



Pyrolysis kinetics of Mn (II) complex derived from Schiff base of 5-amino-1,2,3,4-thiatriazole with-ortho methoxy benzaldehyde

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ABSTRACT

The Mn (II) complex derived from the Schiff base of 5-amino-1,2,3,4-thiatriazole with ortho-methoxy benzaldehyde exhibits significant structural stability and functional versatility. This study explores its thermal decomposition behaviour, stability, and reaction kinetics using thermogravimetric analysis (TGA). The Schiff base ligand, acting as a bidentate donor, enhances the stability of the coordination complex through the electron-donating properties of nitrogen and sulphur atoms. Kinetic analysis, performed using Doyle's modified method and Freeman and Carroll's approach, identified an activation energy of 11.93 kcal/mol, a reaction order of $b = 0.43$, and an entropy of activation (ΔS) of -25.33 e.u., demonstrating the reliability of statistical models in correlating experimental and theoretical data. The findings provide valuable insights into the thermal stability and degradation pathways of the complex under different conditions. Additionally, the Mn (II) complex exhibits diverse applications, including catalysis, material science, and environmental remediation, along with antimicrobial and antioxidant properties, making it a promising candidate for pharmaceutical applications. Its potential extends to the development of Metal-Organic Frameworks (MOFs), thin films, electrochemical sensors, batteries, fuel cells, and supercapacitors, highlighting its significance across multiple scientific and industrial domains.

Keywords: Schiff base, Entropy of activation, Activation energy, Freeman and Carroll's.

INTRODUCTION

Researchers have shown significant interest in studying the thermal decomposition of metal complexes in the solid state, along with their kinetic evaluation [1]. Thermal analysis techniques play a vital role in various scientific fields, including biological, medical, polymer, and chemical sciences. Methods such as differential scanning calorimetry (DSC), differential thermal analysis (DTA), and thermogravimetric analysis (TGA) enable scientists to investigate how materials respond to temperature variations [2]. These techniques are particularly valuable for analysing thermal stability, phase transitions, and decomposition behaviour in materials such as polymers, pharmaceuticals, and biomaterials. In the pharmaceutical industry, they are instrumental in evaluating drug-excipient interactions, optimizing formulation processes, and ensuring product stability. Additionally, thermal analysis aids in understanding polymer composition, thermal transitions, and degradation characteristics [3].

Thermogravimetric analysis (TGA) is a highly effective thermal analysis technique that provides precise quantitative measurements of mass changes in materials as a function of temperature or time. Its ability to detect even the smallest weight variations makes it superior to other thermal analysis methods for studying thermal stability, material composition, and decomposition kinetics, including reaction processes and activation energy. TGA is particularly useful for examining physical and chemical transformations such as evaporation, desorption, and adsorption, making it an essential tool for comprehensive material characterization across a wide range of applications, including polymers, ceramics, metals, and composites [4-6]. Moreover, its integration with analytical techniques like mass spectrometry and Fourier-transform infrared spectroscopy (FTIR) allows for a more detailed investigation of gaseous by-products during decomposition. When applied to metal complexes, TGA plays a critical role in assessing their thermal behaviour, which is vital for their applications in catalysis, pharmaceuticals, and material science. By analysing weight loss patterns under controlled heating conditions, TGA helps identify decomposition stages, evaluate ligand stability, and determine metal content, thereby offering deeper insights into the oxidation states and degradation mechanisms of metal complexes. This study explores the thermogravimetric analysis of metal complexes to assess their thermal stability, investigate decomposition pathways, and understand their potential applications in high-temperature environments [7-9].

Schiff bases, distinguished by their azomethine (-C=N-) functional group, play a crucial role in coordination chemistry due to their strong binding capabilities. These compounds serve as effective ligands for transition metal complexes, offering diverse structural and electronic properties that make them valuable in various fields. The presence of donor atoms such as phenoxy oxygen, imine nitrogen, and sulphur enhances their coordination abilities, leading to the formation of stable metal-ligand frameworks [10-13]. This stability makes Schiff base complexes highly useful in catalysis, bioinorganic chemistry, material science, and pharmaceutical applications. Their catalytic efficiency has been particularly noted in enzyme inhibition, biological replication, and antimicrobial treatments. The ability of Schiff bases to form coordination compounds with transition metals underscores their significance in both synthetic and biological chemistry [14-16]. This paper examines their synthesis, structural attributes, and functional applications in modern research.

Schiff bases and their metal complexes have garnered significant research interest due to their diverse chemical and functional properties. Their strong ability to coordinate with toxic metal ions makes them valuable in metal chelation and environmental applications. Additionally, they serve as efficient catalysts in olefin hydrogenation, facilitate amino group transfer, and exhibit reversible oxygen-binding capabilities. These compounds are widely used as chelating ligands in coordination chemistry and have demonstrated medicinal potential, including anticancer, antiallergic, and antibacterial activities. Recent studies have particularly focused on Schiff base metal complexes derived from heterocyclic compounds, as nitrogen- and sulphur-containing ligands play a crucial role in biological systems and cancer treatment [17-19].

Manganese complexes play a crucial catalytic role in enzyme active sites, where redox-active manganese ions facilitate oxygen metabolism. Schiff base ligands, known for their N and O donor atoms, are highly suitable for forming stable manganese complexes. These ligands can be electronically and sterically modified by introducing substituents on the aromatic ring, enhancing the catalytic efficiency of manganese complexes. Additionally, they stabilize manganese in different oxidation states, regulating its catalytic activity. Manganese-based complexes are biologically significant, as seen in enzymes like Mn-peroxidase, Mn-ribonucleotide reductase, and superoxide dismutase. Studies on manganese chemistry have focused on structural models to analyse magnetic, structural, and electrochemical properties, leading to their application in catalytic and magnetic chemistry [12, 20, 21]. Schiff base-derived Mn (II) complexes have demonstrated a range of potential applications, including antibacterial, antiviral, anticancer, and catalytic activities. The choice of ligand and metal ion significantly influences the bioactivity of these complexes, as Schiff bases exhibit various biological properties such as enzyme inhibition, antioxidant effects, and free radical scavenging.

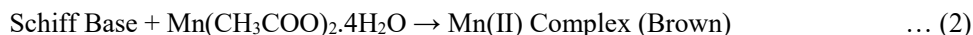
Research on Mn (II)-Schiff base complexes have revealed a two-stage thermal degradation process, where initial water loss in hydrated forms is followed by organic ligand decomposition at higher temperatures. Thermal analysis using TGA and DTA has been employed to determine kinetic parameters, including activation energy and reaction order, providing insights into decomposition mechanisms. Electron-donating substituents, such as methoxy groups on benzaldehyde, enhance ligand stability and thermal resilience, making these complexes suitable for thermally stable catalytic applications [18, 22, 23].

Additionally, the thiazotriazole ring, with its nitrogen and sulphur donor atoms, provides a unique coordination environment for Mn (II) complexes. The 5-amino-1,2,3,4-thiazotriazole ligand strengthens metal-ligand interactions through its electron-donating amino group. This Mn (II) complex exhibits potential catalytic properties and biological significance due to its moderate toxicity and pharmacological relevance. The study applies kinetic models such as the Freeman-Carroll and modified Doyle-Zsako methods to analyse reaction order and activation energy, further supporting the promising applications of Mn (II)-thiazotriazole complexes in coordination chemistry and medicinal fields.

MATERIALS AND METHODS

The detailed experimental procedure for the preparation of 5-amino-1,2,3,4-thiazotriazole ($\text{CH}_2\text{N}_4\text{S}$) and ortho-methoxy benzaldehyde ($\text{C}_8\text{H}_8\text{O}_2$) has been previously reported [14]. In a laboratory, the Mn (II) complex is synthesized by dissolving 10 g of 5-amino-1,2,3,4-thiazotriazole ($\text{CH}_2\text{N}_4\text{S}$) in 50 mL ethanol, followed by the addition of 8 g ortho-methoxy benzaldehyde ($\text{C}_8\text{H}_8\text{O}_2$) under reflux for 2 h to form the Schiff base ligand. Then, 6 g of Mn (II) acetate ($\text{Mn}(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O}$) is added, and the mixture is stirred for 3 h. The brown Mn (II) complex precipitates, which is filtered, washed with ethanol, and then dried.

Reaction Scheme



RESULTS AND DISCUSSION

This work investigates the thermal behaviour and degradation properties of a synthesized Mn (II) complex derived from a Schiff base reaction between 5-amino-1,2,3,4-thiazotriazole and ortho-methoxy benzaldehyde. Key kinetic parameters, including reaction order (n), activation energy (E_a), and entropy of activation (ΔS), were determined using the Freeman–Carroll method and Doyle's modified technique by Zsako [2]. Thermogravimetric analysis (TGA) was conducted by heating approximately 10–15 mg of the complex in an alumina crucible at a controlled rate of 10°C per minute under a nitrogen atmosphere. The percentage mass loss at each 10°C interval was recorded to assess the thermal decomposition stages. The study provides insights into the complex's stability and decomposition kinetics, which are crucial for understanding its potential applications.

The Freeman and Carroll method is a differential approach in thermogravimetric analysis (TGA) that enables the simultaneous determination of activation energy (E) and reaction order (n). This method relies on analysing the correlation between the rate of mass loss and the residual weight of the sample throughout the decomposition process. A plot is drawn to analyze the decomposition stage, where the y-axis represents $\frac{\Delta \log \frac{dw}{dt}}{\Delta \log w_r}$ and x-axis represents $\frac{\Delta T^{-1} \times 10^3}{\Delta \log w_r}$. Here, W_r is the remaining mass fraction defined as $W_r = W_c - W$, with W_c being the total mass loss at the completion of the reaction, and W being the total mass loss at time t . The TG curve was used to directly determine the residual mass (W_r) at uniform temperature intervals, and the collected data were analysed using the Freeman and Carroll method, as

summarized in Table 1. The graph of $\frac{\Delta \log d/dt}{\Delta \log(w_r)}$ versus $\frac{\Delta T^{-1} \times 10^3}{\Delta \log(w_r)}$ is obtain approximately straight line. The intercept of straight line is 0.43. The intercept suggests a reaction order of 0.43, and the activation energy is calculated as 11.93 kcal mol⁻¹ using the equation $E = 2.303R \times \text{slope}$.

Table 1. Data obtained by Freeman and Carroll Method

S. No.	Temp (°C)	Weight (mg)	$W_r = W_c - W$	$\frac{\Delta \log dw/dt}{\Delta \log(w_r)}$	$\frac{\Delta T^{-1} \times 10^3}{\Delta \log(w_r)}$
1	210	5.5621	2.61430	6.49114	1.60805
2	220	5.4768	2.52900	20.62907	2.91504
3	230	5.2417	2.29390	-10.39081	0.95166
4	240	4.9563	2.00850	-1.45923	0.67161
5	250	4.6834	1.73560	0.30668	0.58768
6	260	4.5971	1.64930	22.57305	1.61956
7	270	4.3748	1.42700	-6.53557	0.54953
8	280	4.2174	1.26960	2.95397	0.65611
9	290	3.9052	0.95740	-2.42653	0.26204
10	300	3.6126	0.66480	0.17777	0.19569
11	310	3.4985	0.55070	5.00133	0.36606
12	320	3.2812	0.33340	-1.28366	0.13272

Initial weight at 200°C = 5.7312 mg and Final weight at 330°C = 2.9478 mg.

The study utilized mass readings at different temperatures to analyse the thermal decomposition kinetics of the synthesized Mn (II) complex using Doyle's method, as modified by Zsako. The log F(α) values from table 2 were used to calculate activation energies (δ_0 , δ_1 , δ_2) and kinetic parameters (B0, B1, B2) for assumed reaction orders, summarized in table 3-5. The most accurate activation energy (E) was identified by selecting the standard deviation (δ) with the lowest value, which also indicated the best fit for the decomposition function F(α). By comparing $\delta_{\min}$ values across different kinetic equations, the function with the lowest $\delta_{\min}$ was determined to provide the highest consistency with experimental data. The analysis revealed that $\delta_{\min} = 0.10683$ corresponded to a reaction order of $b = 0$, activation energy $E = 12$ kcal/mol, and $(\overline{B_0}) = 6.61306$, highlighting the most reliable kinetic model for the decomposition process. By considering alternative kinetic equations and determining their corresponding $\delta_{\min}$ values, the function F(α) associated with the lowest $\delta_{\min}$ ensures the highest consistency with the experimental data. Table 6 presents a comparison of the $\delta_{\min}$ values for various assumed reaction orders. It indicates that the minimum value, $\delta_{\min} = 0.10683$, corresponds to a reaction order of $b = 0$, an activation energy (E) of 12 kcal mol⁻¹, and $(\overline{B_0}) = 6.61306$.

Table 2. Data of log F(α) values for the Fe-complex calculated at different temperatures

S. No.	Temp (°C)	$\alpha = \frac{W_o - W_t}{W_o - W_f}$	Log(α)	$\text{Log} \left(\ln \frac{1}{1-\alpha} \right)$	$\text{Log} \left(\frac{\alpha}{1-\alpha} \right)$
1	210	0.06075	-1.21643	-1.20289	-1.18921
2	220	0.09140	-1.03906	-1.01841	-0.99743
3	230	0.17586	-0.75482	-0.71350	-0.67082
4	240	0.27840	-0.55533	-0.48640	-0.41363
5	250	0.37645	-0.42430	-0.32576	-0.21917
6	260	0.40745	-0.38992	-0.28123	-0.16265
7	270	0.48732	-0.31219	-0.17516	-0.02204
8	280	0.54387	-0.26451	-0.10515	0.07640
9	290	0.65603	-0.18307	0.02825	0.28041
10	300	0.76116	-0.11853	0.15593	0.50336
11	310	0.80215	-0.09575	0.20958	0.60792
12	320	0.88022	-0.05541	0.32676	0.86620

Table 3. Calculation of $B_0 = \text{Log}(\alpha) - \text{Log P(x)}$ for different activation energies and values at different temperature

S. No.	Temp. (°C)	E=12 kcal	E=14 kcal	E=16 kcal
1	210	6.46657	7.50057	8.51357
2	220	6.51894	7.52994	8.52694
3	230	6.68318	7.67718	8.65618
4	240	6.76167	7.73867	8.70367
5	250	6.78170	7.74070	8.68870
6	260	6.70508	7.65208	8.58208
7	270	6.67681	7.60581	8.52181
8	280	6.62249	7.53849	8.43749
9	290	6.60693	7.50493	8.39093
10	300	6.57447	7.46447	8.33347
11	310	6.50525	7.37725	8.23725
12	320	6.45359	7.31659	8.16459
Average B_0		6.61306	7.55389	8.47972
Standard Deviation δ_0		0.10683	0.12850	0.16577

Table 4. Calculation of $B_1 = \text{Log} \left(\ln \frac{1}{1-\alpha} \right) - \text{Log P(x)}$ for different activation energies and δ_1 values at different temperature

S. No.	Temp. (°C)	E=18 kcal	E=20 kcal	E=22 kcal
1	210	9.52811	10.52411	11.50611
2	220	9.53059	10.50459	11.47459
3	230	9.66350	10.61950	11.56750
4	240	9.72160	10.66060	11.59060
5	250	9.71824	10.63924	11.55724
6	260	9.60377	10.51277	11.41177
7	270	9.55984	10.45584	11.33784
8	280	9.48485	10.36285	11.23185
9	290	9.47825	10.34225	11.19425
10	300	9.46893	10.31593	11.15993
11	310	9.39058	10.22458	11.05558
12	320	9.38176	10.20176	11.01976
Average B_1		9.54417	10.44700	11.34225
Standard Deviation δ_1		0.10945	0.15024	0.19608

Table 5. Calculation of $B_2 = \text{Log} \left(\frac{\alpha}{1-\alpha} \right) - \text{Log P(x)}$ for different activation energies and δ_2 values at different temperature

S. No.	Temp. (°C)	E=24 kcal	E=26 kcal	E=28 kcal
1	210	12.50179	13.47179	14.43679
2	220	12.45357	13.40757	14.35557
3	230	12.55118	13.48718	14.41818
4	240	12.58737	13.50537	14.42437
5	250	12.56983	13.47583	14.37183
6	260	12.42435	13.31235	14.19535
7	270	12.36596	13.24196	14.10796
8	280	12.27640	13.13340	13.98640
9	290	12.29641	13.14041	13.98041
10	300	12.34436	13.17036	13.99736
11	310	12.27792	13.09092	13.90492
12	320	12.37020	13.17020	13.97020
Average B_2		12.41828	13.30061	14.17911
Standard Deviation δ_2		0.10904	0.15366	0.20120

Table 6. Comparison of the values of δ and their activation energies for the assumed reaction order

b = 0		b = 1		b = 2	
E (Kcal mol ⁻¹)	δ_0	E (Kcal mol ⁻¹)	δ_1	E (Kcal mol ⁻¹)	δ_2
12	0.10683	18	0.10945	24	0.10904
14	0.12850	20	0.15024	26	0.15366
16	0.16577	22	0.19608	28	0.20120

The bold values show the minimum deviation for the activation energy 12 kcal.

Table 7. Orders of reaction (n) and activation energy (E) by both the adopted procedures

Methods	Order of reaction (n)	Activation Energy 'E' (Kcal mol⁻¹)
Freeman and Carroll	0.43	11.93
J. Zasko	0	12

Using the given values, the apparent frequency factor (Z) was calculated to be 113.22 S⁻¹ using Equation 3, while the apparent entropy of activation (ΔS) was found to be -25.33 e.u., using Equation 4 for the considered transformation stage.

$$\log Z = \bar{B} + \log Rq - \log E \quad \dots (3)$$

$$\Delta S = 2.303 \times \log \frac{Zh}{K_B T} \quad \dots (4)$$

where $\bar{B}$ represents the average of the most consistent series of B_i values, is the universal gas constant, q is the heating rate, E denotes the activation energy, h is Plank's constant, K_B refers to Boltzmann's constant. The absolute temperature at which half of the total mass loss occurs during pyrolysis is 543 K. The similarity in kinetic parameters obtained from two different evaluation methods suggests consistency in the decomposition analysis, as presented in table 7.

APPLICATION

The Mn (II) complex derived from the Schiff base of 5-amino-1,2,3,4-thiatriazole with ortho-methoxy benzaldehyde exhibits a wide range of applications due to its distinctive structural and functional attributes. It acts as an efficient catalyst in various organic transformations, including oxidation and polymerization reactions, benefiting from the stabilizing influence of the Schiff base ligand. Additionally, its notable antimicrobial and antioxidant properties make it a promising candidate for pharmaceutical applications. Beyond these roles, the complex holds significant potential in material science, contributing to the synthesis of advanced materials such as, electrochemical sensors, batteries, and fuel cells, demonstrating its versatility across various technological fields. Similarly, Mn (II) complexes also exhibit broad applicability due to their coordination flexibility and electronic properties. These complexes are widely utilized in bioinorganic chemistry for enzyme mimetics, particularly in catalytic oxidation reactions. Their role in MRI contrast agents and therapeutic applications has gained attention due to their paramagnetic nature and biocompatibility. Furthermore, Mn (II) complexes contribute significantly to photocatalysis, supercapacitors, and luminescent materials, reinforcing their importance in both industrial and biomedical advancements.

CONCLUSION

The study revealed that mass measurements taken at various temperatures, analysed using Doyle's method as modified by Zsako, facilitated the determination of kinetic parameters for the presumed reaction order. The most precise activation energy (E) was identified based on the smallest standard deviation (δ), offering

insights into how well the decomposition process aligned with the assumed kinetic function $F(\alpha)$. Findings showed that a reaction order of $b = 0.43$, an activation energy of 11.93 kcal/mol, and an average $(\overline{B_0}) = 6.61306$ resulted in the minimum $\delta_{\min} = 0.10683$. By comparing different kinetic models, the research emphasized the effectiveness of statistical methods in accurately matching experimental results with theoretical predictions. The apparent entropy of activation (ΔS) was found to be -25.33 e.u.. The results highlight the effectiveness of Doyle's modified method and its consistency with Freeman and Carroll's approach, ensuring a reliable determination of kinetic parameters and confirming the relevance of the proposed kinetic models.

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