

# Thermal analyses and spectral characterization of some synthesized metal(II) Schiff base complexes

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**Abstract** Schiff base metal complexes derived from 2-thiophenecarboxylidene-4-anisidine, 3,4-dihydroxy-5-nitrobenzylidene-2-amino-5-methylthiazole and 3,4-dihydroxy-5-nitrobenzylidene-4-chloroaniline have been synthesized and characterized by elemental analysis, IR, UV–Vis, molar conductance and thermal analyses. The complexes are coloured and stable in air at room temperature. The complexes exhibit coordination number to be 4 and 6. The thermal behaviour of metal complexes shows that the hydrated complexes lose water molecules of hydration in the first and then is immediately followed by decomposition of ligand molecules in the subsequent steps.

**Keywords** Schiff base complexes · Spectral analyses · Thermal analyses

## Introduction

Association of a metal ion with an organic ligand through a chemical bond, changes drastically the properties of ligand and metal. It stabilizes the system and changes the solubility and reactivity. Metal complexes of Schiff bases are studied extensively due to synthetic flexibility of these compounds and their selectivity as well as sensitivity towards the central metal atom [1]. The information on the thermal stability of diverse materials is important to study

the complexes in material science technology. The term thermal analyses incorporate the technique in which some physical parameters of the system are determined or recorded as the function of temperature [2–5]. Thermogravimetric analysis is one of the oldest thermal analytical procedures and has been used extensively in the study of polymeric systems. Among the different thermal methods, the most widely used techniques are TG, DTA and DSC which find extensive use in all fields of inorganic, organic chemistry, metallurgy, mineralogy, etc. TG and DTA are used to obtain the maximum usable temperature and rate of determination of materials in high temperature environments of science. Thermogravimetric studies can also be supported by X-ray crystallography, infrared and DTA studies so that possible modes of dehydration and decomposition can be established. When the thermogravimetry is used in association with other physicochemical techniques, its field of application widens and provides a clear geometry for the complexes [6–10].

We report herein Schiff base complexes viz.  $[\text{Fe}(\text{TCA})_2(\text{H}_2\text{O})_2] \cdot \text{SO}_4 \cdot \text{H}_2\text{O}$  (**1**),  $[\text{Zn}(\text{DAT})(\text{H}_2\text{O})_2] \cdot \text{Cl}_2 \cdot \text{H}_2\text{O}$  (**2**) and  $[\text{VO}(\text{DCA})_2(\text{H}_2\text{O})_2] \cdot \text{SO}_4 \cdot 2\text{H}_2\text{O}$  (**3**). The present course of study was initiated with the preparation, spectral characterization and thermal analyses of the complexes of metal with relevant ligands.

## Experimental

### Apparatus and reagents

All the used chemicals and solvents were of A. R. grade. All the reagents used for the preparation of the Schiff bases were obtained from Sigma Aldrich. Metal salts were purchased from Loba Chemie. Elemental analyses were

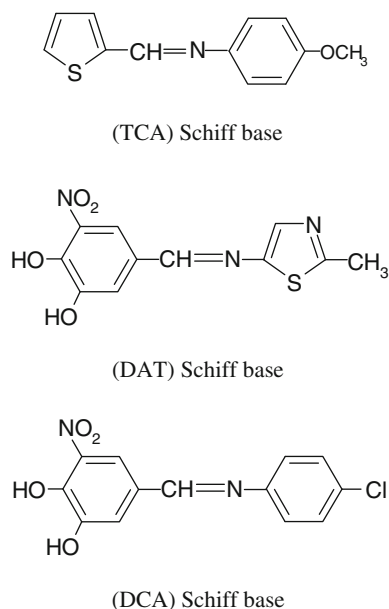
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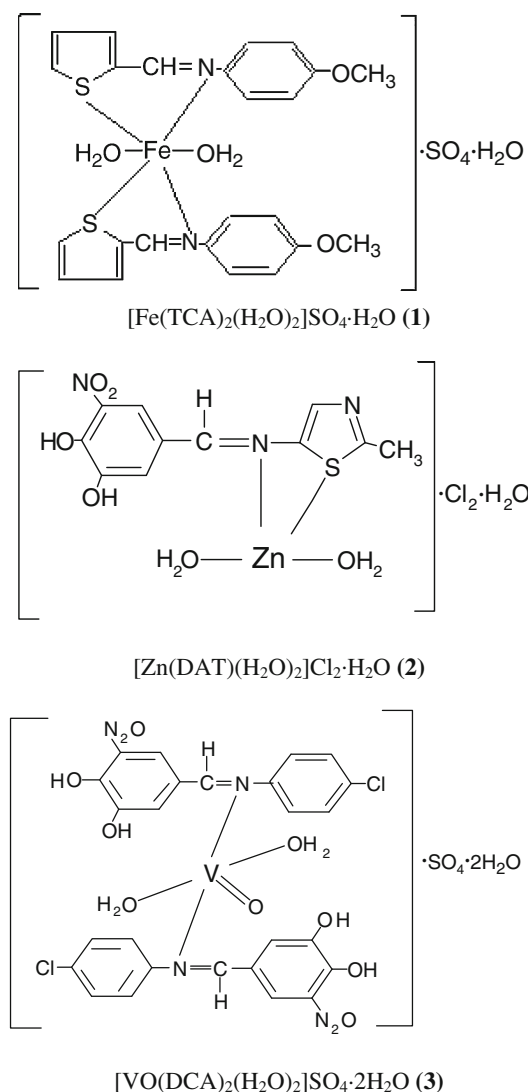
performed on an Elemental Vario EL III Carlo Erba 1108 analyzer. Molar conductance measurements were conducted using  $10^{-3}$  M solutions of the complexes in DMSO on Elico-CM 82 Conductivity Bridge at room temperature. FT-IR spectra were recorded in KBr pellet on a Perkin Elmer RX1 spectrophotometer in wave number region  $4,000\text{--}400\text{ cm}^{-1}$ . Electronic spectra (in DMSO) were recorded on Perkin Elmer Lambda-2B-spectrophotometer. Magnetic susceptibility measurements were carried out on a Gouy balance at room temperature using mercuric tetrathiocyanatocobaltate(II) as the calibrant. Thermal measurements were carried out on Perkin Elmer Thermal Analyzer in nitrogen atmosphere with a heating rate  $10\text{ }^{\circ}\text{C min}^{-1}$  from room temperature to  $800\text{ }^{\circ}\text{C}$  at I.I.T., Roorkee.

### Synthesis of Schiff bases

The Schiff bases (TCA, DAT and DCA) (Fig. 1) have been synthesized by the condensation of equimolar ratio of 2-thiophenecarboxaldehyde with 4-anisidine (TCA) and 3,4-dihydroxy-5-nitrobenzaldehyde with 2-amino-5-methylthiazole (DAT) and 4-chloroaniline (DCA) dissolved in ethanol. The resulting reaction mixture was stirred well, refluxed for 3–4 h and then allowed to cool overnight. The coloured solid precipitate of Schiff base obtained was filtered, washed with cold ethanol several times and dried under reduced pressure in a desiccator. The purity of synthesized compounds was checked by TLC using silica gel G (yield: 82–85 %).



**Fig. 1** Suggested structure of Schiff base ligands



**Fig. 2** The proposed structures of the Schiff base metal complexes

### Synthesis of metal complexes

Schiff base metal(II) complexes (Fig. 2) were prepared by refluxing ethanolic solution of metal salts with Schiff bases (TCA, DAT and DCA) in 1:1 and 1:2 molar ratio on a water bath for about 7–10 h. A coloured product appeared on standing and cooling the above solution. The precipitated complex was, filtered washed with ether and re-crystallized with ethanol several times and dried under the reduced pressure over anhydrous  $\text{CaCl}_2$  in a desiccator. It was further dried in electric oven at  $50\text{--}70\text{ }^{\circ}\text{C}$  (yield: 64–70 %).

### Results and discussion

All the metal complexes are coloured, solid and stable towards air and moisture at room temperature. They

decompose on heating at high temperature, more or less soluble in common organic solvents. Analytical data of the compounds, together with their physical properties are consistent with proposed molecular formula are given in Table 1. The observed molar conductances of the complexes in DMSO at room temperature are consistent with the electrolytic nature of the complexes.

### IR spectra

The IR spectra of the complexes were compared with those of the free ligand in order to determine the involvement of coordination sites in chelation. Characteristic peaks in the spectra of the ligand and complexes were considered and compared.

IR spectrum of Schiff base (TCA) ligand shows a strong band at  $1,614\text{ cm}^{-1}$  due to  $\nu(\text{C}=\text{N})$  azomethine group. This shifted down in Fe(II) complex and appear at  $1,589\text{ cm}^{-1}$ , suggesting participation of azomethine nitrogen in chelation. A strong ligand band was observed at  $852\text{ cm}^{-1}$ , assignable to thiophene  $\nu(\text{C}-\text{S}-\text{C})$ . On coordination of metal ion with sulphur of thiophene, the band position of  $\nu(\text{C}-\text{S}-\text{C})$  group has shifted to lower side by  $16\text{ cm}^{-1}$ , in complex. The appearance of broad band at  $3,452$  and  $815\text{ cm}^{-1}$  in the spectrum of Fe(II) complex, have been assigned to associated and coordinated water molecules, respectively. In Fe(II) complex new bands at  $506$  and  $412\text{ cm}^{-1}$  have been assigned to  $\nu(\text{M}-\text{N})$  and  $\nu(\text{M}-\text{S})$ , respectively.

The IR spectrum of Schiff base (DAT) shows a strong band at  $1,622\text{ cm}^{-1}$  due to  $\nu(\text{C}=\text{N})$  azomethine group. On the chelation with Zn(II) ion it has shifted down by  $22\text{ cm}^{-1}$ , due to coordination of the azomethine nitrogen to the central metal ion. The ligand spectrum shows band at  $1,398\text{ cm}^{-1}$  due to deformation of phenolic  $-\text{OH}$  and an

intense band at about  $1,271\text{ cm}^{-1}$  due to phenolic  $\text{C}-\text{O}$ . These bands remain unaltered in complex indicating no deprotonation of phenolic  $-\text{OH}$  group and its non-involvement in chelation. Thiazole ring  $\nu(\text{C}-\text{S}-\text{C})$  shows absorption band at  $842\text{ cm}^{-1}$  in the ligand spectrum. This band shows lower shift  $818\text{ cm}^{-1}$  in complex, suggesting participation of thiazole  $\nu(\text{C}-\text{S}-\text{C})$  sulphur in complexation. The appearance of broad band around  $3,406\text{ cm}^{-1}$  in complex is due to  $\nu(\text{O}-\text{H})$  water molecules. Lowering of this band in some complexes gives inference about hydrogen bonding. A medium intensity band at  $790\text{ cm}^{-1}$  suggests the presence of coordinated water in Zn(II) complex. In Zn(II) complex new bands at  $491$  and  $408\text{ cm}^{-1}$  have been assigned to  $\nu(\text{M}-\text{N})$  and  $\nu(\text{M}-\text{S})$ , respectively.

The  $\nu(\text{C}=\text{N})$  azomethine band observed at  $1,614\text{ cm}^{-1}$  in the spectrum of the Schiff base ligand, shows a lower shift by  $20\text{ cm}^{-1}$  in the VO(II) complex. This suggests participation of the azomethine group in coordination. The ligand spectrum shows bands at  $1,396$  and  $1,276\text{ cm}^{-1}$  due to phenolic  $-\text{OH}$  and phenolic  $\text{C}-\text{O}$  groups. These bands remain unaltered after complexation. These suggest no deprotonation and no coordination of the phenolic  $-\text{OH}$  group with metal ions. Thus, the ligand acts as a monodentate, coordinating through the  $\nu(\text{C}=\text{N})$  group only. The appearance of broad band around  $3,151\text{ cm}^{-1}$  in complex may be due to  $\nu(\text{O}-\text{H})$  of water molecules. A band at  $802\text{ cm}^{-1}$  suggests the presence of coordinated water in VO(II) complex, The new weak band at  $498\text{ cm}^{-1}$  in complex has been assigned to  $\nu(\text{M}-\text{N})$  mode [11, 12].

### Electronic spectra and magnetic moment

The electronic spectral data of the metal complexes observed in DMSO solution. The nature of the ligand field

**Table 1** Analytical and physical data of metal complexes and ligands

Complex no.	Compounds/molecular formulae mol. mass/colour	Yield/%	Elemental analysis; found/calc. /%				$\mu_{\text{eff}}$	B. M.	$^a \Lambda_m$
			C	H	N	Metal			
1.	$\text{C}_{12}\text{H}_{11}\text{NOS}$ (TCA) 217/Dark grey	85	66.30/66.36	5.08/5.07	6.38/6.45	–	–	–	
	$[\text{Fe}(\text{TCA})_2(\text{H}_2\text{O})_2]\text{SO}_4 \cdot \text{H}_2\text{O}$ 640/Dark brown	67	45.02/44.0	3.30/3.44	4.88/4.37	8.52/8.70	5.30	95.5	
2.	$\text{C}_{11}\text{H}_9\text{N}_3\text{SO}_4$ (DAT) 279/Fibrous orange	84	47.07/47.31	3.09/3.23	14.98/15.05	–	–	–	
	$[\text{Zn}(\text{DAT})(\text{H}_2\text{O})_2]\text{Cl}_2 \cdot \text{H}_2\text{O}$ 469/Brown golden	70	28.67/28.14	1.77/1.92	8.46/8.95	13.4/13.9	Diam.	105.6	
3.	$\text{C}_{13}\text{H}_9\text{N}_2\text{Cl}$ (DCA) 292/Deep orange	82	54.46/53.42	3.09/3.08	9.39/9.59	–	–	–	
	$[\text{VO}(\text{DCA})_2(\text{H}_2\text{O})_2]\text{SO}_4 \cdot 2\text{H}_2\text{O}$ 819/Black	64	38.50/38.09	2.62/2.20	7.01/6.83	8.45/8.10	1.81	105.0	

<sup>a</sup>  $\Lambda_m = \Omega^{-1}\text{ cm}^2/\text{mol}$

around the metal ion has been deduced from the electronic spectra. The Fe(II)–TCA complex exhibits one band at  $19,417\text{ cm}^{-1}$ , which may be attributed to the transition  ${}^5T_{2g} \rightarrow {}^5E_g$  for octahedral complex. The magnetic moment of Fe(II) complex is 5.30 B.M. Zn(II)–(DAT) complex did not show  $d-d$  transition due to diamagnetic nature of Zn(II)–(DAT) complex, therefore tetrahedral geometry for Zn(II) complex has been suggested. The electronic spectrum of VO(II)–(DCA) complex displayed two bands at  $13,698$  and  $18,867\text{ cm}^{-1}$ , assignable to transitions  ${}^2B_2 \rightarrow {}^2E$  ( $\nu_1$ ) and  ${}^2B_2 \rightarrow {}^2B_1$  ( $\nu_2$ ), respectively. The geometry of this one coordinated complex can be described in terms of square pyramid geometry. The value of the magnetic moment for this complex is 1.81 B.M., indicating the presence of one unpaired electron [13, 14].

### Thermal analyses

TG, DTA and DTG thermal studies of complexes have been recorded in nitrogen atmosphere. Thermal analysis data of complexes are given in Table 2. The thermal behaviour of metal complexes shows that the hydrated complexes lose molecules of hydration first; followed by decomposition of ligand molecules in the subsequent steps.

As the evident from TG, DTA and DTG curves of  $[\text{Fe}(\text{TCA})_2(\text{H}_2\text{O})_2] \cdot \text{SO}_4 \cdot \text{H}_2\text{O}$  (**1**) complex is found to be stable up to  $60\text{ }^\circ\text{C}$ . Elimination of one lattice water molecule has been observed on raising the temperature up to

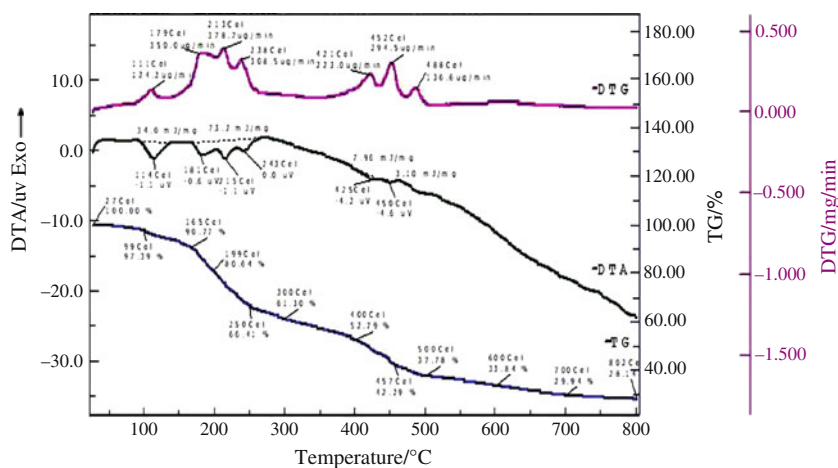
$100\text{ }^\circ\text{C}$ . After  $100\text{ }^\circ\text{C}$  weight loss has been observed up to  $170\text{ }^\circ\text{C}$ , correspond to two coordinated water molecules. After that three main steps have been found; Step-1 above  $170\text{ }^\circ\text{C}$  temperature, a gradual weight loss has been found up to  $250\text{ }^\circ\text{C}$ , correspond to the release of sulphate moiety and methoxy groups from the metal chelate (Remaining mass %, obs/calc., 66.87/66.41). The DTA and DTG curve of this complex shows peaks at  $181$ ,  $215$  and  $243\text{ }^\circ\text{C}$  (endothermic) and  $179$ ,  $213$  and  $238\text{ }^\circ\text{C}$ , respectively. An endothermic peak at  $114\text{ }^\circ\text{C}$  in the DTA curve of Fe(II)–TCA is also observed which may be due to the change in composition of the complex. Step-2, after  $250\text{ }^\circ\text{C}$  a slow weight loss continues up to  $450\text{ }^\circ\text{C}$ , indicates the loss of ligand parts (Remaining mass %, obs/calc., 43.13/43.00). The DTA curve depicts endothermic peaks at  $425$  and  $450\text{ }^\circ\text{C}$ , respectively, whereas the corresponding DTG curve show peaks at  $421$  and  $452\text{ }^\circ\text{C}$ , respectively. The decomposition of remaining ligand moiety occurs between  $450$  and  $750\text{ }^\circ\text{C}$  (Remaining mass %, obs/calc., 28.14/24.84). In this step, DTA and DTG curve of this complex show peak at  $460$  (endothermic) and  $488\text{ }^\circ\text{C}$ , respectively. After  $750\text{ }^\circ\text{C}$  a horizontal curve has been obtained up to  $800\text{ }^\circ\text{C}$ . This suggests the formation of an ultimate metal oxide in nitrogen atmosphere (Fig. 3) [14–23].

Study of TG curve, indicates that  $[\text{Zn}(\text{DAT})(\text{H}_2\text{O})_2] \cdot \text{Cl}_2 \cdot \text{H}_2\text{O}$  (**2**) complex is stable up to  $50\text{ }^\circ\text{C}$ . Elimination of one lattice water molecule has been observed on raising the temperature up to  $150\text{ }^\circ\text{C}$ . Weight

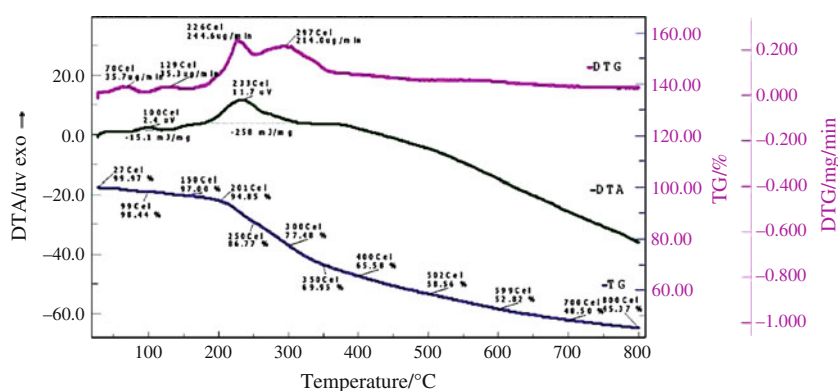
**Table 2** Thermal analyses data of complexes

Complex	Temperature range/ $^\circ\text{C}$	Remaining mass nbsp;% obs./calc.	Corresponding mass loss	DTA peak/ $^\circ\text{C}$	DTG peak/ $^\circ\text{C}$
$[\text{Fe}(\text{TCA})_2(\text{H}_2\text{O})_2] \cdot \text{SO}_4 \cdot \text{H}_2\text{O}$ ( <b>1</b> )	Room temp.	100/100	No loss	–	–
	60–100	97.39/97.18	One lattice water molecule	–	–
	100–170	89.35/91.56	Two coordinated water molecules	114 (endothermic)	111
	170–250	66.87/66.41	Sulphate + methoxy groups moiety	181 (endothermic)	179
				215 (endothermic)	213
				243 (endothermic)	238
	250–450	43.13/43.00	Ligand part	425 (endothermic)	421
			450 (endothermic)	452	
450–750	28.14/24.84	Ligand moiety	460 (endothermic)	488	
$[\text{Zn}(\text{DAT})(\text{H}_2\text{O})_2] \cdot \text{Cl}_2 \cdot \text{H}_2\text{O}$ ( <b>2</b> )	50–150	97.00/96.16	One lattice water molecule	100 (exothermic)	129
	150–225	91.85/88.48	Two coordinated water molecules	–	–
	225–325	73.00/73.35	Chloride moiety	233 (exothermic)	226
				–	297
325–800	45.37/40.94	Ligand moiety (metal oxide)	370–800 (br) (endothermic)	575	
$[\text{VO}(\text{DCA})_2(\text{H}_2\text{O})_2] \cdot \text{SO}_4 \cdot 2\text{H}_2\text{O}$ ( <b>3</b> )	40–100	92.70/95.60	Two lattice water molecules	64 (endothermic)	58
	100–200	87.78/91.20	Two coordinated water molecules	–	175
	200–500	53.98/52.38	Two chloroaniline and sulphate moiety	274 (exothermic)	270
				375 (exothermic)	368
500–800	37.81/32.84	Ligand moiety	500–800 (br) (endothermic)	750 (br)	

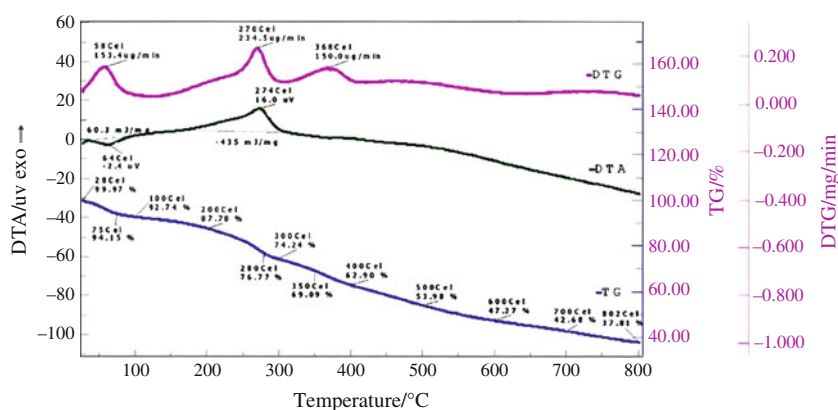
**Fig. 3** TG, DTA and DTG curves of  $[\text{Fe}(\text{TCA})_2(\text{H}_2\text{O})_2]\text{SO}_4 \cdot \text{H}_2\text{O}$  (1) complex



**Fig. 4** TG, DTA and DTG curves of  $[\text{Zn}(\text{DAT})(\text{H}_2\text{O})_2]\text{Cl}_2 \cdot \text{H}_2\text{O}$  (2) complex



**Fig. 5** TG, DTA and DTG curves of  $[\text{VO}(\text{DCA})_2(\text{H}_2\text{O})_2]\text{SO}_4 \cdot 2\text{H}_2\text{O}$  (3) complex



loss during 150–225 °C; correspond to two coordinated water molecules. An exothermic peak at 100 °C in DTA curve of complex is also observed, which may be due to the change in composition of complex. Above this temperature, the complex degrades in two steps: Step-1; above 225 °C temperature, a gradual weight loss has been found up to 325 °C, this corresponds to decomposition of chloride moiety from the metal chelate (Remaining mass %, obs/calc., 73.00/73.35). In this step, DTA and DTG curve of this complex show peaks at 233 (exothermic) and 226,

297 °C, respectively. Step-2; above 325 °C a slow weight loss has been observed up to 800 °C indicating loss of remaining ligand moiety (Remaining mass %, obs/calc., 45.37/40.94). In this step, DTA as broad peak appears at 370–800 °C (endothermic), whereas DTG peak at 575 °C (Fig. 4) [14–23].

A careful analysis of TG curve, indicates that  $[\text{VO}(\text{DCA})_2(\text{H}_2\text{O})_2]\text{SO}_4 \cdot 2\text{H}_2\text{O}$  (3) complex is stable up to 40 °C. Elimination of two lattice water molecules has been observed on raising the temperature up to 100 °C. An

endothermic peak at 64 °C in the DTA curve of the complex has been recorded, which may be due to change in composition of the complex. After 100 °C weight loss has been observed up to 200 °C, corresponding to two coordinated water molecules. Above 200 °C temperature, a gradual weight loss was observed up to 500 °C, corresponding to release of two chloroaniline moiety and sulphate from the metal chelate (Remaining mass %, obs/calc., 53.98/52.38). In this stage the DTA curve depict exothermic peak at 274 °C, whereas the corresponding DTG curve show peaks at 270 and 368 °C. The subsequent step 500–800 °C corresponds to the removal of the remaining part of organic ligand, leaving metal oxide as residue (Remaining mass %, obs/calc., 37.81/32.84). In this step, DTA as broad peak appears at 500–800 °C (endothermic), whereas in DTG very low intensity broad appears around 750 °C (Fig. 5) [14–23].

## Conclusions

In the present research studies, our effort was to synthesize some new compounds. These synthesized compounds have been characterized by various physicochemical and spectral techniques. Thermal data show degradation pattern of the complexes. TG, DTA and DTG studies also guide to thermodynamic, kinetic and reactivity behaviour of materials and metal–ligand interaction.

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