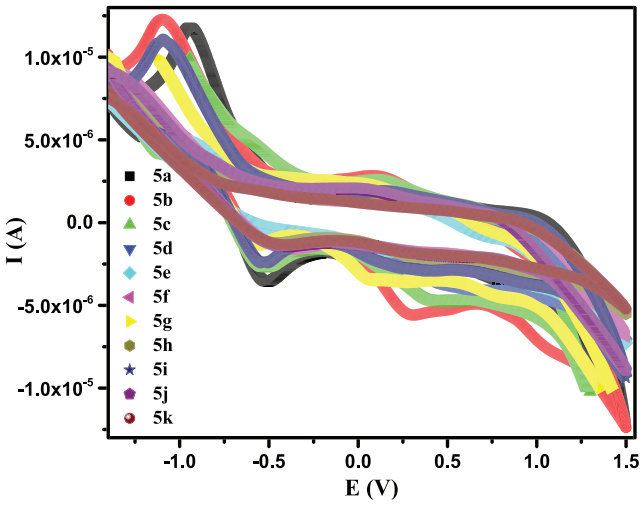


FIGURE 5 Cyclic voltammogram of thiazolylcoumarin derivatives (**5a**–**5k**) in ethanol

performance of halogens (as electron donor and acceptor). Conversely, the lowest redox potential value was seen for **5i** and was due to sulfur atoms in the thiophene ring that had electron-donating effects and mutable oxidation states.

3.5.2 | Bandgap energy (E_g) of thiazolylcoumarin derivatives (5a–5k)

To calculate the optical bandgap values, a standard previously reported procedure was used. Every solid has an energy-band value that is

TABLE 4 Bandgap energy and LUMO/HOMO energy levels of thiazolylcoumarin derivatives (**5a**–**5k**)

	LUMO energy levels		HOMO energy levels	
Sr. no.	$E_{1/2}$ (V)	LUMO (eV)	E_g (V)	HOMO (eV)
5a	−0.7232	−4.07	3.51	−7.58
5b	−0.83	−3.97	3.38	−7.35
5c	−0.817	−3.98	3.25	−7.23
5d	−0.806	−3.99	3.60	−7.59
5e	−0.465	−3.94	3.71	−7.65
5f	−0.01	−4.39	3.44	−7.83
5g	−0.601	−4.20	3.29	−7.49
5h	−0.488	−3.99	3.68	−7.67
5i	−0.807	−3.99	3.30	−7.29
5j	−0.469	−4.86	3.28	−8.14
5k	0.361	−5.16	3.35	−8.54

responsible for its electronic characteristics. The bandgap energy values for the coumarinyl derivatives are given in Table 4 and ranged from 3.25 to 3.71 eV. The bandgap energy value was maximum for **5d**, **5e**, **5h**, and lowest for **5c**, **5g**, and **5j**, and was dependent on the precursor used and as well as on the substituents. Br and NO_2 groups involved in **5c** and **5f** are at *m* to each other and act as donor and acceptor groups. When increasing the electron density, energy levels become closer and, therefore, the bandgap energy value is reduced.

3.5.3 | LUMO and HOMO energy levels of thiazolylcoumarin derivatives (5a–5k)

To evaluate the LUMO energy related to the vacuum level, redox data were standardized by a ferricenium/ferrocene pair taking an absolute energy value of −4.8 eV. Electron-donating groups (EDG) on the coumarinyl side lowered the LUMO energy, and vice versa; LUMO energies shown in Table 4 are in the range −4.94 eV to −5.16 eV. LUMO energy varies through increases in delocalization of electrons and energy modifications decrease by increasing conjugation, and vice versa. The lowest LUMO energy was found for **5i**, based on chlorothiophene, in which sulfur provided electrons and decreased the LUMO energy. Table 4 illustrates the HOMO energy level values in the range −7.23 to −8.54 eV for **5a**–**5k**. The pyridine and thiophene rings attached to chromophores lowered the HOMO energy levels for **5h** and **5i**; **5f** had the highest energy levels due to a nitro group inductive effect. Electron-withdrawing groups (EWG) increase energy, whereas EDG decrease energy as the energy gaps between the HOMO and the LUMO are increased.

4 | CONCLUSION

Novel thiazolylcoumarins derivatives (**5a**–**5k**) were synthesized with a high yield through a multistep process. Yellow-coloured thiazolylcoumarins derivatives (**5a**–**5k**) produced strong absorption in the 370–400 nm range. UV–vis light absorption of these novel thiazolylcoumarins derivatives (**5a**–**5k**) was determined in ethanol, ethyl acetate, and DMF solvents; the highest value was obtained for DMF as the polar solvent shifted the π–π^* transition wavelength to a higher value. Thiazolylcoumarins showed a solvatochromic effect in absorbance and fluorescence. This effect was observed in different solvents, but with increase in the polarity of the solvents, a bathochromic shift was observed. The maximum emission and a higher Stokes' shift were observed for **5h**. Thiazolylcoumarins derivatives containing CH_3O– groups had high HOMO energy levels and the lowest bandgap. The as-synthesized thiazolylcoumarin derivatives showed a sharp emission in the 430–510 nm range in fluorescence spectra and UV absorption in the 330–400 nm range. These high fluorescence properties make the as-synthesized thiazolylcoumarin derivatives useful for bio-imaging and bio-labelling applications.

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CONFLICT OF INTEREST

There is no conflict of interest.

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SUPPORTING INFORMATION

Additional supporting information may be found online in the Supporting Information section at the end of this article.

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