

SYNTHESIS AND CHARACTERIZATION OF HEXAMINE COBALT (III) CHLORIDE USING PLANT-SOURCE ACTIVATED CARBON AS A CATALYST

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ABSTRACT

The activated carbon plays a vital role as an adsorbent, reducing agent, and catalyst in all branches of chemistry. The present study was focused on the preparation of *sweet corn cop*, *tamarind bark*, *ficus racemosa*, and *macaranga peltata* activated carbon by the H₂SO₄ leaching method activation through a muffle furnace at 600-800°C. Percentages of yield of hexamine cobalt (III) chloride using these activated carbons as catalyst were recorded as 60.8, 52.2, 43.4, and 53.0, as compared with commercial charcoal, which has 54.0 percent. The yields were directly related to the nature of the activated carbon and its efficiency. Sweet corn cop activated carbon has a higher yield compared to *ficusracemosa*. The electronic transition of the hexamine cobalt (III) chloride shows peaks at 345 nm and 481nm and is assigned as ¹A_{1g} → ¹T_{1g} and ¹A_{1g} → ¹T_{2g}. The major infrared peaks at 3420cm⁻¹ and 1620-1630cm⁻¹ is the indication of the presence of water and Vibartional band at 630cm⁻¹ is the indication of the presence of metal – oxygen (M-O) interaction in CoCl₂. 6H₂O. The peaks at 3156 cm⁻¹ and 831cm⁻¹ are attributed to the stretching vibrations of ammonia molecules, whereas 1589cm⁻¹ and 1329cm⁻¹ cm⁻¹ correspond to asymmetric deformation of NH and symmetric deformation of NH₃. The band around the region 367cm⁻¹ indicates the formation of the Co-N bond in the complex. Thus, the yield of the complex is directly related to the nature of the plant source activated carbon as a catalyst.

Keywords: Hexamine Cobalt (III) chloride, Leaching, Activated Carbon, Catalyst, Electronic Transition, IR-band, Efficiency.

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INTRODUCTION

Activated charcoal from plant sources finds universal importance in many scientific fields, including nanotechnology. Activated carbon (AC) is mainly used in adsorption chemistry as one of the best adsorbents for organic and inorganic pollutants in industrial wastes. It finds wide applications as a catalyst or reducing agent in many organic syntheses and pharmaceutical industries. Activated carbons are considered versatile catalysts utilized in heterogeneous catalysis. Carbon-supported catalysts are the main routes for new chemical catalytic processes based on their intrinsic features.¹ Fireweed (*Chamerion angustifolium*) carbons are used to adsorb metal ions, and they have shown considerable advances compared to commercially available activated carbons. The activated carbon from Fireweed leaves shows relatively comparable efficiency in the adsorption of cobalt, Co (II), and nickel, Ni(II) ions.² Many dyes and heavy metals are effectively removed from aqueous solution using carbon from coal ash obtained from zeolites, which also finds several environmental applications.³ Cadmium and lead ions are effectively removed from aqueous solutions using commercially available carbonised carbon, where the adsorbent had a coarse surface with crevices and a high surface area.⁴ Many organic pollutants, along with heavy metal ions, were absorbed by Black carbon (BC). The adsorption of polar compound, 4,4-dichlorophenol, and non-polar compounds 1,2 1,2-dichlorobenzene and benzene, shows a decreased trend in the presence of Cu²⁺ as it forms surface complexation. But, adsorption of organic solutes increases in the presence of Ag⁺ ions because of the decline in hydrophilicity of the region around Ag⁺ complexed

functionalities due to the softness of the cation.⁵ Oak activated carbon was modified to remove Pb^{2+} ions through a modified route using glutaraldehyde as the active complexing ligand.⁶ There are reports about the synthesis of beads of polymeric gel inorganic salts (magnetic) with activated carbon, which shows versatile and strong adsorption for magnetic substances.⁷ The coexistence of SO_4^{2-} and PO_4^{3-} gives a noticeable effect on the adsorption of Se(II) whereas Cl^- and NO_3^- show an insignificant effect. An⁸ Maximized catalytic system was prepared by mixing metal acetates with Norit charcoal and Sn^{2+} , K^+ , and activated carbon, which enhanced the decomposition of lead acetate to metallic palladium, where Sn(II) lowers the activation energy.⁹ The photochemical decomposition of methylene blue was achieved by developing chloroindium-based porphyrin and oxygen-functionalized norit granule.¹⁰

A composite bead of iron-modified chitosan/coconut shell activated carbon (Fe/CSCC) composite shows good efficiency in scavenging Cr (VI).¹¹ Multi-doping and C defects micro-sized coffee charcoal are functionalized for hydroxyl and carboxyl functional groups, showing effective catalytic activity and recyclability.¹² Many organic ligand-grafted polymers with activated carbon contribute the best efficiency for divalent metal ions.¹³ Magnetic composites were also produced with fish scale activated carbon and nickel-iron bedded bi-oxide.¹⁴ The present work was concentrated on the preparation of plant-sourced activated carbon (PAC), such as sweet corn cop, tamarind bark, *Ficus racemosa*, and *Macaranga peltata*, by the H_2SO_4 leaching method. These PAC was used as synthetic catalysts in the preparation of hexamine cobalt (III) chloride by reporting yield and characterising the complex for purity.

EXPERIMENTAL

Materials and Methods

Glass distilled water, $CoCl_2 \cdot 6H_2O$, Liq NH_3 , NH_4Cl , HCl , H_2O_2 , Commercial Activated charcoal, H_2SO_4 . All chemicals are analytical and lab grade and purchased from local chemical suppliers Sweet corn cop, Tamarind bark, *Ficus racemosa*, and *Macaranga peltata* samples were collected from the local area, Mysore city, and shadow dried by making them into small slices. Then the sample materials were preserved to get activated carbon.

Preparation of Activated Carbon

Collected samples were uniformly wetted by adding concentrated H_2SO_4 in a 500ml beaker and leached for 48 hours with occasional stirring. The charred mass was collected carefully and washed using distilled water till the pH value of the washing is 7. The collected charred mass was dried at $1100^\circ C$ for one hour, cooled to room temperature, and ground into a fine powder. Then the charred powder was maintained in a muffle furnace for five hours by varying the furnace temperature from $400-800^\circ C$ in the interval $20^\circ C$ per minute. The charred mass was removed from the furnace, cooled to room temperature, and ground into fine powder to the micro and nano levels. The yield was determined, and the resulting activated carbon was preserved in an air-tight bottle for further experimentation.

Preparation of Hexamine Cobalt (III) Chloride Complex

Synthetic Route 1

1.5 g ammonium chloride, 2 g cobalt chloride were mixed in 3 ml of boiling water to dissolve, and then 0.5 g of plant source activated carbon was taken in a 250ml beaker. The mixture was stirred well using a glass rod, cooled in an ice bath, and 4 mL of aqueous ammonium solution was added. The solution was maintained at $10^\circ C$ or lower. 4ml of hydrogen peroxide was added slowly in small portions and mixed well. The temperature of the reaction mixture increased gradually to 50 to $60^\circ C$, and the beaker was kept at this temperature with occasional shaking until the last trace of pink coloration was removed.

The resulting solution was cooled and filtered. The crystals formed were transferred into a beaker along with the activated carbon and dissolved in 25 ml of boiling water containing 3 ml of con.HCl. When all crystals, except the carbon, dissolved, the solution was filtered while hot. 5 ml of concentrated HCl was added to the filtrate and cooled in an ice bath. Golden brown crystals appeared which were filtered, washed with acetone, and dried. The complex was recrystallized from water containing a trace of hydrochloric acid. The crystals were dried in a desiccator and weighed.

Synthetic Route 2

1.5g ammonium chloride, 2 g cobalt chloride were dissolved in 3 ml of boiling water, and 0.5g of plant source activated carbon was taken in a 250ml beaker. The mixture was stirred well using a glass rod, cooled in an ice bath, and 4 mL of aqueous ammonium solution was added. The solution was maintained at 10°C or lower. 4ml of hydrogen peroxide was added slowly in small portions and mixed well. The temperature of the reaction mixture increased gradually to 50 to 60°C, and the beaker was kept at this temperature with occasional shaking until the last trace of pink coloration was removed. The resulting solution was cooled, and 25 mL of boiling water containing 5 mL of con.HCl is heated to boiling. The hot solution was filtered, the filtrate cooled in an ice bath to form orange coloured crystals, and then filtered. The complex was recrystallized from water containing a trace of hydrochloric acid. The crystals were dried in a desiccator and weighed.

Detection Method

Electronic Spectra

Shimadzu UV-1800UV/Visible scanning Spectrophotometer; 115VAC.

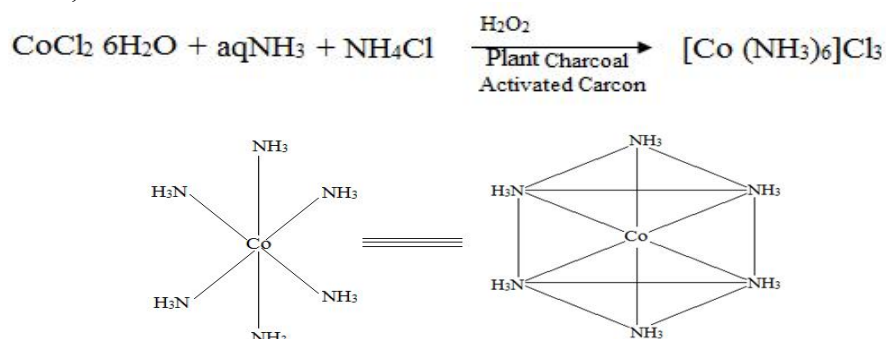
FTIR Spectra

FTIR spectrum was recorded using a Nicolet- 5700 FTIR spectrophotometer. The complex was taken with a KBr pellet under the specification Thermospectra-Tech, KBr disc 32x3mm, drilled 7000-467.

RESULTS AND DISCUSSIONS

Synthesis of Hexamine Cobalt (III) Complex

The complex was prepared in the presence of H₂O₂ and catalyzed by plant-source activated carbon. Sweet corn cop and tamarind bark activated carbons were found to be effective catalysts with commercial charcoal. The yields of plant source activated carbons show relatively comparable yields to those of commercial charcoal, as recorded in Table-1.



The complex has been prepared by using both routes in triplicate in the presence of plant source activated carbon. The catalytic activity of the activated carbon is directly related to the percentage of carbonization, which in turn influences the amount of yield of the complex. AC of sweet corn-cop has noticeable catalytic activity, forming relatively more yield than Ficus racemosa. Comparatively, plant charcoal-activated carbon recorded a conspicuous yield with reference to commercially available charcoal for synthesis. The percentage of yield of hexamine cobalt (III) complex in the presence of ACs of sweet corn-cop, Tamarind bark, Ficus racemosa, and Macaranga peltata is relatively comparable to that of commercial charcoal.

Table-1: % of Yield of Hexamine Cobalt (III) Complex with Reference to the Catalytic Activity of Plant Source Activated Carbon

Wood charcoal activated carbon	Amount of activated carbon(gm)	Complexes	Yield	% of yield
Sweet corn cop	0.5		0.681	60.8

Tamarind bark	0.5		0.585	52.2
Ficus racemosa	0.5		0.486	43.4
Macaranga peltata	0.5		0.594	53.0
Commercial-grade charcoal	0.5		0.605	54.0

Spectroscopic Analysis

UV-Vis Spectra of Hexamine Cobalt Chloride Complex

The UV-Vis spectra of the complex were recorded as shown in Fig.-1, which consists of two peaks at 345 nm and 481 nm. These two peaks correspond to the d-d transition states of the metal ion in the octahedral field for the d^6 system. The assigned transition for d^6 was $^1A_{1g} \rightarrow ^1T_{1g}$, having a frequency of transition at 345 nm and $^1A_{1g} \rightarrow ^1T_{2g}$ at 481 nm as per the Tanabe-Sugano diagram.

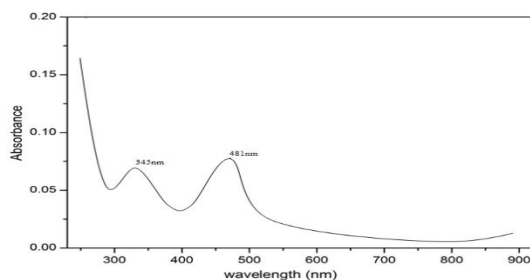


Fig.-1: UV-Visible Spectrum of $[Co(NH_3)_6Cl_3]$

IR Spectral Analysis of $CoCl_2 \cdot 6H_2O$

FTIR spectrum for $CoCl_2 \cdot 6H_2O$ was recorded using an analytical grade commercial sample as shown in Figure Fig.-2. The characteristic spectrum consists mainly of three vibrational peaks at 3416 cm^{-1} , 1630 cm^{-1} , and 632 cm^{-1} due to stretching and deforming of the O-H bond. These vibrational peaks indicate the presence of water molecules in $CoCl_2$.¹⁵ Experimental data was well comparable with the data reported by researchers.¹⁶ As per the literature survey, the maximum absorption peak in the region of 3420 cm^{-1} and another peak at $1620\text{--}1630\text{ cm}^{-1}$ is an indication of the presence of water. Vibrational band at 630 cm^{-1} is an indication of the presence of metal-oxygen (M-O) interaction¹⁵, which was highly comparable with the experimental value 632 cm^{-1} . These data were further confirmed by Mitic Z *et al.*¹⁷ These observations clarify the purity of the complex formed.

IR Spectral Analysis of Hexamine Cobalt (III) Complex $[Co(NH_3)_6Cl_3]$

The FTIR spectra were recorded as shown in Fig.-3 with respect to different plant source activated carbon. The complex formed by various activated carbon-catalysed reactions was analysed for the formation of hexamincobalt (III) chloride. The spectrum of the complex from each route was recorded, and the main bands that appeared are shown in the table. The band at 3259 cm^{-1} is the indication of the asymmetric stretching frequency of NH_3 . The peaks at 3156 cm^{-1} and 831 cm^{-1} are attributed to the stretching vibrations of NH_3 molecules, whereas 1589 cm^{-1} and 1329 cm^{-1} correspond to asymmetric deformation of NH and symmetric deformation of NH_3 .^{15,18} Many researchers reported similar data in the same regions of the spectrum¹⁹ while reporting for different d-orbitals for ammine complexes. In addition, the Co-N bond is confirmed by the stretching frequency at 367 cm^{-1} .^{15,18}

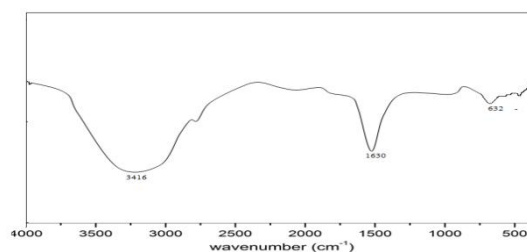
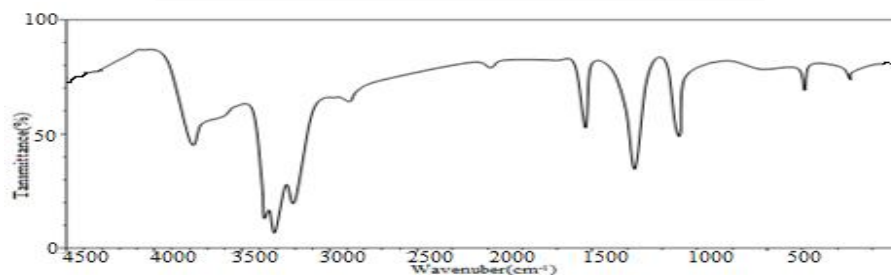


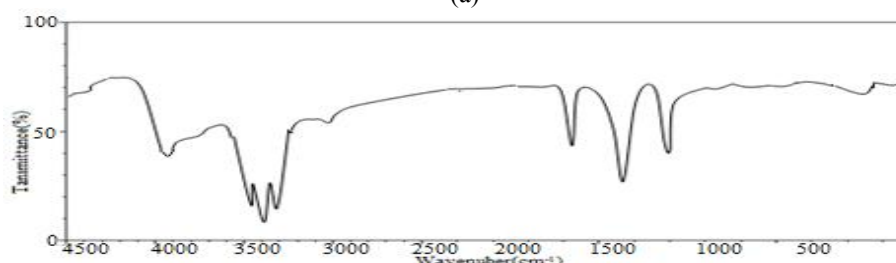
Fig.-2: FTIR Spectrum of $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$

Table-2: IR Frequencies Against Mode of Vibration of Bonds in $[\text{Co}(\text{NH}_3)_6\text{Cl}_3]$

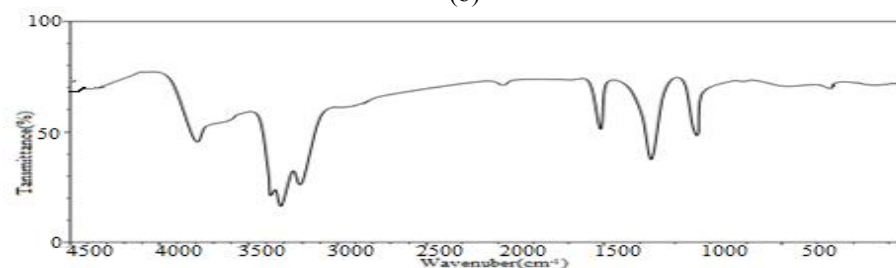
IR Frequencies	Assignment
3259	$\nu_{\text{as}}\text{NH}_3$
3156	$\nu_{\text{s}}\text{NH}_3$
1589	$\nu_{\text{asd}}\text{NH}_3$
1329	$\nu_{\text{sd}}\text{NH}_3$
831	$\nu_{\text{s}}\text{NH}_3$
367	$\nu_{\text{s}}\text{Co-N}$



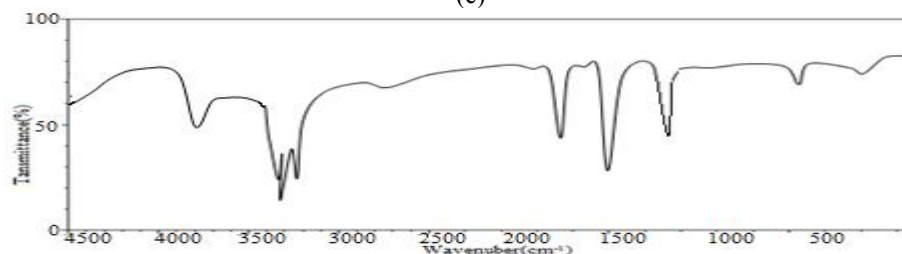
(a)



(b)



(c)



(d)

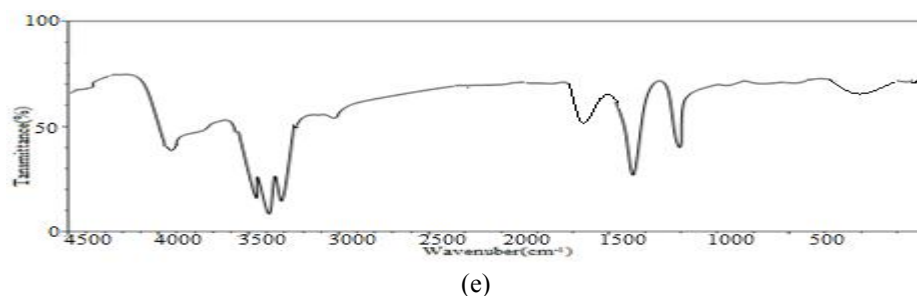


Fig.-3: FTIR Spectrum of $[\text{Co}(\text{NH}_3)_6\text{Cl}_3]$ Synthetic Catalyzed by (a) Sweet Corn Cop, (b) Tamarind Bark, (c) Ficus racemosa, (d) Macaranga Peltata, (e) Commercially Activated Charcoal

CONCLUSION

Activated carbon (AC) from plant sources plays a vital role in the basic chemistry field, which can be utilized as a catalyst, adsorbent, reducing agent, etc. The present experiment was focused on the catalytic synthesis of hexamine cobalt (III) chloride using AC of corn cop, Tamarind bark, Ficus racemosa, and Macaranga peltata. The catalytic activity of the activated carbon is directly related to the percentage of carbonization, which in turn influences the amount of yield of the complex. AC of sweet corn-cop has noticeable catalytic activity, forming relatively more yield. Comparatively, plant charcoal activated carbon recorded a conspicuous yield with reference to commercially available charcoal for the synthesis of hexamine Cobalt (III)chloride. The UV-Vis spectrum of the complex consists of two peaks at 345 nm and 481nm. These two speak correspond to d-d transition states of the metal ion in the octahedral field for the d^6 system. The peaks at 345nm and 481 nm clearly indicate the electronic transition spectra of the cobalt amino complex due to the transitions $^1\text{A}_{1g} \rightarrow ^1\text{T}_{1g}$ and $^1\text{A}_{1g} \rightarrow ^1\text{T}_{2g}$.

FTIR spectrum for $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ was recorded using analytical grade commercial sample and the characteristic spectrum consists of mainly three vibrational peaks at 3416cm^{-1} , 1630cm^{-1} and 632cm^{-1} corresponds to stretching and deforming of O-H bonds which confirm the presence of water molecules in $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$. Vibrational band at 630cm^{-1} is indication of the presence of metal – oxygen (M-O) interaction which was highly comparable with experimental value 632cm^{-1} . The FTIR spectra were recorded for hexamine Cobalt (III)chloride complex with respect to different plant source activated carbon, comprising bands at 3259cm^{-1} indicating asymmetric stretching frequency of NH_3 , 3156cm^{-1} and 831cm^{-1} attributed to the symmetric stretching vibrations of NH_3 molecules, whereas 1589cm^{-1} and 1329cm^{-1} are corresponding to asymmetric deformation of NH and symmetric deformation of NH_3 ^{15,18}. Many researchers reported similar data in the same regions of the spectrum¹⁹ while analyzing d-orbitals for metal amino complexes. Further, it is confirmed that the stretching frequency at 367cm^{-1} is due to the Co-N bond.^{15,18} Thus, plant source activated carbons are a general catalyst for the synthesis of hexamine cobalt (III) chloride complexes with sustainable yield.

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CONFLICT OF INTERESTS

The authors declared no conflict of interest.

AUTHOR CONTRIBUTIONS

The present work is related toSDG-9:Industry, Innovation and Infrastructure.All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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