

ORIGINAL ARTICLE

SYNTHESIS AND CHARACTERIZATION OF CHROMIUM NANOPARTICLES FROM
TANNERY INDUSTRIES USING PLANT EXTRACTS

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ABSTRACT

Oxidized form of Cr(VI) is extremely toxic, while Cr(III) is relatively inert and easily adsorbed on mineral surface. Indian Tannery industries adopt chromium tanning process owing to its speed, less cost, light colour and greatest quality of the resulting leather. Tanning industry is one of the largest polluters of chromium all over the world. In this process, the leather takes up only 60–80% of applied chromium, and the rest is discharged into the sewage system causing serious environmental impact. Hence, it becomes imperative for tanning industries to minimize the loads of Cr(VI) in the resulting effluents. The aim of the present study was to synthesize and characterize chromium nanoparticle using plant extracts. The present investigation is an attempt to convert chromium present in tannery effluent to chromium nanoparticle using the extracts of Curry leaf and Radish. Chromium nanoparticles were also synthesized from varying concentrations of $K_2Cr_2O_7$ and $KCrO_4$ solution using the extracts of Curry leaf and Radish. Chromium nanoparticles produced from $K_2Cr_2O_7$, $KCrO_4$ and tannery effluents were characterized using spectral studies.

Keywords: Chromium(III), Nanotechnology, Tannery effluent

1. INTRODUCTION

The branch of science that deals with very small nanosized particles of less than 100 nm are known as the nanotechnology and they are all involved in the synthesis, characterization of materials (Sriharitha, 2016). Nanoparticles research is an important aspect in the field of nano technology due to its innumerable application (Kavitha et al., 2013). In the present study chromium nanoparticles are synthesized from the tannery effluents using plant extracts.

Chromium is the 7th most abundant metal in earth's crust (Katz and Salem, 1994) and an important environmental contaminant released into the atmosphere due to its huge industrial use (Nriagu, and Nieboer, 1988). Among the entire industrial waste tannery effluents are ranked as the highest pollutant and which includes compounds like Arsenic, Cadmium, Cobalt, Copper, Chromium, Nickel, Lead, Phenol, Chloride, Sulphide, tannin and formaldehydes (Hasegaven, 2010). Among these chromium plays a major role in polluting water environment. Chromium exists in nine valence states ranging from -2 to +6 of these states, only the Trivalent chromium [Cr(III)] and Hexavalent

chromium [Cr(VI)] have primary environmental significance because they are the most stable oxidation forms in the environment (Smith et al., 2002). Both are found in various forms of water and wastewater. Hexavalent chromium and trivalent chromium species have different chemical and biological characteristics (Cervantes et al., 2001).

Cr (VI) is comparatively more toxic than Cr (III) due to the characteristic like high solubility, rapid permeability through biological membranes and subsequent interactions with nucleic acids & intercellular protein. Cr (VI) more toxic (Sharma and Adholeya, 2012), it is usually associated with oxygen such as chromate (CrO_4) and dichromate ($Cr_2O_7^{2-}$) due to solubility and mobility in soil environment and water. Moreover, Cr (III) is in the form of oxides, hydroxides or poorly soluble sulfates by which it is less mobile (Nriagu, and Nieboer, 1988), (Lofroth and Ames, 1978). Cr (VI) is a strong oxidizing agent and in the presence of organic matter is reduced to Cr (III); this transformation is faster in acidic environment. However, chromium is one of the important essential elements for plants and animals. But in higher amount it becomes toxic. The maximum level of Cr permitted in wastewater is 5mg/L for trivalent chromium and 0.05mg/L for hexavalent chromium (Acar and Malkoc, 2004). But the effluents of these industries contain chromium at concentrations ranging from tenths to hundreds of milligrams per liter (Dermouet et al., 2005).

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In order to reduce the toxicity, it is essential for industries to treat their effluents to reduce the Cr to acceptable levels. Due to more stringent environmental regulations, most of the wastewater produced in huge quantities, laden with Cr. Several methods have been used for removal of toxic metal ions from the effluent. The most commonly used conventional processes to remove Cr (VI) are Precipitation (Patterson,1977), although ion exchange (Tiravanti,1997) and adsorption (Dahbiet *al.*, 1999)and(Orhan and Buyukgangor,1993).However, these technologies have certain limitation such as high operation cost, low removal efficiency, generation of toxic sludge and requirement of high energy. Therefore, Recent developments in nanoscience and nanotechnology pave the way to solve the technological constraint of water purification (Das and Mandal, 2014).

The nanoparticles are synthesised through physical, chemical and biological methods (Chen *et al.*, 2008).The physical and chemical methods are extremely pricey(Li *et al.*, 2009).The biological methods of nanoparticles synthesis would assist to remove ruthless processing conditions, by allowing the synthesis at physiological pH, temperature, pressure, and at the same time, at negligible cost. During the past decade, it has been demonstrated that many biological systems, including plants (Govindarajuet *al.*, 2008) can transform metal ions into metal nanoparticles via the reductive capacities of the proteins and metabolites present in them. It is significant that the nanoparticle production using plants have an important advantages over other biological systems(algae, diatoms, bacteria, yeast, fungi and human cells).Plants are able to reduce the metal ions, especially those which have strong metal ions hyper accumulating reducing capacity and so plant extracts have been used for green synthesis of Nanoparticles.

In an effort towards minimizing the use of toxic chemicals and reduce the generation of hazardous waste while obtaining metal nanoparticles, CSIR–Central Leather Research Institute (CLRI), Chennai has found suitable biosynthetic and/or biomimetic processes of metal nanomaterial synthesis for environmental and biomedical applications(Das and Mandal, 2014). Hence, the present study has been aimed at Chromium Nanoparticles Synthesis using leaf extracts of *MurrayaKoenjil*leaf extracts and*Raphanussativus*(tuber) extracts from Potassium dichromate,Potassium Chromate and tannery effluents.

2.METHODOLOGY

TANNERY EFFLUENTS

The effluents samples were collected from discharge site of the leather in the Dindigul District.

ANALYSIS OF PHYSICAL AND CHEMICAL PARAMETERS

The physicochemical properties such as appearance, odor, pH, Electrical conductivity(EC), Biological oxygen demand (BOD), total dissolved (TDS),Total hardness, Salinity, conductivity, alkalinity were analysed using standard analytical methods.

DETERMINATION OF CHROMIUM (Diphenylcarbazide method)

The hexavalent chromium is determined spectrophotometrically by reaction with diphenylcarbazide in acid solution and the red violet solution is read at 540nm after the incubation for 20mins. (Karaleet *al.*, 2007)

PLANT MEDIATED SYNTHESIS OF CHROMIUM NANOPARTICLES

The plant materials such as leaves of *Murrayakoenigi*and tubers of *Raphanussativus* were washed thoroughly in tap water. 1g of the leaves were weighed using the electronic balance and ground using the mortar and pestle with 10 ml of water and centrifuged at 3000 rpm 2 mins. After that the supernatant were taken and made upto 50ml. 50ml of plants extracts obtained from the leaves of *Murrayakoenigi*and tubers of *Raphanussativus* mixed separately with 0.1M $K_2Cr_2O_7$, 0.1M K_2CrO_4 solutions and Tannery Effluents.These reaction mixture were kept in the dessicator and the obtained powder form of chromium nanoparticled were subjected to futher analysis.

S.No	Reaction Mixtures
1	<i>Murrayakoenigii</i> & $K_2Cr_2O_7$
2	<i>Murrayakoenigii</i> & K_2CrO_4
3	<i>Murrayakoenigii</i> & Tannery effluents
4	<i>Raphanussativus</i> & $K_2Cr_2O_7$
5	<i>Raphanussativus</i> & K_2CrO_4
6	<i>Raphanussativus</i> & Tannery effluents

CHARACTERIZATION TECHNIQUES:

The characterization of chromium nanoparticles were done by UV-spectroscopy(UV-Vis,FTIR and SEM Anlaysis.

Sample preparation for UV-Vis Spectroscopy

A small aliquot of liquid samples was taken and used for the Characterization of Chromium Nanoparticles a UV-Vis spectrometer.the absorbance was measured in the wave length ranging between 400 and 700 nm.

Sample preparation for FTIR and SEM Analysis

The powdered Samples were prepared in a easiest way by placing the samples in between the calcium chloride salts in an air tight container,hence the moisture will be absorbed by $CaCl_2$ salt and the samples are completely dried and then powder were collected from it scrubbing the dried sample sticking on the surface of the beakers. The collected powders were used for FTIR and SEM Analysis.

3.RESULTS AND DISCUSSION

Tannery effluents constitute the bulk of the industrial effluents of the Dindigul area. In the present study, the effluents were collected, analysed for Physico-Chemical parameters. Finally attempts were made with the chromium present in the effluents

Analysis of Effluents:

The effluents collected from the tannery industries around Dindigul areas are subjected to physical analysis.

Colour & Appearance:

The present investigation shows that the effluents were yellow in color with an offensive odor. The color of the sample resembles the potassium chromate solution. (Fig-1) Our results were not in consonance with the findings of (Sugasini and Rajagopal, 2015) who observed, the color of the effluents was blackish in color. Yellow colour of the effluent may be due to the absence of biodegradable and non-biodegradable high molecular weight organic compounds and inorganic chemicals.



Fig 1:- Tannery Effluent

Physio-chemical parameters:

The tannery effluents were subjected to Physio-chemical analysis such as pH, EC, TDS, Total Hardness, Total alkalinity, DO, BOD, Salinity, Conductivity. The results are tabulated below in (Table-1).

Temperature:

The observed temperature of the samples was 29.4 C less than the atmospheric temperature of the Dindigul district. Similar results were reported by (Farhad Ali *et al.*, 2014) in the tannery effluents in Bangladesh.

pH:

The present study observed the average value pH of the effluent sample as 12.13 and hence the sample was found to be highly alkaline in nature. The observed pH values were deviated from the standard values quoted by the Environmental (Protection) rules, 1986. Similar study was also reported by Farhad Ali *et al.*, (2015) in the tannery effluents of Bangladesh who attribute the alkaline condition of the sample to industrial waste of effluents.

Electrical Conductivity (EC):

The electrical conductivity of water or sample is its ability to conduct electric current and it depends on the presence of ions, their total concentration and temperature of water. In the present study, effluents EC value is 1924.75 $\mu\text{s}/\text{cm}$ which exceeds the limits prescribed by WHO (1400 $\mu\text{s}/\text{cm}$) which indicates that discharge of cations and anions were higher in

effluents. This higher conductivity alters chelating properties of water bodies and creates an imbalance of free metal availability for flora & fauna (Akan *et al.*, 2008). Sugasini & Rajagopal (2013) and Farhad Ali *et al.*, (2015) reported the high electrical conductivity similar to the present investigation.

Total Dissolved Study (TDS) :

The entire TDS value evaluation of entire combination of minerals and salts can be known by Nivrutti *et al.*, (2013). In this research the TDS values of the tannery effluent (5340 mg/L) were higher than the standard one (2100 mg/L).

Hardness:

The Hardness of the effluent reported in the present study was found to be less (6 mg/l) than the hardness reported by Sugasini & Rajagopal (2013).

Alkalinity:

Alkaline nature of the tannery effluent may be due to the presence of carbonates and bicarbonates present in the effluent. The investigation reveals that alkalinity of the sample is 210 mg/l and it was less than the Sugasini and Rajagopal (2015)

DO:

The sample contains 8.76 mg/L of Oxygen dissolved and it varies based on the changes in the temperature.

BOD:

In the present study, BOD was found to be .83 mg/L. The microbes present in the effluents consume particular amount of Oxygen to break down the waste.

Salinity and Conductivity:

Salinity is the total concentration of all dissolved salts in the effluents. The salinity of the effluent was 7.02 ppt and therefore, salinity is the strong contributor to conductivity and so 9.89 ms in the measured conductivity value in the effluents

S.No	Parameters	Values
1	Temperature	29.4 C
2	pH	12.13
3	EC $\mu\text{s}/\text{cm}$	1924.75
4	TDS (ppt)	5340
5	Hardness (mg/l)	6
6	Alkalinity (mg/l)	210
7	DO (mg/l)	8.7
8	BOD (mg/l)	.83
9	Salinity (ppt)	7.02
10	Conductivity (ms)	9.89

Table:1 – Physio-chemical parameters of effluents

Determination Of Hexavalent Chromium:

The hexavalent Chromium present in the effluents were determined using the Diphenylcarbazide method, Dichromate and Chromate Solutions were used as the standard for determination of Hexavalent Chromium present in the effluents.

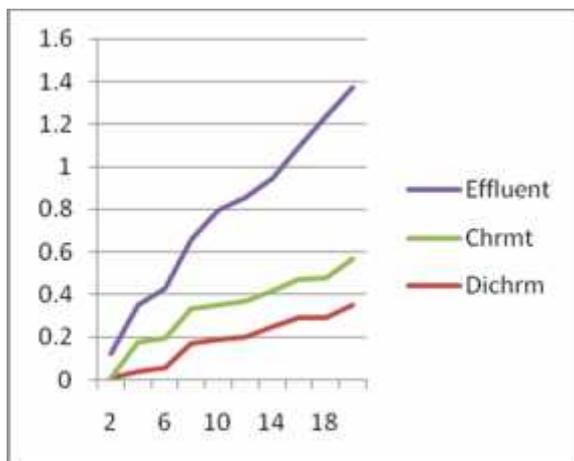


Fig:-2 Shows that the Sample Contained Hexavalent Chromium

Synthesis and Characterization of Chromium Nanoparticles

In the present study the tannery effluent were mixed with the Extracts of *Murrayakoenigii*, *Raphanussativus*, $K_2Cr_2O_7$, K_2CrO_4 for the synthesis of chromium Nanoparticle and are subjected to

Spectrometric Analysis, FTIR Analysis, and SEM Analysis.

Spectrometry

Synthesis and Characterization of Chromium Nanoparticles from $K_2Cr_2O_7$ and *Murrayakoenigii*

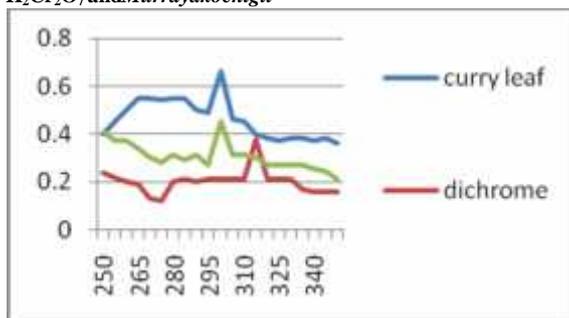


Fig 3:-Absorption Spectrum of $K_2Cr_2O_7$ and *Murrayakoenigii*

Fig 3 shows the Absorption Spectrum of dichromate solution which had λ max 315nm upon mixing with the *Murrayakoenigii* λ max shifts to 300nm. The observed Shift of plasmon peak in an indication of production of Chromium Nanoparticles.

4.5.1.b.Synthesis and Characterization of Chromium Nanoparticles from K_2CrO_4 and *Murrayakoenigii*

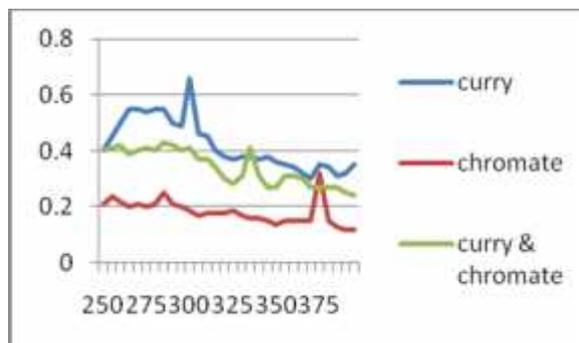


Fig 4:- Absorption Spectrum of K_2CrO_4 and *Murrayakoenigii*

Fig 4 shows that Absorption Spectrum of Chromate solution which had λ max 375nm upon mixing with the *Murrayakoenigii* λ max shifts to 355nm. The observed Shift of plasmon peak in an indication of production of Chromium Nanoparticles.

Synthesis and Characterization of Chromium Nanoparticles from Tannery Effluents using the Extracts of *Murrayakoenigii*

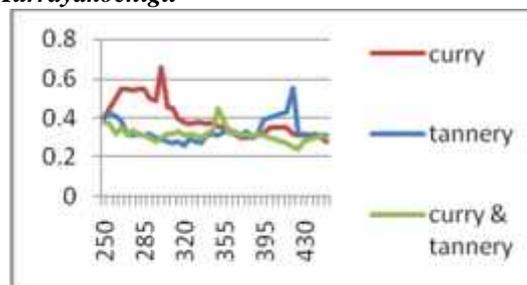


Fig.5 Absorption Spectrum of Tannery Effluents & *Murrayakoenigii*

From the Fig.5 It was quite interesting to note that the λ max of effluent was found to be 420nm and shifted to 350nm in the reaction mixture of *Murrayakoenigii* and tannery effluent after the incubation. The observed shift of plasmon peak in an indication of production of chromium nanoparticles.

Synthesis and Characterization of Chromium Nanoparticles from $K_2Cr_2O_7$ and *Raphanussativus*

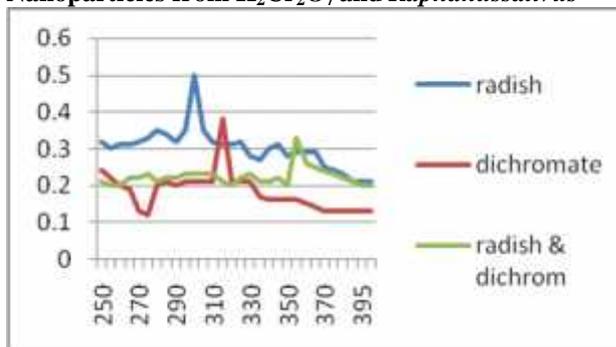


Fig .6 Absorption Spectrum of $K_2Cr_2O_7$ and *Raphanussativus*

Fig 6 shows that Absorption Spectrum of dichromate solution which had λ max 315nm upon mixing with the *Raphanussativus* λ max shifts to 335nm. The observed Shift of plasmon peak in an indication of production of Chromium Nanoparticles.

4.5.1.e. Synthesis and Characterization of Chromium Nanoparticles from K_2CrO_4 and *Raphanussativus*

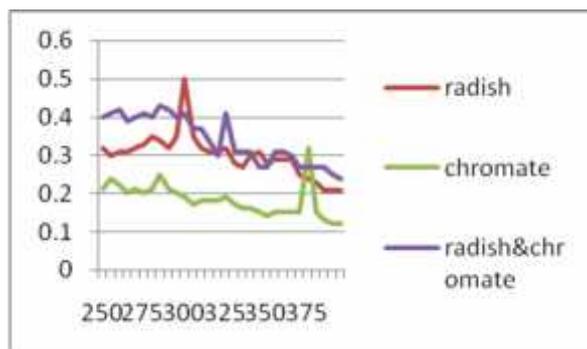


Fig.7 Absorption Spectrum of K_2CrO_4 and *Raphanussativus*

Fig 7 shows that Absorption Spectrum of Chromate solution which had λ max 375nm upon mixing with the *Raphanussativus* λ max shifts to 325nm. The observed Shift of plasmon peak in an indication of production of Chromium Nanoparticles

Synthesis and Characterization of Chromium Nanoparticles from Tannery Effluents Using the Extracts of *Raphanussativus*

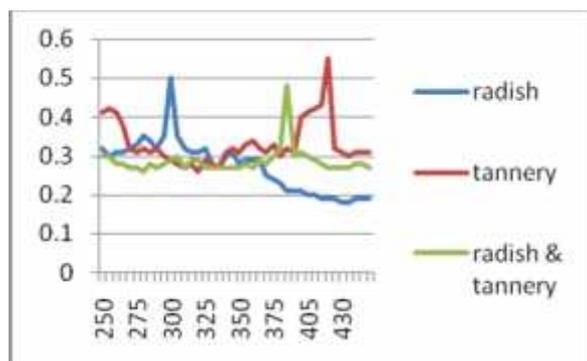


Fig.8 Absorption Spectrum of Tannery Effluents and *Raphanussativus*

From Fig 8 It is quite interesting to note that the λ max of tannery effluent was found to be 420nm and shifted to 385nm in the reaction mixture *Raphanussativus* and tannery effluent after the incubation. The observed shift of plasmon peak in an indication of production of chromium nanoparticles.

FTIR spectroscopic analysis

FTIR spectroscopy is widely used to study the nature of surface absorbents in nanoparticles. Since the nanoparticles possess large surface area, the modification of the surface by a suitable absorbate can generate different properties. The FTIR spectra of the nanoparticles, which contain some absorbates, possess additional peaks in comparison with the FTIR pattern of a bare nanoparticle. So the property change with different absorbates can easily be detected with FTIR Spectroscopy. FTIR analysis of respective leaf broths of *Murrayakoenigii*, *Raphanussativus*, Tannery effluent, Reaction mixture of Dichromate solution & *Murrayakoenigii*, Chromate solution & *Murrayakoenigii*, Tannery effluent

& *Murrayakoenigii*, Dichromate solution & *Raphanussativus*, Chromate solution & *Raphanussativus*, Tannery effluent & *Raphanussativus* and were done using FTIR spectrometer

FTIR Spectroscopic analysis of Reaction mixture of Dichromate solution & *Murrayakoenigii*

FT-IR Analysis of Dichromate solution & *Murrayakoenigii* were done using FTIR Spectrometer is given below.

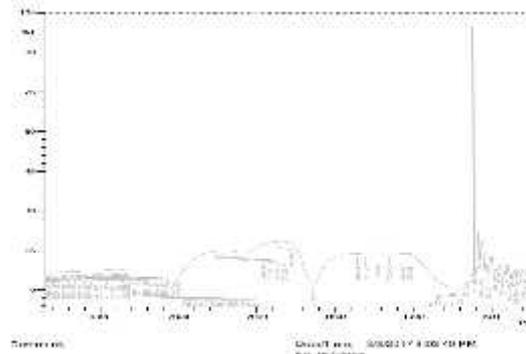


Fig.9 FTIR Spectrum of Reaction mixture of Dichromate solution & *Murrayakoenigii*

The characteristics of Chromium Nanoparticles were observed FTIR spectrum. Fig.9 Shows the peak at 542 cm^{-1} , 569 cm^{-1} , 1301.95 cm^{-1} , 3417.86 cm^{-1} in the region 400 cm^{-1} - 4000 cm^{-1} . The peak at 3417.86 cm^{-1} is the characteristic band of hydrogen bonded OH groups present in aqueous phase. The peak at 1301.95 cm^{-1} indicates the presence of (-COO-) carboxylate ions, responsible for stabilizing the Chromium Nanoparticles. The peak at 569 cm^{-1} and 542 cm^{-1} indicates that Cr=O and Cr-O vibration of Chromium Nanoparticles

FTIR Spectroscopic analysis of Reaction mixture of Chromate solution & *Murrayakoenigii*

FT-IR Analysis of Chromate solution & *Murrayakoenigii* were done using FTIR Spectrometer is given below.

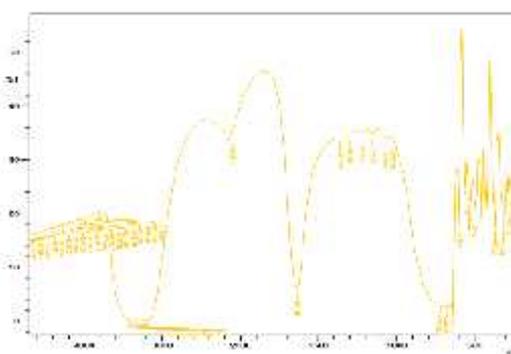


Fig.10 FTIR Spectrum of Reaction mixture of Chromate solution & *Murrayakoenigii*

The characteristics of Chromium Nanoparticles were observed FTIR spectrum. Fig.10 Shows the peak at 563 cm^{-1} , 1301.95 cm^{-1} , 1359.82 cm^{-1} , 3419.86 cm^{-1} in the region 400 cm^{-1} - 4000 cm^{-1} . The peak at 3419.86 cm^{-1} is the characteristic band of hydrogen bonded OH groups present in aqueous phase. The peak at 1359.82 cm^{-1} and 1301.95 cm^{-1}

indicates the presence of (-COO-) carboxylate ions, responsible for stabilizing the Chromium Nanoparticles. The peak at 563cm⁻¹ indicates that Cr=O and Cr-O vibration of Chromium Nanoparticles.

FTIR Spectroscopic analysis of Reaction mixture of *Murrayakoenigii* & Tannery Effluent.

FT-IR Analysis of *Murrayakoenigii* and Tannery effluent were done using FTIR Spectrometer is given below

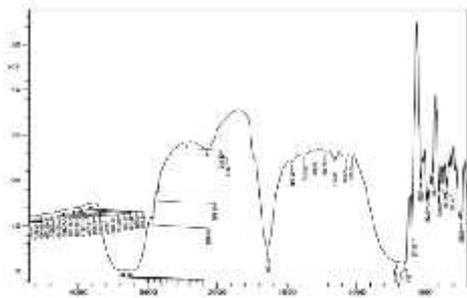


Fig.11 FTIR Spectrum of Reaction mixture of *Murrayakoenigii* & Tannery Effluents

The characteristics of Chromium Nanoparticles were observed FTIR spectrum. Fig.11 Shows the peak at 534 cm⁻¹, 601.79cm⁻¹, 1300.02 cm⁻¹, 1373.32 cm⁻¹, 3412.08 cm⁻¹ in the region 400cm⁻¹-4000cm⁻¹. The peak at 3412.08cm⁻¹ is the characteristic band of hydrogen bonded OH groups present in aqueous phase. The peak at 1373.32 cm⁻¹ and 1300.02cm⁻¹ indicates the presence of (-COO-) carboxylate ions, responsible for stabilizing the Chromium Nanoparticles. The peak at 534cm⁻¹ and 601.79cm⁻¹ indicates that Cr=O and Cr-O vibration of Chromium Nanoparticles.

FTIR Spectroscopic analysis of Reaction mixture of Dichromate solution & *Raphanussativus*

FT-IR Analysis of Dichromate Solution and *Raphanussativus* and were done using FTIR Spectrometer is given below

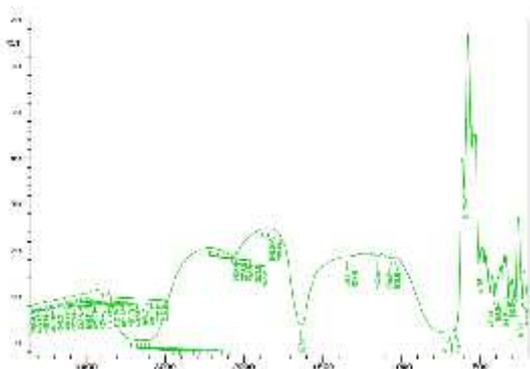


Fig.12 FTIR Spectrum of Reaction mixture of Dichromate solution & *Raphanussativus*

The characteristics of Chromium Nanoparticles were observed FTIR spectrum. Fig.12 Shows the peak at

601.79cm⁻¹, 1300.02 cm⁻¹, 1346.31 cm⁻¹, 3431.36 cm⁻¹ in the region 400cm⁻¹-4000cm⁻¹. The peak at 3431.36cm⁻¹ is the characteristic band of hydrogen bonded OH groups present in aqueous phase. The peak at 1346.31 cm⁻¹ and 1300.02cm⁻¹ indicates the presence of (-COO-) carboxylate ions, responsible for stabilizing the Chromium Nanoparticles. The peak at 601.79cm⁻¹ indicates that Cr=O and Cr-O vibration of Chromium Nanoparticles

FTIR Spectroscopic analysis of Reaction mixture of Chromate solution & *Raphanussativus*

FT-IR Analysis of Chromate solution and *Raphanussativus* and were done using FTIR Spectrometer is given below

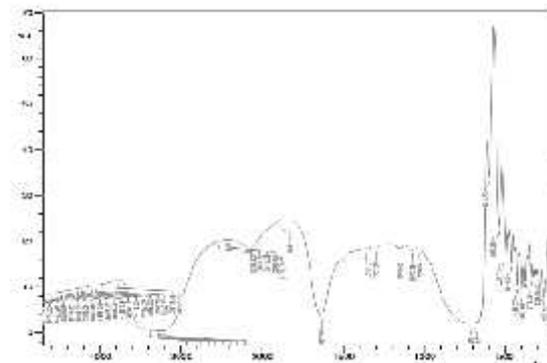


Fig.13 FTIR Spectrum of Reaction mixture of Chromate solution & *Murrayakoenigii*

The characteristics of Chromium Nanoparticles were observed FTIR spectrum. Fig.13: Shows the peak at 534.28 cm⁻¹, 601.79cm⁻¹, 1301.95 cm⁻¹, 1352.1 cm⁻¹, 3379.29 cm⁻¹ in the region 400cm⁻¹-4000cm⁻¹. The peak at 3379.29cm⁻¹ is the characteristic band of hydrogen bonded OH groups present in aqueous phase. The peak at 1352.1 cm⁻¹ and 1301.95 cm⁻¹ indicates the presence of (-COO-) carboxylate ions, responsible for stabilizing the Chromium Nanoparticles. The peak at 534.28 cm⁻¹ and 601.79cm⁻¹ indicates that Cr=O and Cr-O vibration of Chromium Nanoparticles.

FTIR Spectroscopic analysis of Reaction mixture of *Raphanussativus* & Tannery Effluent.

FT-IR Analysis of *Raphanussativus* and Tannery Effluents were done using FTIR Spectrometer is given below

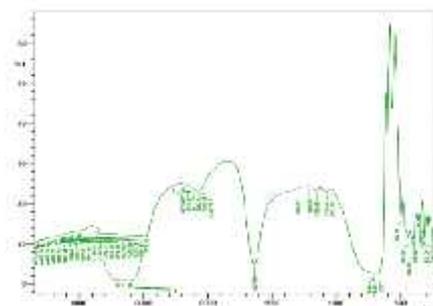


Fig.14 FTIR Spectrum of Reaction mixture of *Raphanussativus* and Tannery Effluents

The characteristics of Chromium Nanoparticles were observed FTIR spectrum. Fig.14: Shows the peak at 497.63 cm⁻¹, 671.23cm⁻¹, 1300.02 cm⁻¹, 3417.86 cm⁻¹ in the region

400 cm^{-1} -4000 cm^{-1} . The peak at 3417.86 cm^{-1} is the characteristic band of hydrogen bonded OH groups present in aqueous phase. The peak at 1300.02 cm^{-1} indicates the presence of (-COO-) carboxylate ions, responsible for stabilizing the Chromium Nanoparticles. The peak at 497.63 cm^{-1} and 671.23 cm^{-1} indicates that Cr=O and Cr-O vibration of Chromium Nanoparticles. Similar Study of FTIR Analysis of Chromium Nanoparticles were reported by Farzaneh and Najafi(2011).

Scanning Electron Microscopic Studies

SEM analysis of Chromium nanoparticles that were synthesized from the reaction media was done using Scanning electron microscopy. It is a type of electron microscope that images the sample surface by scanning it with high energy streams of electrons in a raster scan pattern, and the beam's position is combined with the detected signal to produce an image. The most common SEM mode is detection of secondary electrons emitted by atoms excited by electron beam. By scanning the sample and collecting the secondary electrons that are emitted using a special detector, an image displaying the topography of the surface is created.

Fig .(E-H).SEM image of Chromium Nanoparticles Synthesized from *Murrayakoenigii* & Tannery Effluent

From the SEM Analysis it has been found that the Size of the Chromium Nanoparticles ranged from 20-900nm. Further Studies is Warrented to have the size Controlled production of Nanoparticles.

Fig(A-D) SEM Image of Chromium Nanoparticles Synthesized from *Murrayakoenigii* & Dichromate Solution

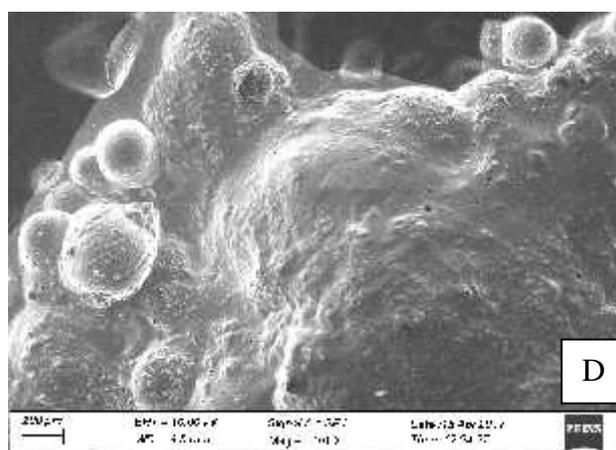
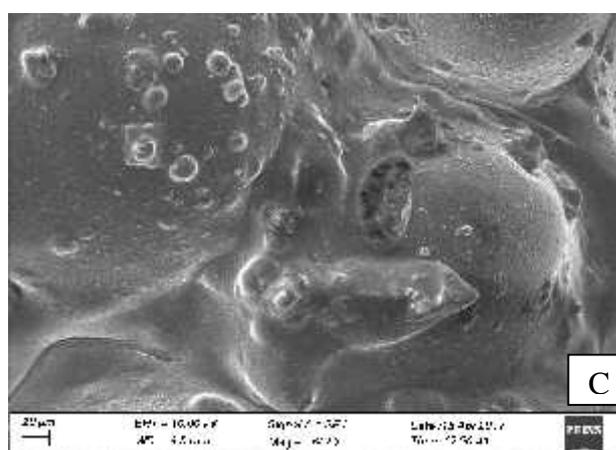
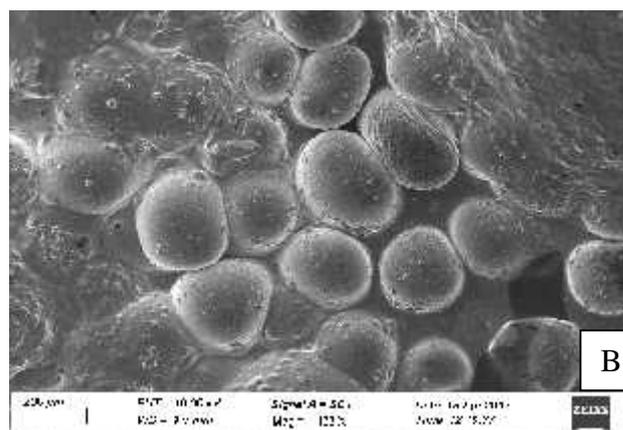
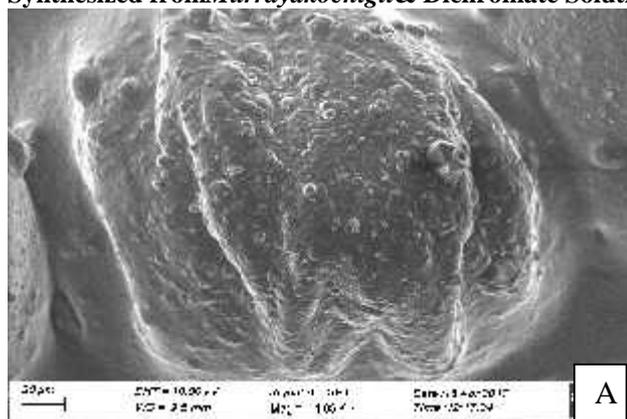
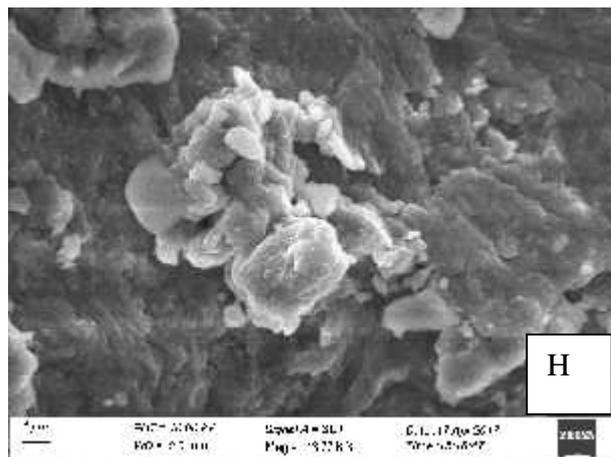
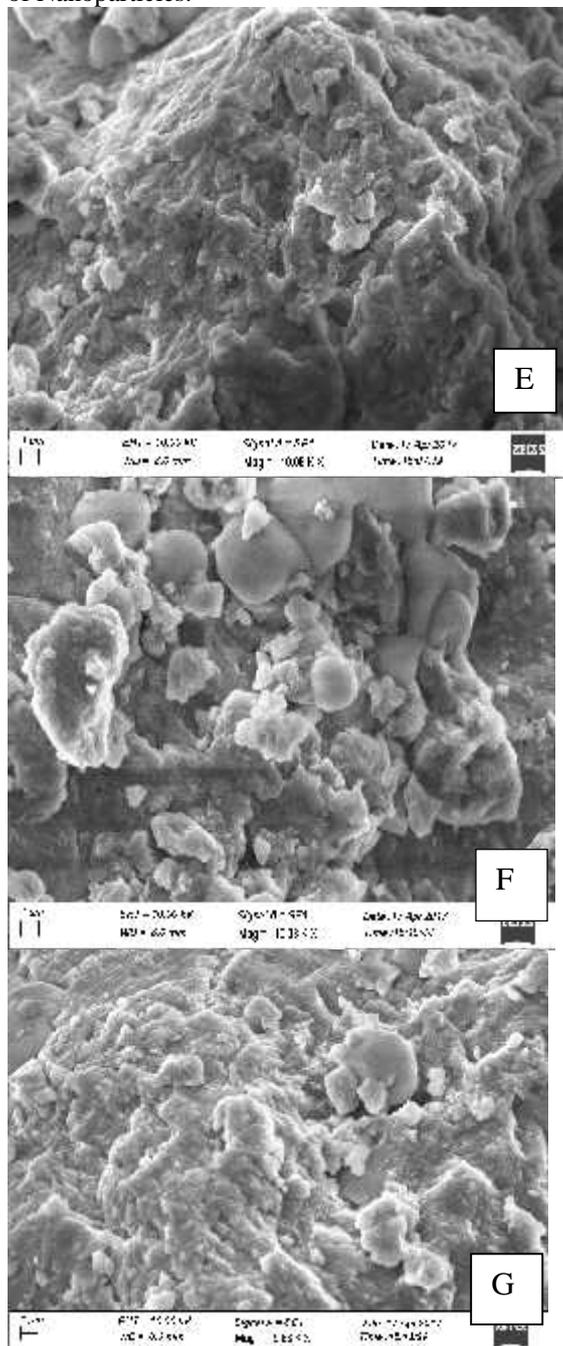


Fig .(E-H).SEM image of Chromium Nanoparticles Synthesized from *Murrayakoenigii* & Tannery Effluent
From the SEM Analysis it has been found that the Size of the Chromium Nanoparticles ranged from 20-900nm. Further Studies is Warrented to have the size Controlled production of Nanoparticles.



4.ACKNOWLEDGEMENT

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