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Synthesis, characterizations and Antibacterial Studies of Chromium trioxide Nanoparticles

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ABSTRACT

Chromium trioxide (CrO_3) nanoparticles weresynthesized by solvothermal method. Thesynthesized CrO_3 nanoparticle was characterized by XRD, SEM, EDX, FTIR analysis techniques. From XRD study, it is found that the prepared samples are rhombohedra system, and scanning electron microscopy (SEM) pattern confirmed the spherical morphology.Nanoparticles. The various functional groups in CrO_3 nanoparticles were ascertained by FTIR analysis. Antimicrobial study of CrO_3 NPs showed excellent antibacterial activity against various bacterial strain like. Escherichia Coli,Enterobacteraerogen, Klebsillaspp,Serratiamarcescens.moreoverSerratiamarcescens, Serratiamarcescensexhibited the highest sensitivity to CrO_3 NPs whereEscherichia Coli, Klebsillaspphas least sensitivity. The possible mechanisms of antimicrobial activity of CrO_3 NPs werefunctionally investication.

Key words; XRD, SEM,EDX,FTIR,Antimicrobial activity

1. INTRODUCTION

In chemical sciences. The synthesis of transition metal and metal oxide nanoparticles is a growing research field.[1-3] As the metal particles are reduced in size, bulk properties of the particles disappear to be substituted to that of quantum dot following quantum mechanical rules.[5-7] It can thus be easily understood that metal nanoparticles chemistry differs from that of the bulk materials.[4] Since with size reduction the high surface area to volume ratio lead to enhanced catalytic activity.[9-12]. In the nanotechnology, development to rapid, simple, cost-effective, and ecofriendly procedures for the synthesis of nanoparticle is worth.[13] Furthermore, the improvement of experimental processes for the synthesis of these nanoparticles of different sizes, shapes, and controlled disparity has many importantfeautres. [16-18]Factors strongly affects the physical and chemical properties and their potential application in optoelectronics, electronic, recording media, sensing devices, catalysis, bimolecular detection, and medicine.[22,28,17] To now, several methods have been reported for the synthesis of nanomaterials.[15] However, biological synthesis of nanoparticles has received extensive interest due to chemical methods that are capital intensive, toxic, and have low productivity.[18,19] Several biological method suing microorganisms (including bacteria, fungi, actinomycetes, and yeast),[33,28]Nanoparticles of chromium are important technological materials which show novel electronic, thermal, optical, mechanical and chemical properties. [29]. These properties are significantly different from those of bulk materials because of large surface to value area and extremely small size. Transition metals play an important role in many area of chemistry, physics, material science and electronic engineering chromium nanoparticles have wide industrial application,[27] Transition metal oxide NPs hasmany applications as catalyst, sensors superconductors and adsorbents.[20] Metal- oxides constitute an important class of materials that are involved in environmental science, electrochemistry, biology, chemical sensors, magnetism and other fields. Chromium oxides have attracted much attention recently because of their importance both in science and technology.[28-31] As the chromium has different stable oxidation states, it can form the different types of oxides. Special attention has been focused on the formation and properties of chromium oxide (CrO_3), which is important in specific applied applications such as in high temperature resistant materials. Nanomaterial's, particularly transition-metal oxides play an important role in many areas of chemistry, physics and materials science.[18,22] In technological applications, metal oxides have traditionally been used in the fabrication of microelectronic circuits, sensors, piezoelectric devices, fuel cells, coatings for the passivation of surfaces against corrosion, and as catalysts Among metal oxides, special attention has been focused on the formation and properties of chromium (CrO_3) which is important as heterogeneous catalyst, coating material, wear resistance, advanced colorant, pigment and solar energy collector Transition metal oxide NPs have many applications as catalyst, sensors, superconductors and adsorbents.[30-31] Metal-oxides constitute an important class of materials that are involved in environmental science, electrochemistry, biology, chemical sensors, magnetism and other fields. Chromium oxides have attracted much attention recently because of their importance both in science and technology. [27] As the chromium has different stable oxidation states, it can form the different types of oxides.[18,24,27] Special attention has been focused on the formation and properties of chromium oxide (CrO_3), which is important in specific applied applications such as in high temperature resistant materials. Nano size materials exhibit unique electronic magnetic, optical, catalytic and medical properties as compared with the traditional and commercial bulk materials its due to its quantum size effect, large surface to value ratio.[25,29]

2. CHEMICAL NEEDED

The Chemicals used in the experimental were analyticalgrade. The chemical used in syntheses were chromium trioxide (CrO_3), ethylene glycol ($C_2H_6O_2$), absoluteethanol (C_2H_6O), and citric acid ($C_6H_8O_7$), were purchased from sigma Aldrich.

3. METHODS

1g of chromium oxide and citric acid were dissolved in 50ml of ethanol in two different beakers under stirred at the rotation speed of 300rpm. 0.5 ml of ethylene glycol was slowly then added to the mixture of chromium oxide and absolute ethanol. After the chemical had completely dissolved both solution were mixed together and continued string for 1h to allowed complete reaction. The reaction was carried out under room temperature after the reaction has completed the solution will dry in oven at 800c for overnight. The resulting gel will undergo heat treatment of 4000c for 2 h in furnace. The powder formed after heat treatment was a chromium trioxide nanoparticle which was dark black green in color.

4. RESULT AND DISCUSSION

4.1 X-RAY DIFFRACTION ANALYSIS

X-ray diffraction is obviously the most common tool to study the crystal structure of nanoparticles. The grain size and the structure of the nanoparticles are investigated with a powder diffract meter with radiation at a diffraction angle between 20º to 80 º. The powder X-ray diffraction of chromium trioxide is performed using automated powder X-ray diffract meter (BRUKER D2 PHASER powder diffract meter) operating at wavelength 0.15405 nm. From the XRD pattern the particle size D was calculated using Scherer's formula

$D = K\lambda / \beta \cos \theta$,

Where K is a shape factor, λ is the wavelength of the incident X-ray and θ is diffraction angle and β is the Full width half maximum. The dislocation density (δ) of the nanoparticles, which is a measure of the number of dislocation in a unit volume of a crystalline material by the formula $\delta = 1/D^2$.

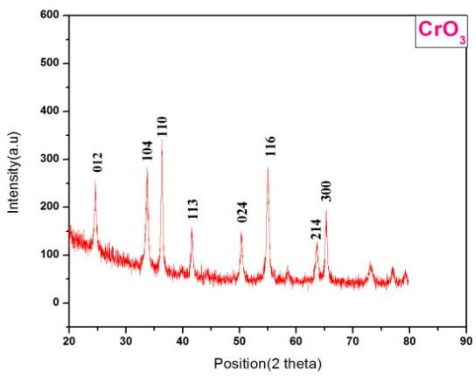
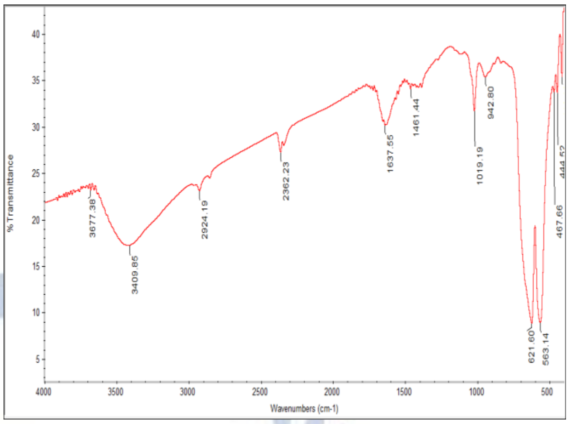


Fig 4.1 XRD PATTERN OF CrO_3NANOPARTICLES

From the Figure it is shown that sharp peaks are observed, which shows the crystallinity perfection ofCrO_3nanoparticles. The inspection of XRD pattern revealed that CrO_3 thus formed is of rhombohedra phase (JCPDS no. 38-1479 with a=4.9587 A˚c=13.594 A˚). The major peaks at 2 theta values of 24.61, 33.7, 36.4, 41.8, 50.32, 54.9, 63.6, and 65.27 are indexed as (012), (104), (110), (113), (024), (116), (214), (300) respectively. Average particle size of theCrO_3nanoparticles was found to be 28.0481nm using Scherrer'sformula. Dislocation density of the prepared nanoparticles is $1.27\times 10^{15} m^3$

4.2 FOURIER TRANSFORM INFARED ANALYSIS

The FTIR spectra for all prepared samples were analysed using Bruker,Alpha T model spectrometer.The elemental chemicals analysis of the synthesized chromiumtrioxide nanoparticles was analyzed by FTIR spectrometer.Fourier Transform spectroscopy is a technique which is used to obtain an infared spectrum and to measure the vibrational frequencies of bonds in the molecule.Fig 4.2 shows the FTIR spectrum of the CrO_3 nanoparticles synthesis by sol gel method.A number of vibration bands can be seen in the region of 4500-500 cm .. The functionalgroups presented in CrO_3 are tabulated in the Table4.2.The properties of peaks are weak,medium or strong.



4.2.1 FTIR Spectrum of chromiumtrioxide Nanoparticles

Fig.4.2 shows that FTIR spectra CrO_3 nanoparticle synthesised by sol-gel technique.FTIR spectroscopy FTIR spectroscopy was carried out to ascertain the purity and nature of metal or metal oxide nanoparticles. The band between 3409 and 1637/cm are due to the-OH stretching and bending vibrations of adsorbed water molecule on the sample. Band at 2924/cm may be due to $-CH_3$ stretching vibrations, 1019/cm is due to Cr-O-Cr vibrations 942/cm are assigned to Cr=O vibrations. The two peaks at 563 and 621/cm are assigned to Cr-O str. modes are evident for the presence of crystalline Cr_2O_3 nanoparticles.

4.2 Wave number assignments of CrO_3 nanopartilces

EXPERIMENTAL VALUE cm^{-1}	REPORTED VALUE cm^{-1}	ASSIGNMENT
3409.85	3400	-OH stretching bending vibrations
1637.55	1622	-OH stretching bending vibrations
2924.19	2929	$-CH_3$ stretching vibrations
1019.19	1072	Cr- O-Cr
942.86	952	Cr = O
621.60	617	Cr – O stretching
563.14	550	Cr-O stretching

4.3 EDAX Analysis:

Fig.4.3 shows the typical EDAX pattern of as prepared CrO_3 nanoparticles. The quantitative composition analysis of CrO_3 nanoparticles was carried out using Energy Dispersive X-Ray Spectroscopy measurement. The concentration of elements Cr and O varies periodically along the atom size being accompanied with maxima of CrK along 14.4% and O along 55% as shown Table4.3,

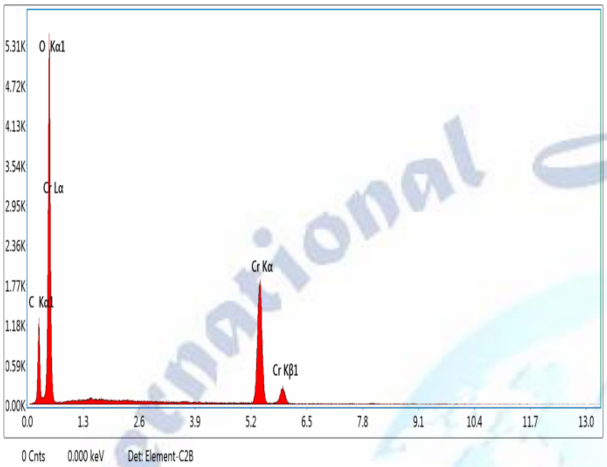


Fig 4.3. EDAX spectrum of as prepared CrO_3 nanoparticles

Table 4.3-2. EDAX data of as prepared CrO_3nanoparticles

Element	Weight %	Atomic %	Error %	Kratio
Ck	18.4	30.6	8.4	0.0795
Ok	44.2	55.1	7.4	0.1967
CrK	37.4	14.4	2.3	0.3189

There is no evidence of impurity in the composition. Fine particles tends to form agglomerated and fine spherical shape of powered particles are frequently seen on the surface of powders from SEM analysis. The surface concentration of oxygen looks like coarse islands the height is also considered. The CrO_3 atom in the oxidation state are concentration in the oxide from is the metallic state as shown in the SEM image.

4.4 SEM studies:

The CrO_3nanoparticle in the SEM image is shown in Fig4.4. Depicts a spherical nanoparticle structure. The size of the CrO_3 nanoparticle ranges between 20 and 60 nm. Moreover, a few of them were slightly agglomerated which due to the range volume of heat is carried out during the synthesis procedure. However the small sized particles were highly reactive due to sharp edges, which contain a high surface to volume ratio and less cohesive energy compared with bulk materials. The instrumental parameters, accelerating voltage spot size and magnification and working distances are marked on the SEM image as shown in the Fig 4.4. The results indicate that monodispersive and highly crystalline CrO_3 nanoparticles are obtained, so we can infer that the as prepared CrO_3nanoparticles are in the nanometer range. The average diameter of the particle observed from SEM analysis 94 nm which is larger than the diameter predicted from the X-ray broadening.

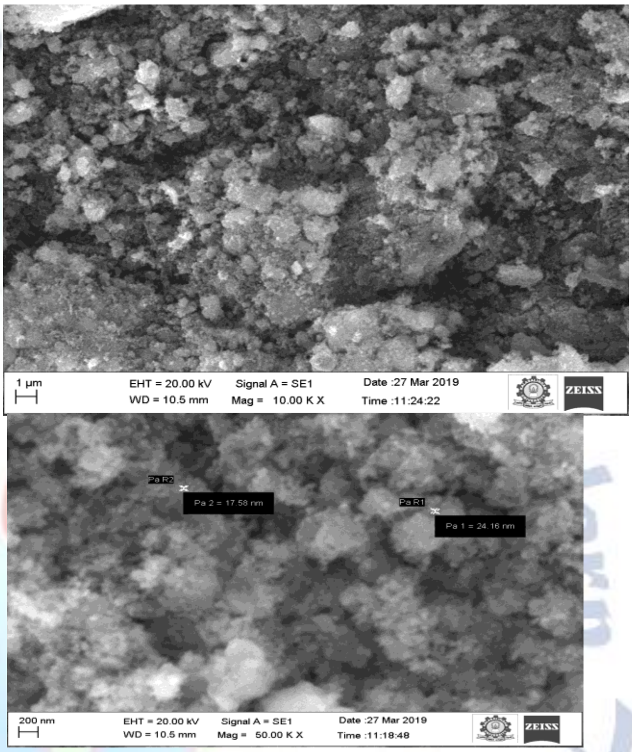


Fig 4.4 SEM Images at 200nm, 1µm of as prepared CrO_3 nanoparticles

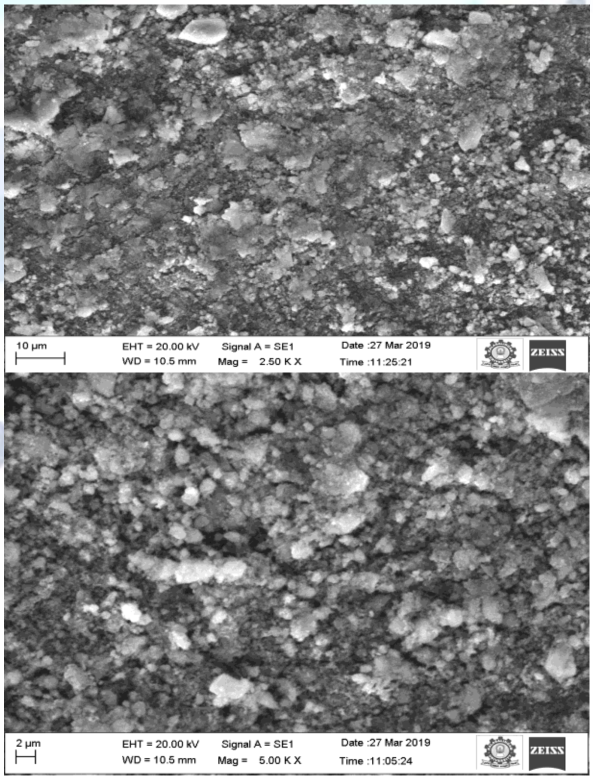


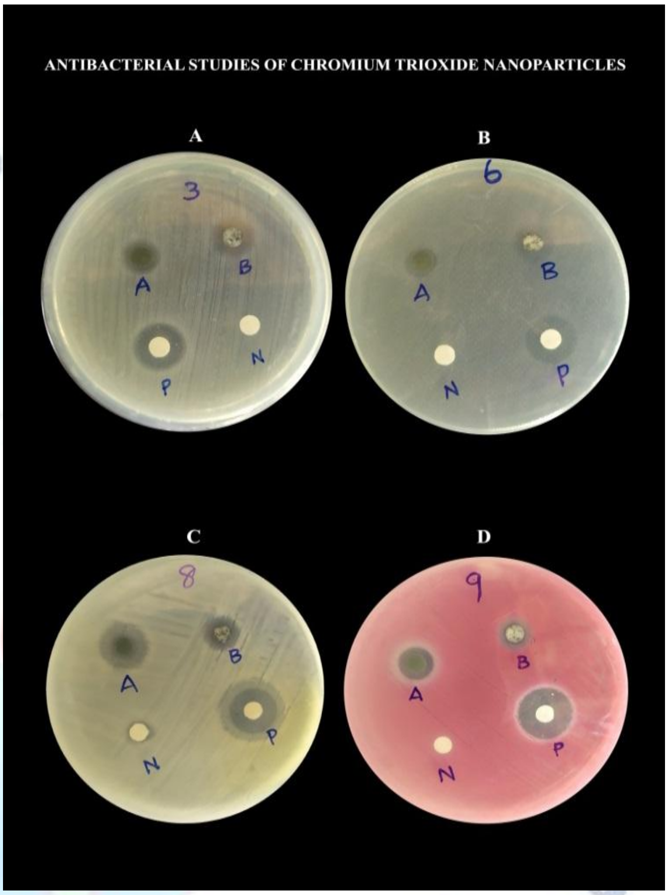
Fig 4.4 SEM Images at 2μm,10μm of as prepared CrO_3 nanoparticles

5. ANTIBACTERIAL ACTIVITY

Antibacterial activity is defined as killing bacteria or reducing their growth without general toxic to surrounding tissue of body. There are several reports about antibacterial properties of NPs. The antibacterial properties of the as synthesized nanoparticles were evaluated by a qualitative method against the aforementioned microorganisms using the agar disk diffusion method as described previously. Gram-positive and Gram-negative bacteria were cultured on LB agar medium (Fluka) while yeast was cultured on potato dextrose agar (Becton Dickinson Difco, Franklin Lakes, NJ, USA). Briefly, 20 mL of liquid Mueller Hinton agar (pH 7.3 ± 0.2 at 25°C) was poured onto disposable sterilized Petri dishes and allowed to solidify. The surfaces of the solidified agar plates were allowed to dry in the incubator prior to streaking of microorganisms onto the surface of the agar plates. Next, 100 μL of the microbial culture suspension in broth containing approximately 106 colony-forming units per mL as measured spectrophoto- metrically was streaked over the dried surface of the agar plate and spread uniformly using a sterilized glass rod and allowed to dry before applying the loaded disks. The CrO_3 nanoparticle compounds were suspended in sterilized distilled water, and blank sterilized Whatman No 1 filter paper disks were loaded with the suspension. The loaded disks were applied carefully to the surface of the seeded agar plates using sterile forceps. The experiment was carried out in triplicate and the diameters of the zones of inhibition were measured after 24 hours of incubation at 37°C. Standard antimicrobial agents including nystatin (for yeast, 100 mg/mL), ampicillin (for Gram-negative bacteria, 100 mg/mL), and streptomycin (Gram-positive bacteria, 100 mg/mL) were used as controls.

Anti bactrial Results;

Excellent antimicrobial activities against a range of bacteria were evaluated. The diameter of inhibition zone reflects magnitude of susceptibility of microbes, the strains susceptibility of microbes. The diameter of inhibition zone reflects magnitude of susceptibility of microbes. The strains susceptible to CrO_3 NPs exhibited larger zone of inhibition, whereas resistant strains exhibit smaller zone of inhibition. According to zone of inhibition*Enterobactoraerogen,Serratiamarcescens*exhibited the highest sensitivity toward CrO_3 NPs while *E. coli, K. pneumonia* showed the least sensitivity among the tested microbes.



A-A.Escherichia Coli, A-B.Enterobacteraerogen, A-C.Klebsillaspp,A-D.Serratiamarcescens.

TABLE 5.2: ANTIBACTERIAL EFFECTS OF VARIOUS EXTRACT

S.No	Tested organisms	Zone of inhibition (mm)	Positive control (mm)	Negative control (mm)	Percentage of inhibition (%)
1	*Escherichia coli*	2	4	0	50
2	*Enterobactera erogenes*	2	12	0	16
3	*Klebsiella pneumonia*	5	6	0	83
4	*Serratiamarc escens*	3	5	0	60

6. CONCLUSION

CrO_3 NPs with rhombohedral structure are synthesized successfully by solvothermal method.SEM micrographic clearly show the surface features by which it is shows that CrO_3 NPs was successfully prepared.EDAX spectra show the elements of crystal matrix CrO_3.The FTIR analysis ascertains all the functional groups in CrO_3 NPs. The main application of such Nano formulations relies on their antimicrobial ability that allows for the development of multiple products, from antimicrobial solutions utilized to disinfect the surfaces and medical devices to antimicrobial wound dressings, textiles and coatings. For improving their applications on the biomedical field, researchers are striving to find optimal synthesis approaches in order to decrease the toxicity of CrO_3NPs but at the same time to keep or even improve their efficiency in diagnosis, therapy and, maybe even prophylaxis. CrO_3 NPs have potential for external uses as antibacterial agents in surface coatings on various substrates to prevent microorganisms from attaching, colonizing, spreading, and forming biofilms in indwelling medical devices. This study suggests that mechanisms of antimicrobial response of CrO_3 NPs in different species of bacterial should be further investigated.

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