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Synthesis Characterization of Photoactive Complex and Study its Photochemical Reaction

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ABSTRACT

The mixed ligand complex of potassium tris(oxalate)ferrate(III)trishydrates have been synthesized & characterized the resulting complex were characterized by Gravimetric analysis, volumetric analysis & spectrophometrically. (uv visible spectro) studied. Also study its photochemical reaction .from the analytical &spectral data the result is conclude that %purity of the complex is of 90-95% exept for the preparation in terms of oxalate ion &Its found that complex shows the colour change on irradiation therefore is Photoactive.

Keywords: Photoactivity, Gravimetric, Volumetric, Spectrophotimetric

I. INTRODUCTION

with multi functional properties due to their potential application in molecular magnet chiral materials super conductors , ferromagnetic metals & photophysics.in the area of photochemistry a photochemical reaction are valuable in organic & inorganic chemistry because they proceed differently than thermal reaction.these reaction are serious nuisance ex.degradation of PVC.

II. EXPERIMENTALS

Materials & Methods:- Under the world of green chemistry we synthesis of characterize the photoactive complex & studied its photochemical reaction all the chemicals & solvent used were of A.R. grade. the spectra were recorded on a spectrophotometer in the frequency range 700nm ferroferri-cynide complex against the non irradiated ferrioxalate solution .and the graph obtain by spectrophotometer rate constant were recorded.

Synthesis of ligand complex:

1) By Single stage method:

Prepartion of potassium trisoxalatoferrate (III) trihydrate

$$K_3[fe\ (C_2O_4)_3].H_2O$$

Weighed quantity of FeCl3 and potassium oxalate mixed. After adding methanol cooling done on ice bath. Crystal of $K_3[Fe(C_2O_4)_3]3\ H_2O$ will appear in solution dry the crystals under vaccum.

From these therotically & practically %Yield is calculated.

2) By Two Stage Method-

1) Preparation of ferrous Oxalate $[Fe(C_2O_4),2\ H_2O]$-

From FAS:

Dissolve Weighed Quantity of FAS in the acidified warm water .then freshly prepaired Oxalic acid solution is Added To the solution Of FAS .Heat The above solution Cautiously to avoid the bumping. Withdraw the heat. And allow the granuler yellow precipitate of iron (II) oxal ate dehydrate to settle. Decant the supernatant liquid. Wash the yellow solid with hot distilled water,and with a few ml of acetone.

2) Preparation of Potassium Trioxalatoferrate (III) trihydrate:

$K3[Fe\ (C_2O_4)_3].3H_2O$ From Ferrous Oxalate-

Weighed quantity of dried iron (II) Oxalate dehydrate prepared in first step .is dissolve in little quantity of distilled water . also prepare the solution of potassium oxalate hydrated $[K_2C_2O_4H_2O]$ in 30 ml distilled water and pour this solution into the above solution . resulting the formation of orange intermediate iron (II) Copmplex. Warm the solution.to it. Add 30% 10 ml H_2O_2 dropwise .at this stage brown precipitate of $Fe(OH)_3$ if

formed. Heat the mixture to boiling to it add 1M Oxalic acid solution .lime green solution should appear. This result formation of tris oxalate ferrate (III) ion $[Fe(C_2O_4)_3^{3-}]$. Cool the above solution add 95% ethanol . cool in ice bath .the product potassium trisoxalatoferrate (III) is Form . Which is Photosensitive. From this practical yield ,theoretical yield ,% Practical yield is calculated.

Characterization of a$K_3[Fe(C_2O_4)_3].3H_2O$:

1) % H2O (moisture) in $K_3[Fe(C_2O_4)_3].3H_2O$can be calculated by gravimetrically.(% Purity)

2) % Of Oxalate ion in $K_3[Fe(C_2O_4)_3].3H_2O$ can be calculated by volumetrically.(% Purity)

3) % Of iron in the $K_3[Fe(C_2O_4)_3].3H_2O$ can becalculated by Spectrophotometrically.

Gravimetric study:

Sr No.	Description	Two Stage Preparation method	Single Stage Preparation method
1	Wt. of the empty silica crucible+lid(w_1)	26.315gm	26.113gm
2	Silica crucible +lid+complex(w_2)	26.856gm	26.613gm
3	Weight of sample taken (W_2-W_1)	0.541gm	0.50gm
4	After heating at $110^0{}_c$ For 45 min	26.725gm	26.585gm
5	(after heating at 100^0c for 60 minW3) & onward	26.797gm	26.558gm
6	Weight of residue (W_2-W_3)	0.059gm	0.0545gm
7	% of moisture	10.9%	10.90%

Volumetric study:

Sr. No.	Description	Result for 2 stage	Direct method
1	Exact normality of KM nO_4	0.055N	0.05N
2	Theoretical %of $C_2O_4^{2-}$ ions	53.76%	53.76%
3	Practical % of $C_2O_4^{2-}$ ions	25.085%	53.33%
4	% purity of complx in terms of oxalate ion	46.85%	99.20%

Spectrophotometric study:

Sr. No	Description	Result forTwo Stage method	Result for single Stage Preparation method
1	Theoretical % Of Fe(III) ions	11.404%	11.404%
2	Practical % of Fe (III) ion sample –I	10%	9.5%
3	% Purity of complex in terms of Fe sample I	87.71%	83.33%

For this preparation of standered Fe(III) solution is done which is used as blank solution and by using blank solution obtained the absorbance at 530 nm then from graph determine the amount of Fe(III) solution Plotting of calibration curve for single stage & double stage method was carried out From this concentration of Fe in Sample is obtained.

Study the Photochemical reaction:

1) By Prepairing a stock solution of ferrioxalate by dissolving 0.06 gm of Fe $NO_3.9H_2O$ in 100 ml distilled water and add 0.04 gm of oxalic asid with A.R. grade is added in above soltion .and dilute it by 100ml distilled water. Store it in adark by wrapping black carbon paper around it.

2) In 8 large test tube take 10ml of above solution .kept one of the test tube in dark which is used as blank solution

3) The solution in other test is exposed to sunlight for the period of 18 min. at the interval of 3 min

4) Add 1ml of 0.05 M $K_3[Fe(CN)_6]$ solution to each of the irradiated solution. On irradiation The reference solution remains pale yellow while the irradiated solution developed th blue colour.

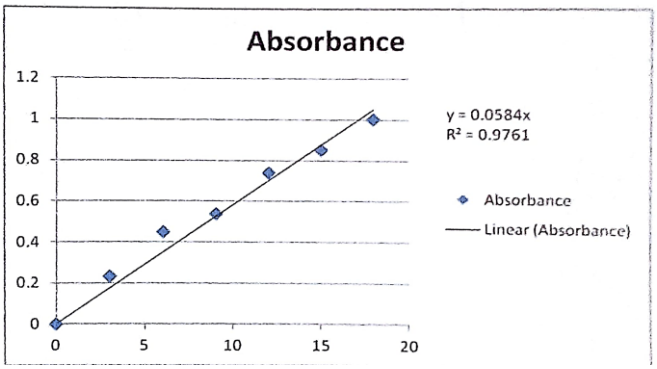
5) above solution is characterizes by spectrophotometer and calculate the rate constant from the first Order reaction kinetics. From the graph of log of absorbance v/s time in minute . rate constant can be calculated as

K= 2.303*slope

Observation Table 1. Irradiation of complex solution (with time Intervals 3 Minute)

Sr no	Time in minute	absorbance	Rate constant
1	0	0	0.133574
2	3	0.233	
3	6	0.449	
4	9	0.537	
5	12	0.737	
6	15	0.852	
7	18	1	

A graph of Absorbance Vs Time in min.



III. RESULT

Fe (II) ions formed by above method is proportional to no of photons absorbed .this in turns is proportional to the time of exposure.

IV. DISCUSSION

1) Photoactive Complex i.e.Potassium trisoxalatoferrate(III) trihydrate $K_3[Fe(C_2O_4)_3].3H_2O$ can be synthesized by both methods Viz. Single stage as well as double stage method.
2) By both method s green crystals of Pottasium trisoxalatoferrate(III)trihydrate complex are obtained out % practical yield by two stage method is more than single stage method
3) though the % practical yield by single stage method is less but % Purity of complex is about 99%
4) As irradiation time interval increases rate constant also increases .
5) Synthesised potassium trisoxalatoferrate(III) trihydrate complex is photoactive.

V. REFERENCES

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