

Thermal decomposition and their kinetics of mercury(ii) perchlorate complex with 4-aminopyridine and water

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ABSTRACT

Copper perchlorate complex with 4-aminopyridine and water has been prepared with molecular formula $[\text{Hg}_2(\text{C}_5\text{H}_6\text{N}_2)_3(\text{ClO}_4)_4] \cdot 2\text{H}_2\text{O}$. It has been characterised by elemental analysis, thermogravimetry, and IR spectroscopic data. Thermal behaviours have been studied by thermogravimetry (TG) in static air and simultaneous thermogravimetry-derivative thermogravimetry analysis (TG-DTG) in flowing nitrogen atmosphere. Complex decomposes in four steps (less resolved). Difference in decomposition under air and inert atmosphere has been also discussed. Kinetics of thermal decomposition has been investigated using isothermal TG data recorded at five different temperatures applying model-fitting as well as isoconversional method on these data. Model-fitting methods have yielded a single value of activation energy whereas isoconversional method has given different values of activation energy for each extent of conversion, α . Response of synthesized complex towards rapid heating has been investigated by recording explosion delay time (D_E) at five different temperatures and using these data kinetics of explosion has been analysed. Activation energy for explosion has been also calculated.

Keywords: 4-aminopyridine, perchlorate, complex, thermolysis, explosion delay, kinetics.

1. INTRODUCTION

When a complexing agent (ligand) ligated with a transition metal ion, resulting in metal nitrate and perchlorate complexes exhibit the properties of insensitive high energy materials [1-3]. These energetic complexes find applications in propellants, explosives and pyrotechnics. Many of these complexes have been found to act as a burn rate catalyst for propellants [4-9]. Mercury is one of the most toxic metal and known to form stable complexes. The complexes of mercury(II) (labile d^{10} complexes) differs from other transition metal complexes as it is a soft Lewis acid. It prefers sulphur and nitrogen donors over oxygen ones and this imparts biological and medical relevance as a potential toxin or as a medicine [10,11]. Mercury and its compounds have found interest due to its severe applications, as a preservative, in

cosmetics, in fluorescent lamps, in paints, in medicine, in pulp and paper industry and for mercury batteries [12-17]. Mercury(II) perchlorate complex with 5-hydrazinotetrazole has been reported as primary explosive sensitive to laser initiation [18-20]. Very recently several mercury(II) complexes with various ligands have also been studied [21-34]. 4-Aminopyridine, commercially known as ampyra has been used for sclerosis. It is a potassium channel blocker and is used for nervous disorders []. The present study will contain preparation, characterisation and thermolysis and their kinetics of the mercury(II) perchlorate complex with 4-aminopyridine and water as a ligand. Response of complex towards sudden high heat has also been investigated including kinetics of explosion.

2. EXPERIMENTAL SECTION

2.1. Materials.

Mercuric oxide, perchloric acid, 4-aminopyridine, methanol (s.d.fine), all of AR grade were used as received.

2.2. Preparation of the complex.

The complex has been prepared following two step procedure. In first step hydrated mercuric perchlorate was obtained reacting mercuric oxide with 60% perchloric acid followed by recrystalliation. In the second step, an ethanolic solution of mercuric perchlorate alcoholic solution of 4-aminopyridine was mixed with continuous stirring at room temperature. Brown colour precipitate was obtained further washed with ethanol and dried.

2.3.Characterisation.

Complex was Characterised by elemental analysis (C, H, N, Thermo Finnigan Flash EA 1112 CHNS analyzer), infrared [35-37] (Perkin Elmer FT-IR spectrometer), and thermogravimetry (Table1).

Table 1.FTIR and elemental analysis data of the complex.

IR		Element	% / (Obs/cald.)
ν / cm^{-1}	Assignments		
436/463	Hg-N(ring)/Hg-NH ₂	C	15.8/16.1
625, 1069	ν_4 and ν_3 of ClO ₄	N	7.5/7.3
1376	Aryl C-NH ₂	H	1.9/1.6
1481	Aryl C=C	Hg	37.3
1554	N-H def.		
1636	H ₂ O		
1637	Aryl C=N		
3251, 3273	N-H (NH ₂)		
3372	H-bonding		
3465	O-H/H ₂ O		

2.4. Thermal Analysis.

TG in static air (Figure 1) was recorded at 10 °C/min (mass 20 mg) using an indigenously fabricated TG apparatus [38]. Simultaneous TG-DTG-DTA traces of the complex were obtained

in flowing nitrogen atmosphere (100 ml/min) using sample mass 1.575 mg at a heating rate of 10 °C/min (Figure 1). Isothermal TG (Figure 2) has been recorded in static air using above said indigenously fabricated TG apparatus at five different temperatures (210, 220, 230, 240 and 250°C).

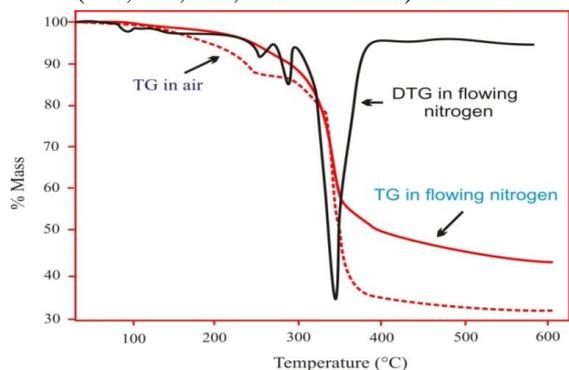


Figure 1. TG trace in static air and TG-DTG traces in flowing nitrogen atmosphere of the complex.

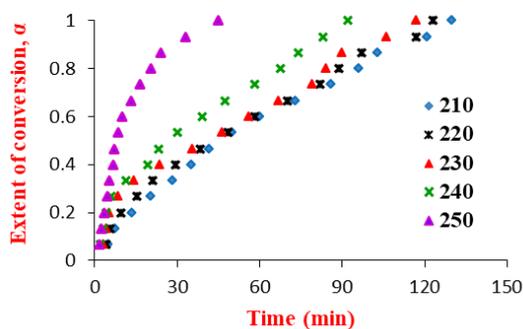


Figure 2. Isothermal TG of the complex.

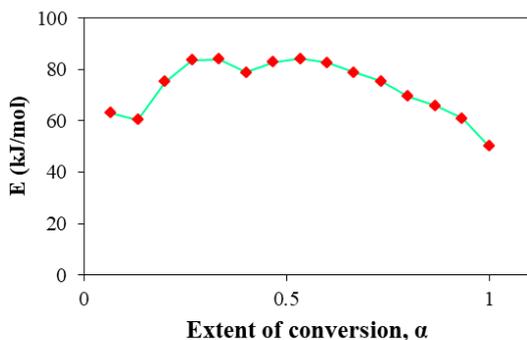


Figure 3. Variation of activation energy with extent of conversion, α .

2.5. Kinetic analysis. Kinetics of thermolysis has been studied using isothermal TG data using model fitting [39] (Table 2) and

3. RESULTS SECTION

Elemental analysis data (C, H, N) presented in Table 1 show a good agreement between the observed and calculated mass percentage of the elements which confirms the proposed molecular formula of the complex as $[\text{Hg}_2(4\text{-Apy})_3(\text{ClO}_4)_4] \cdot 2\text{H}_2\text{O}$. In the FTIR spectra, peaks at 436 and 463 cm^{-1} indicate Hg-N(ring) and Hg-N(NH_2) coordination respectively and a peak at 511 cm^{-1} reveals the formation of Hg-O bond. A sharp at 1090 cm^{-1} is specific of a non-coordinated ionic perchlorate. But in the present case, the corresponding peak has appeared at 1070 cm^{-1} with broadness, which merely indicates the coordination of oxygen atom of perchlorate ion. Sharp peaks at 3465 and 3373 cm^{-1} show the presence of non-coordinated hydrogen bonded water

isoconversional method [40, 41]. Variation in activation energy value with the extent of conversion, α is presented in Figure 3.

2.6. Explosion delay studies.

Explosion delay time was recorded using tube furnace technique [42] (sample mass 8 mg) at five different temperatures 340, 350, 360, 370 and 380 °C with an experimental limit of $\pm 1^\circ\text{C}$ (Table 3). Kinetics of explosion was investigated using D_E data fitted in Arrhenius equation.

$$D_E = A \exp E^*/RT \quad (1)$$

where A is the Arrhenius factor, E^* is the activation energy of explosion, T is absolute temperature and R, the gas constant. A plot of $\ln D_{EVS} 1/T$ is shown in Figure 4.

Table 2. Activation energy (E) and correlation coefficients (r) for the isothermal decomposition of the complex.

S.No.	Model	Slope	E (kJmole ⁻¹)	r	Mean dev	Stddev
1	Power law ^{3/4}	5827.9	48.5	0.8189	2.890	3.047
2	Power law ^{2/3}	5865.6	48.8	0.8192	2.782	2.934
3	Power law ^{1/2}	4940.0	49.4	0.8196	2.684	2.793
4	Power law ^{1/2}	6342.3	52.8	0.8185	2.433	2.566
5	Power law ⁻¹	6511.0	54.2	0.8167	2.418	2.550
6	Mampel (1- α)	7794.8	64.9	0.7951	1.921	2.027
7	$a \cdot e^{-3/4}$	5982.6	49.8	0.8223	2.729	2.877
8	$a \cdot e^{-2/3}$	7424.3	61.8	0.7964	2.434	2.567
9	$a \cdot e^{-1/2}$	7522.5	62.6	0.7965	2.251	2.375
10	cs	6951.9	57.9	0.8081	2.559	2.689
11	T-d-diff	7647.8	63.6	0.8384	2.637	2.781
12	cc	6651.5	55.4	0.8124	2.513	2.650
13	P-T	7428.9	61.8	0.7946	1.619	1.710
14	G-B	7041.5	58.6	0.8079	3.056	3.222

Table 3. Explosion delay, energy of activation for thermal explosion (E^*) and correlation coefficient (r) of synthesized complex.

D_E/s at temperature / °C					E^*/kJmol^{-1}	r	$\ln k$
340 \pm 1	350 \pm 1	360 \pm 1	370 \pm 1	380 \pm 1			
75	66	60	52	47	39.1	0.9997	-3.3481

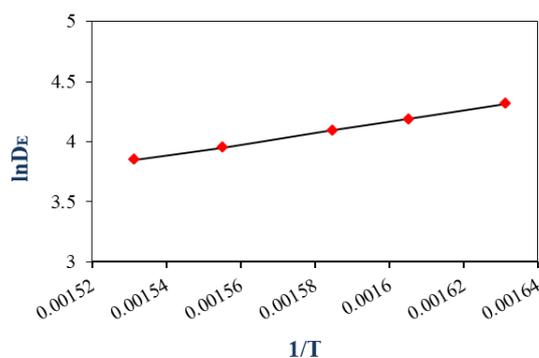


Figure 4. A plot of $\ln D_{EVS} 1/T$.

molecules. Other peaks presented in Table 1 are according to the standard text [35-37]. In the light of above discussion the tentative structure (Figure 5) of the complex has been proposed in which mercury atom is hexacoordinated. Out of six coordination sites, four are ligated with O-atoms of two ClO_4^- ion and two sites are ligated by two N-atoms, one belongs to ring of 4-aminopyridine molecule and other belongs to NH_2 group of another 4-aminopyridine molecule. Thus, a mercury atom is coordinated with two 4-aminopyridine molecules and complex takes the form of a polymer. Water molecules are hydrogen bonded to the coordinated ClO_4^- ion.

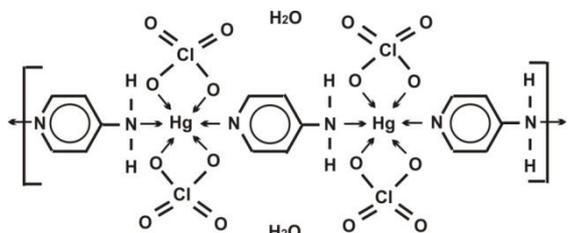
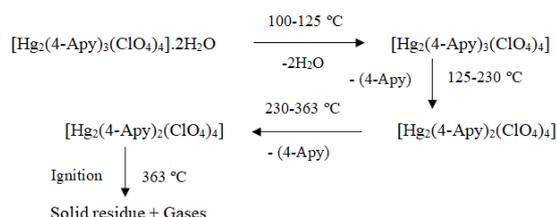


Figure 5. Proposed polymeric structure of the complex.

Thermogravimetric (TG) trace observed in static air and in flowing N_2 atmosphere (Figure 1) shows that the complex decomposes in four steps as indicated by the DTG also. In the first step, two H_2O molecules (3.2%) leave out in between the temperature range 100-125 °C. In the second step (125-230 °C) about 9% mass given off which corresponds to one 4-apy molecule (calculated 8.4%). In the third step second 4-apy molecule releases after that remaining residue decomposes (IV step) with ignition giving fumes, light, a noise and a sharp weight loss in TG traces. In air atmosphere ignition of complex has occurred at 363 °C. In inert atmosphere, fourth step decomposition is gradual relative to the decomposition in oxidative air atmosphere. Rate of decomposition and final residue left in N_2 atmosphere is higher than in air atmosphere due to the oxidative nature of air. The residue left after ignition nearly corresponds to HgO which further degrades on elevated temperatures. Thus, on the basis of the above information the decomposition pathway for the complex can be given as:



4. CONCLUSIONS

Polymeric mercury complex has been prepared and characterised. The complex decomposes in less distinguished four steps. First and second step corresponds to the removal of $2H_2O$ and one of the 4-apy moiety respectively. In third step two more 4-apy molecule releases which are followed by ignition (IV step). Model-

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Figure 6. Proposed thermal decomposition pathways for the complex

The fast decomposition of energetic complexes is caused by chain type propagative reaction which is due to the presence of oxidising (ClO_4) and reducing group (4-Apy) in the same molecule. Since the complex itself contains oxidising group, it shows a similar pattern of thermolysis in air (real) as well as in nitrogen (inert) atmosphere.

Kinetics of isothermal decomposition of the complex (Figure 2) has been evaluated by both model-fitting and isoconversional method in between the temperature range of 210-250 °C for 30% decomposition which corresponds to the removal of two H_2O molecule and three 4-apy moieties. Fourteen kinetic models (Table 2) have been applied to investigate the kinetics of thermolysis in model-fitting method. Various kinetic parameters such as activation energy, slope, correlation coefficient, mean and standard deviation are presented in Table 2. The average activation energy value (E) was found to be 56.4 kJmol^{-1} . The model-fitting methods yield a single value of activation energy for such complex solid state thermolysis but the isoconversional method has yielded a series of activation energy values varying with the extent of conversion, α (Figure 4). This value of E varies between 50.1 to 84.1 kJmol^{-1} which reveals the change in mechanism of thermolysis as decomposition proceeds.

Yet the complex is stable at room temperature, but when kept under sudden high temperature it explodes with a noise, light and fumes. Explosion delay time has been recorded at five different temperature and using these data kinetics of explosion has been evaluated (Table 3). Activation energy of the explosion was found to be 39.1 kJmol^{-1} .

fitting methods have yielded a single value of activation energy but isoconversional method has shown that activation energy value changes with the extent of conversion, α . Complex is stable at room temperatures but explodes when kept under high temperatures.

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