

Synthesis and Characterization of Chromium Oxide Nanoparticles by Chemical Route and its Antibacterial Activity against E.Coli Bacteria

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Abstract - Chromium oxide nanomaterials are well known for its sensing and antibacterial property. In this paper the precipitation method involves precipitation of the precursor salt solution with precipitants. This method has a good control of factors such as chemical composition, homogeneity, crystalline, particle size and morphology. In a chemical route method different precursor such chromium sulphate and chromium chloride were used with liquor ammonia as precipitating agent. The prepared samples were characterized by using XRD and UV-Vis Spectrophotometer to analyze its structural and optical properties. The material was confirmed to be chromium oxide using XRD analysis having hexagonal structure. Almost the single phase of all the nanomaterial has been achieved with fewer impurity peak. Thus chromium oxide nanoparticle prepared by chemical route has been used for studying the antibacterial effect on E. Coli bacteria. It has been found that the zone of incubation has been developed which confirms the effect of chromium oxide for degrading the activity of E. Coli bacteria. Thus chromium oxide nanoparticles have been prepared by precipitation method using different reducing agent and its properties has been analyzed and discussed.

Key Words: Chromium Oxide Nanoparticles, Precipitation, Hexagonal Structure, XRD, UV-Vis Spectrophotometer, E. Coli

1. INTRODUCTION

Chromium oxide nanoparticles are of much interest because of its multiple properties such as semiconducting [1], optical, catalytically [1] and its antibacterial phenomenon[2]. Chromium oxide present in various oxidation states, hence it has a large range of complexions so it is recognized as transition metal oxide [3]. Because of its small size and high density at the corners surface sites these transition metal oxides vary in its physical and chemical properties [2,3]. These materials can either be synthesized by chemical or biological approaches. Both the approaches have a lot of factors such as the solution properties, solvent viscosity, dielectric constant, the solubility of metal oxide and the metal oxide surface energy [4].

In this research paper the main focus is on the factors like pH and temperature, which affects the formation of chromium oxide nanoparticles [5]. Nanosized chromium oxide can be synthesized using different methods such as precipitation, co-precipitation, sol-gel, hydrothermal, gel citrate, mechanochemical process, urea-assisted homogenous precipitation, gas condensation, microwave plasma, sonochemical etc. [1,2,5]. In this present work chemical precipitation method have been selected which involves the reduction of salt with base. Transition metal oxides have evolved to play a prominent role in antibacterial activity. C. Ramesh et. al discussed the effect of chromium oxide nanoparticle against E.coli bacteria which reveals it be an effective bactericide [6].

The present work is based on preparation of chromium oxide nanoparticle using two different precursors (chromium sulphate and chromium chloride) for antibacterial activity. Here environmental conditions such as pH, temperature of the starting material are varied thus, giving a better result compared to previous works done. The prepared sample was further characterized by XRD, UV-vis spectroscopy [1,6] to find its average crystalline size, shape and its wavelength. Then the material was used to show the antibacterial effect on E. Coli bacteria [2,6,7]. The disc diffusion method [7] has been used for growing bacteria in nutrient agar solution. The zone of incubation has been measured thus, justifying the antibacterial activity of chromium oxide nanoparticles.

2. EXPERIMENTAL PROCEDURE

Chromium (III) Chloride (hexahydrate), Chromium Sulphate, Ammonium Hydroxide (liquor ammonia), Distilled water were used throughout the experiment

2.1 Chemical Method (By using chromium sulphate as Precursor)

0.1M of chromium sulphate was mixed with 100ml of distilled water and was kept in a magnetic stirrer for 30 minutes to get a homogenous solution. Liquor ammonia was added drop wise with a constant stirring until the pH of the solution reaches 10 at the room temperature. The as the prepared sample was washed several times (3-5) using distilled water with the help of centrifuge machine. The obtained precipitates were then dried in hot air oven at 70°C for 24 hours and were calcinated at 650°C in a muffle furnace about 5-6 hours. Prepared sample was mashed by motar&pestal.

2.2 Chemical Method (By using chromium chloride as a Precursor)

0.2M of chromium chloride was mixed with 100ml of distilled water and was kept in a magnetic stirrer for 30 minutes to get a homogenous solution. Liquor ammonia was added drop wise with a constant stirring until the pH of the solution reaches 12 at the temperature 50°C. The as the prepared sample was washed several times (3-5) using distilled water with the help of centrifuge machine. The obtained precipitates were then dried in hot air oven at 70°C for 24 hours and were calcinated at 550°C in a muffle furnace about 5-6 hours. Prepared sample was mashed by motar&pestal.

2.3 Disc Diffusion Method

Nutrient agar plates were being prepared. They act as a colony for the growth of bacteria. The antibacterial activity was performed on E. coli, which is present in sewage water. The prepared agar plates are kept in incubator at room temperature for 24 hours. After that 6mm circular filter paper was encapsulated with two prepared sample. These discs were kept on the agar surface and incubated at 37°C for 24 hours. The zone of inhibition was measured which showed some antibacterial activity.

2.4 Material Characterization

The prepared material was characterized by Bruker D8 Advance XRD using Cu-K α (0.154A $^\circ$) radiation and UV-Visible spectrophotometer using Perkin Elmer model lambda 950. The average crystalline size of prepared sample was calculated by Scherrer formula. The band gap of the sample can be calculated from absorption peak.

3. RESULT AND DISCUSSION

3.1 XRD Analysis

The structural characterization of chromium oxide nanoparticles was carried out by X-ray Diffraction. The 2 θ and intensity data were analyzed and graphs were plotted as shown in **Chart-1**. The X-ray diffraction pattern, crystalline phases were identified by comparison with reference data from the JCPDS file no 850869 for chromium oxide nanoparticles. All Bragg reflections have been indexed which confirms the formation of a well-defined single phase hexagonal structure with some impurity peaks.

The hkl parameters for obtaining 2 θ values are framed in table 1 and the Average crystalline size was obtained from chromium sulphate was about 27 nm.

Table-1: Crystalline size and lattice parameter of the prepared chromium oxide nanoparticles by Chromium Sulphate

2 θ	MILLER INDICES	CRYSTALLINE SIZE
24.51 $^\circ$	012	24nm
36.17 $^\circ$	110	26nm
50.18 $^\circ$	024	22nm
63.48 $^\circ$	214	27nm

Similarly the average crystalline size was obtained from chromium chloride was about 24 nm and the hkl parameters to obtained 2θ values are framed in table 2.

Table-2: Crystalline size and lattice parameter of the prepared chromium oxide nanoparticles Chromium Chloride

2θ	MILLER INDICES	CRYSTALLINE SIZE
24.49°	012	27nm
36.28°	110	26nm
50.27°	024	18nm
63.66°	214	39nm

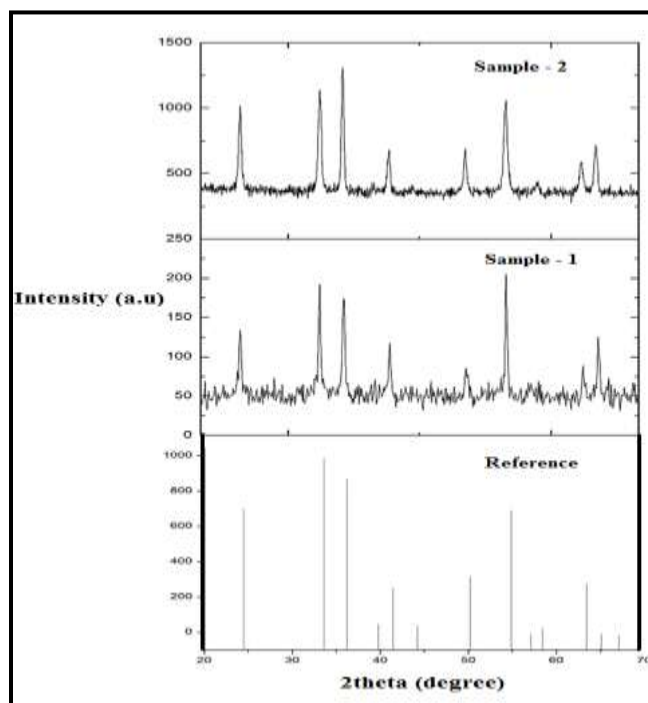


Chart-1 : X-ray diffraction pattern of synthesized chromium oxide nanoparticle using several precursors by chemical method ; sample1- by chromium sulphate; sample2-chromium chlorides; reference

3.2 UV-Visible spectrophotometer

Analysis was carried out using Perkin Elmer model lambda 950 from UGC-DAVV, Indore. The wavelength of above UV-Visible spectrophotometer ranges from 200 to 900nm. The chart 2 shows that the absorbance peak for the particular samples and has been compared in table 3. From the table, we can identify that the s1 and s2 having a band gap approximately equal to 4.8 eV. And other two samples were having a band gap near about 3.7eV which is near to the bulk chromium oxide (3.4eV). From the XRD results it has been concluded that the s3 and s4 are in intermediate states, hence they are having a low bandgap. The bandgap of samples can be calculated by-

$$E_g = \frac{hc}{\lambda}$$

Where h is plank's constant ($6.64 \times 10^{-34} \text{J/sec}$), c is the velocity of light in vacuum or air ($3 \times 10^8 \text{sec}$) and λ is wavelength where the highest absorbance peak.

Table-3: Bandgap analysis of different chromium oxide nanoparticles

Sample with specifications	Wavelength (nm)	Energy Bandgap
Sample-1 (Using Chromium Sulphate)	262	4.7
Sample-2 (Using Chromium Chloride)	256	4.8
Sample-3 (Using Pumpkin Leaves)	337	3.6
Sample-4 (Using Mellberry Leaves)	331	3.4

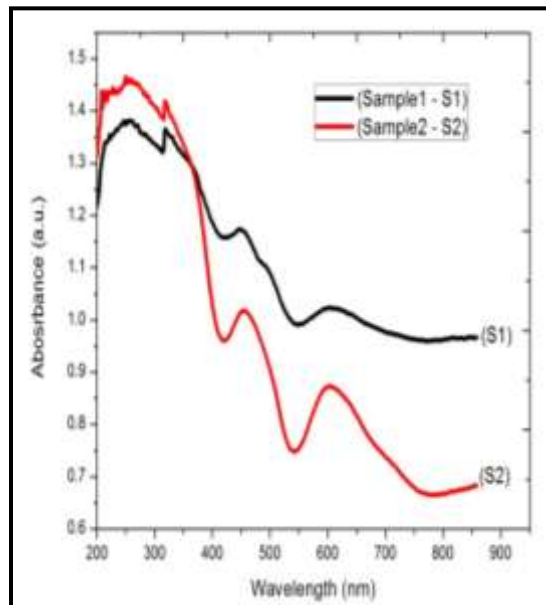


Chart-2 : UV-Vis absorption spectrum of different chromium oxide nanoparticles

3.3 Antibacterial Assay

The figure shows the zone of inhibition of bacteria growth. The different concentration of nanoparticles on agar plates shows a different zone of inhibition. A zone of inhibition depends on the concentration of chromium oxide nanoparticle. If the concentration of nanoparticles increases, then the zone of inhibition also increases. In the results, it was confirmed that the chromium oxide nanoparticles are used as an antibacterial agent. In the figure zone of inhibition was found 10mm and 8mm for sample-1 and sample-2 respectively.

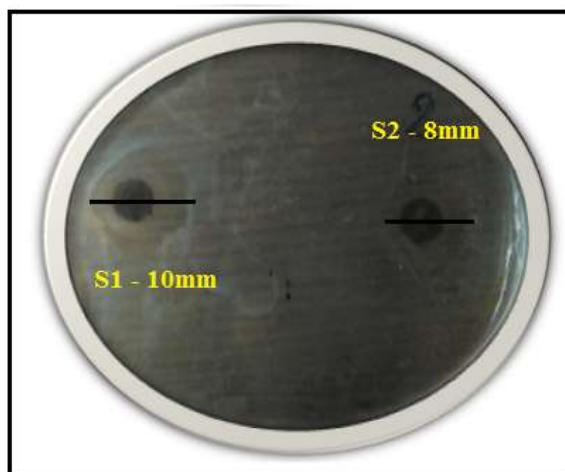


Fig - 1: Image of Antibacterial activity of different concentration of chromium oxide nanoparticles (S1-0.005g and S2-0.005g) on E. Coli

4. CONCLUSION

Chromium oxide nanoparticles were synthesized using chemical method. These prepared samples were characterized by XRD, UV-visible spectrophotometer to analyze its structural and optical properties. The material was confirmed to be nano chromium oxide having hexagonal structure. From XRD we found that the phase was formed with some impurities. The single phased chromium oxide has been prepared by using chemical method. From the XRD analysis, it has been found the crystalline size obtained was of 24nm, 27nm for sample 1 and sample 2 respectively. Therefore the chromium oxide nanoparticles were used as an antibacterial agent for controlling the growth of E.coli bacteria. The Disc diffusion method was mainly used for the antibacterial effect. The zone of inhibition was 10mm and 8mm for sample1 and sample2 respectively. Thus we concluded that the chromium oxide nanoparticles was synthesized by precipitation method and it can be used to show antibacterial activity.

ACKNOWLEDGEMENT

Auhtors would like to thank the following: Medicaps University, UGC-CSR, DAVV indore.

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