



Eco-Friendly Green Synthesis of Chromium Oxide Nanoparticles and Their Characterization and Application

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Abstract

In recent years the nanoparticle design synthesis methods conducted by plants replaced the traditional chemical procedures of nanoparticles preparations. This study demonstrates the environmental kindly preparations of chromium oxide nanoparticles (Cr_2O_3 -NPs) using leaf extract of *Mentha pulegium* plant. The obtained Cr_2O_3 -NPs was characterized by Ultra Violet/Visible spectroscopy, Fourier Transform Infra-Red (FTIR), Scanning Electron Microscopy (SEM), Energy Dispersive X-ray spectrophotometer (EDX) and X-ray diffraction studying. The presence of the Sharp peak at 462 nm in the Ultraviolet/Visible spectrum and the color changed by surface plasma resonance explains that Cr_2O_3 -NPs is formed. The presence of oxygen and chromium in Cr_2O_3 -nanoparticles is reported by EDX spectrum, which was found to consist of 32.57% Cr and 44.41% O, confirming the high purity of the Cr_2O_3 -NPs powder. Since Scan Electron Microscope studying reported an irregular round morphology shape. In addition, X-ray diffraction studying suggested their crystalline feature by the characteristic peaks at $2\theta = 24.3^\circ, 33.5^\circ, 36.2^\circ, 41.5^\circ, 50.3^\circ, 54.8^\circ, 63.4^\circ, \text{ and } 65.2^\circ$, respectively. The average crystallite size for Cr_2O_3 -NPs was found to be 58 nm. The photo catalytic activity of green synthesized chromium oxide-NPs was also analyzed.

Keywords: Chromium Oxide nanoparticles, Green Synthesis, *Mentha Pulegium* Extract, Methyl Orange

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1. Introduction

Nanotechnology is a developing area in biomedical sciences which can be used intensively in health care sciences [1]. Nanotechnology is a rapidly expanding field that employing nanomaterial for diagnosis and treatment aims [2]. The advancement of nanomaterials is one of the most hopeful advances for the therapy of a wide range of illnesses, such as fungal and bacterial infections, as well as different types of cancer [3-4]. Nanoparticles (NPs) have a noticeable physicochemical property, including small sizes, large surface-to-volume ratios, and size-dependent optical properties, have received reasonable care in potential increased biological uses [5-6]. NPs are the building blocks of nanotechnology and nanomedicine, they have been used in different types of applications, which include diagnosis, detection, drug delivery, and therapy of various diseases, and therapy of different types of cancer [7]. For example, metal oxide NPs are used intensively in a variety of products for different objectives [8], like photocatalysis [9] and adsorption [10].

Due to their good stability, hardness, high resistivity, high melting temperature, and wide band gap of 3.4 e V, chromium oxide nanoparticles (Cr_2O_3) have gained particular among metal oxide NPs [11-12]. Cr_2O_3 NPs could be employed in materials for catalysis [13], photocatalysis [14], super capacitors [15], lithium-ion batteries [16],

sensing [17], and other biological events owing to their special properties [18]. Biocompatibility of Cr_2O_3 nanoparticles is an important factor for their employ in most biological assays [19]. Medically Cr_2O_3 NPs were notably used as potent antioxidants, anti-bacterial, anticancer, antiviral and antidiabetic etc. [19-21]. Due to the abundant uses of harmful chemicals such as starting materials and solvents, with a greater amount of heat at different pH, the Cr_2O_3 NPs synthesized by traditional chemical or physical methods exhibits a number of disadvantages. The use of such factors can bring more threatening characters, involving carcinogenicity and the toxicity of the environment, that limit the arrangement of nonmaterial in different biomedical and clinical uses [22]. Thus, the green preparations of nano particles has grown as an alternative to traditional physical and chemical procedures and has the potential to mitigate some of their damaging impacts [23]. Green synthesis is an exciting method for preparing nanoparticles since it is straight forward, cost-effective, and eco-friendly [24].

Biologically synthesized metallic NPs are cancer-fighting cytotoxic agents [25]. Compared to the synthesis of nanoparticles by bacteria and fungus, the preparations of nanoparticles with plant extracts are easy and straight forward approach for producing nanoparticles on a large scale [26]. Many researchers today are eager in green

preparations of Cr₂O₃ NPs with extracts of plants [27]. Callistemon viminalis, extracts of flower of Callistemon viminalis (Bottle Brush) plant [28], Leaf Extract of Tridax procumbens plant [29], plant extract of cactus (Opuntia ficus-indica) [30], have been employed in the synthesis of Cr₂O₃ NPs. The extensively plant *Mentha pulegium* belongs to the Lamiaceae family, and the leaves of *Mentha pulegium* are used to therapy nausea, bloating, dyspepsia, diarrhea, gastrointestinal ailments, headaches, cough and influenza, and tuberculosis [31–33]. These plants have phytochemicals compounds, which is a useful factor for the reduction of metal ions. According to this study, the extraction of different plant was used for the green preparation of the Cr₂O₃-NPs, including Hyphaene baica [34], Cannabis sativa [35], the Manihot esculenta [36], Zainab, Ahmad S and co others [37] described green preparation of Cr₂O₃-NPs using extract of Erythrophleum guineense plant.

Dyes are an abundant class of colored organic compounds that represent an increasing environmental danger. During dye production and textile manufacturing processes a large amount of wastewater containing dyestuffs with intensive color and toxicity can be introduced into aquatic systems [38]. In our world today, there is an increasing need in many countries to access clean and safe water both for domestic use and other purposes [39]. The demand for clean water surpasses its supply. This means that water emitted out of industries should be recycled, purified and later used for other purposes. The pollution that accompanies the dye industry is of great significance and importance. More than 15% of the total world production of dyes are lost during the dyeing process and is released as a source of textile pollution [40]. Wastewater is known to contain considerable amounts of non-fixed dyes, especially azo dyes, and huge amounts of inorganic salts.

It is well known that some azo dyes and degradation products such as aromatic amines are carcinogenic [41]. Due to the large degree of aromatics present in dye molecules and the stability of modern dyes, conventional biological treatment methods are ineffective for decolorization and degradation [42]. This has led to study of other methods and recent studies have demonstrated that photocatalytic degradation can be used to destroy dye compounds using metals oxides nanoparticles such as Cr₂O₃ and UV light irradiation. This technique has developed a useful process for the reduction of water pollution caused by dyeing compounds. Photocatalytic degradation of methyl orange dye using Cr-doped ZnS nanoparticles under visible radiation was studied [43].

In present studies green synthesis of chromium oxide nanoparticles (Cr₂O₃ NPs) using leaf extract of *Mentha pulegium* plant employed and characterized by UV/Vis spectroscopy, Fourier Transform Infra-Red (FTIR), EDX, SEM, and XRD techniques and photo catalytic degradation activity of Cr₂O₃-NPs were also analyzed.

2. Materials and Methods

The present research work was performed in chemistry laboratory, Department of Chemistry, Faculty of Science, Albaha University. All chemicals used were of analytical grade and commercially available. In this research work, chemicals come from Merck (Darmstadt, Germany) and Sigma Chemicals Co. (St. Louis, MS, USA). Chemicals used in this work are 0.5 Molar of CrCl₃.6H₂O, 1M sodium

hydroxide (NaOH, > 97%), and demonized water (DI). 25 ppm solutions of methyl orange (C₁₄H₁₄N₃SO₃Na).

2.1. Plant Material Collection

Mentha pulegium Leaf plant was collected from the local market of the Alaqiq, in Albaha Province, Saudi Arabia. Its identification was made by the Department of Botany, Albaha University, Alaqiq, Saudi Arabia.

2.2. Preparation of Plant Extract

A total of 20 g of the fresh leaves of *Mentha pulegium* were taken. The leaves of the *Mentha pulegium* were washed with deionized water (DI) and were dried in an oven at 80 C°. The dried leaves of *Mentha pulegium* were crushed to fine particles with a mortar and pestle. The crushed fine leaves were got mixed in 150 cm³ of the deionized water, and heated with stirring at 65 C° for half an hour until the solution color changed to dark red, then filtered through the Whatman filter paper no.1, and maintained at 4 C° for further studied.

2.3. Green Synthesis of Chromium Oxide (Cr₂O₃) Nanoparticles

For the green synthesis of the Cr₂O₃ nanoparticles, 40 ml of 0.5 Molar of the CrCl₃.6H₂O was added drop wise to 20 ml of the *Mentha pulegium* extract in a 500 ml beaker and the 1M NaOH was also added drop wise with continuous stirring for 2 hours at temperature of 80 C°. A sharp change in color from red to green was observed when the solution of chromium chloride was added, due to surface Plasmon resonance indicating the formation of the required the Cr₂O₃ nanoparticles. After that the solution was centrifuged, then the precipitate was washed many times with deionized water (DI) and ethanol to remove the impurities, and then was dried in the oven for two hours. It is finally calcined in the Muffle furnace at 500 C° for 3 hours and green colored the Cr₂O₃ nanoparticles were obtained. Finally, obtained nanoparticles were stored in an air-tight container for characterization and photochemical applications. The experimental stages involved in the Cr₂O₃-NPS preparation, and its characterization and application have been shown in Figure 1.

2.4. Photocatalytic activity Studies

The effect of prepared Chromium oxide nanoparticles on the photocatalytic degradation of Methyl orange was investigated and studied. The photo degradation properties of prepared samples were evaluated in the presence of UV-VIS light to assess their ability to degrade methyl orange dye. The degradation of the methyl orange dye was tracked by observing the gradual decline in the absorption peak in the UV-VIS absorption spectra as the reaction time increased. This absorption peak corresponds to methyl orange and is indicative of its concentration in the solution. To the 20 ml of 25 ppm of several dye solutions of the Methyl Orange, 100 mg of our Cr₂O₃ NPs as catalysts were added, and the degradation was conducted under visible light at room temperature at an interval of 20 min for 2 hrs. In each time shaking is performed after which centrifugation is done for at least 10 minutes. The clear suspension was filtered, and the solution then stored in the small bottles. The absorbance of dye solutions for each time measured using a spectrophotometer. The absorbance value of the methyl orange then used to calculate the extinction coefficient

derived from the Lambert-Beer law, and then amount of dye adsorbed in (mol/g) was obtained.

3. Results and discussion

3.1. Characterization

Mentha pulegium leaf extract employed as capping and a reducing agent for preparation of Cr₂O₃ nanoparticles. Cr₂O₃ nanoparticles preparation was checked visibly by identifying the change of color during addition of metal salt solution in extract of leaf. Formation of our nanoparticles can be checked by change of color of reaction from red to black. This transition of color happened as a result of resonance of surface plasmon (SPR) phenomenon on nanoparticle's surface [11-28]. *Mentha pulegium* leaf extract is a rich source of biological active phytochemicals such as, polyphenols, flavonoids, terpenoids, alkaloids, etc. [44]. These phytochemicals can play as ligands and chelating with different types of metal ions to stabilize and reduce the ions to nano shape [45-46]. Chromium Chloride (CrCl₃.6H₂O), have free movement upon dissolved in water. Freely moving Cr³⁺ ions are electron-deficient centers which can be attracted towards phytochemicals polyphenols, etc.). Chelate complex formed between metal ions and plant's phytochemicals upon transferring electrons (donor-acceptor process) from oxygen to Cr³⁺ [46-54]. Oxidation of polyphenols, flavonoids, etc., takes place and changes these molecules into keto-form. Prior to this process, Cr³⁺ is reduced to zero valent Cr⁰ and spontaneously is stabilized by other plant's phytochemicals (alkaloids, flavonoids, polyphenols, etc.) found in their area. They are easily oxidized and changed into Cr₂O₃ nanoparticles capped with phytochemicals of *Mentha pulegium* leaf extract during air-drying and calcination [45-47]. Same green synthesis mechanism also reported to synthesize Cr₂O₃ nanoparticles using Erythrophleum guineense plant extract [37], ZnO, zinc oxide-silver, Fe₃O₄, and magnetite (Fe₃O₄) employing different plants [45-48].

3.1.2. Ultraviolet/Visible Study

Ultraviolet /Visible spectroscopy utilized to identify preparation of Cr₂O₃ nanoparticles. Fig.2 explains the spectra of Ultraviolet /Visible of Cr₂O₃ nanoparticles prepared from *Mentha pulegium* extract. There was a prominent peak in the visible spectrum at around 462 nm. Presence of these peaks explains that the extract of plant merely decreased amount of chromium salt into chromium oxide nanoparticles. Jaswal et al.,[49], also documented Ultraviolet /Visible spectrum for Cr₂O₃ nanoparticles prepared by precipitation procedure from their precursor.

3.1.3 Elemental composition analysis

Energy Dispersive X-ray (EDX) analysis is utilized to identify which elements are in a compound or material. Fig.3 explains the EDX spectrum of chromium oxide nanoparticles prepared from the extract of *Mentha pulegium*, and shows that the elemental composition was found to be 44.41% oxygen and 32.57% chromium in our synthesized Cr₂O₃-NPs, and confirm the presence of chromium and oxygen in our prepared Cr₂O₃-NPs.

3.1.4. Scanning Electron Microscope (SEM) Analysis

Fig.4 shows scanning electron microscope images of the green prepared chromium oxide nanoparticles from Babiker et al., 2024

the extract of *Mentha pulegium*, at two different magnifications. It is clear from lower magnification image1(1 μm, x 5,000) that prepared nanoparticles are clusters in a large-scale area and have approximately uniform morphologies. Image2(1μm, x20, 000) shows a higher magnification image of such morphologies. General structure of prepared Cr₂O₃-NPs shows little variation. Scanning Electron Microscope imaged displayed that prepared nanoparticles agglomerated with each other, resulting in irregular, round morphology, may be due to green synthesis technique adopted. Primary particles of Cr₂O₃-NPs are in a nanostructure. Cr₂O₃ nanoparticles size ranged from 1μm -100 nm.

3.1.5 Infrared Spectroscopy

The characterization of prepared chromium oxide nanoparticles was done by using the Perkin Elmer Fourier Transform Infra-Red (FTIR) spectrometer. The Fourier Transform Infra-Red spectra of the extract of *Mentha pulegium* plant and chromium oxide nanoparticles are presented in Fig.5 (a, b) respectively. The FTIR response for the *Mentha pulegium* plant extract confirms the presence of, phenolic groups, polyols, alkaloids, terpenoids, flavonoids and steroids. The broad strong peaks in Fig.5(a) at 3273 cm⁻¹ is due to the presence of alcoholic or phenolic-OH groups, the absence of this peak in fig.5(b) compared to Fig.5(a) confirms that the organic molecules have been involved in the formation of chromium oxide nanoparticles. Peaks at 2159 cm⁻¹ in both spectra indicate the stretching of alkynes. The peak at 2031cm⁻¹ and 1977 cm⁻¹ in both spectra, represents aromatic ring and C=C alkene bond of flavonoids and terpenoids. The peaks at 1635 cm⁻¹ in fig.5(a) correspond to C=O stretching of carbonyls, the absence of this peak in fig.5(b) compared to Fig.5(a) confirms the involvement of this group in the formation of chromium oxide nanoparticles. The sharp peak at 610 cm⁻¹ in fig.5(b) compared to fig.5(a) is due to the presence of Cr-O bonding which explains the presence of Chromium oxide. The presence of the above-mentioned groups indicates explains that phyto constituents can possibly increase the stabilization of chromium oxide nanoparticles.

3.1.6 X-Ray diffraction Analysis

The crystallinity of the green prepared Cr₂O₃ nanoparticles was determined by X-ray diffraction studying, and the results are presented in Fig.6. The X-ray diffraction spectrum of synthesized the Cr₂O₃ nanoparticles revealed eight different Bragg's diffraction peaks at 2θ= 24.3°, 33.5°, 36.2°, 41.5°, 50.3°, 54.8°, 63.4°, and 65.2°, respectively. The peaks of diffraction of Cr₂O₃ nanoparticles are exact-matched with the Joint Committee on Powder Diffraction Standards (JCPDS) 38-1479 [50-51]. Thus, explaining the crystalline character of our prepared Cr₂O₃-NPs, Tsuzuki and McCormick, et al, also reported the same results of Cr₂O₃ nanoparticles by mechanochemical processing,[52]. X-ray diffraction analysis is a convenient procedure for determination of the mean size of nano crystallites in nano-crystalline bulk materials. From the well-known Scherrer formula the average crystallite size, $D = k \lambda / (\beta \cos\theta)$ where λ is the X-ray wavelength in nanometer (nm), ($\lambda = 1.5406$ Å), β is width of the peak of diffraction profile at half maximum height in radians, resulting from small crystallite size, and K is a constant related to crystallite shape,

normally taken as 0.9. Value of β in 2θ axis of the diffraction profile must be in the radians. The Scherrer equation, $d = 0.9\lambda / (\beta \cos \theta)$ used to estimate grain average sizes of crystallites. Calculated average of crystallite size for the Cr_2O_3 -NPs evaluated by sharpest peak at $2\theta = 33.50^\circ$ which found to be 58 nm.

3.1.7. Methyl Orange Degradation

Initially, influence of amount of adsorbent on photocatalytic degradation of Methyl orange dye was investigated using Cr_2O_3 -Nps. To determine amount of dye adsorbed (in mol/g), absorbance values of methyl orange used, from which extinction coefficient derived from Lambert- Beer law calculated, in 100 mg of Cr_2O_3 -Nps, at an interval of 20 min for 2 hrs, plot of C/C_0 versus time is shown below in fig.7, where C is concentration of methyl

orange at various time intervals and C_0 initial concentration of methyl orange. Graph explains a systematic decrease in dye concentrations with time when dye Methyl Orange is irradiated with UV light in presence of Cr_2O_3 nanoparticles. A linear fit plot also obtained when $\ln(C/C_0)$ is plotted versus time, fig.8, explains that reaction followed pseudo first order rate kinetic. Experiment conducted without Cr_2O_3 nanoparticles catalyst showed no change in concentration. This proves that photo catalytic degradation using Cr_2O_3 nanoparticles is a successful means of dye removal from water. Photo-degradation of four different organic dyes in green synthesized chromium nanoparticles studied by G. Vanitha and co-authors [53], and they found same trend of degradation.

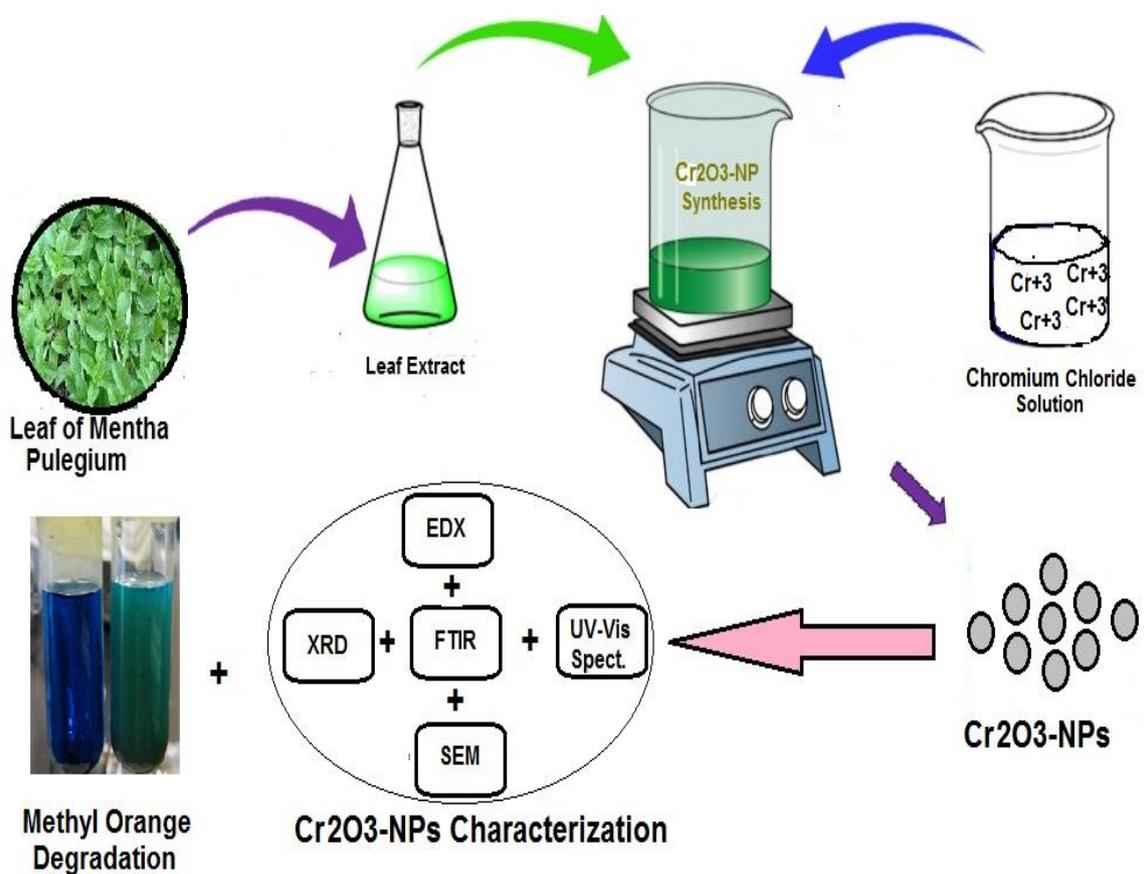


Figure 1. Representation of the experimental stages involved in the Cr_2O_3 -NPS preparation and its characterization and Application

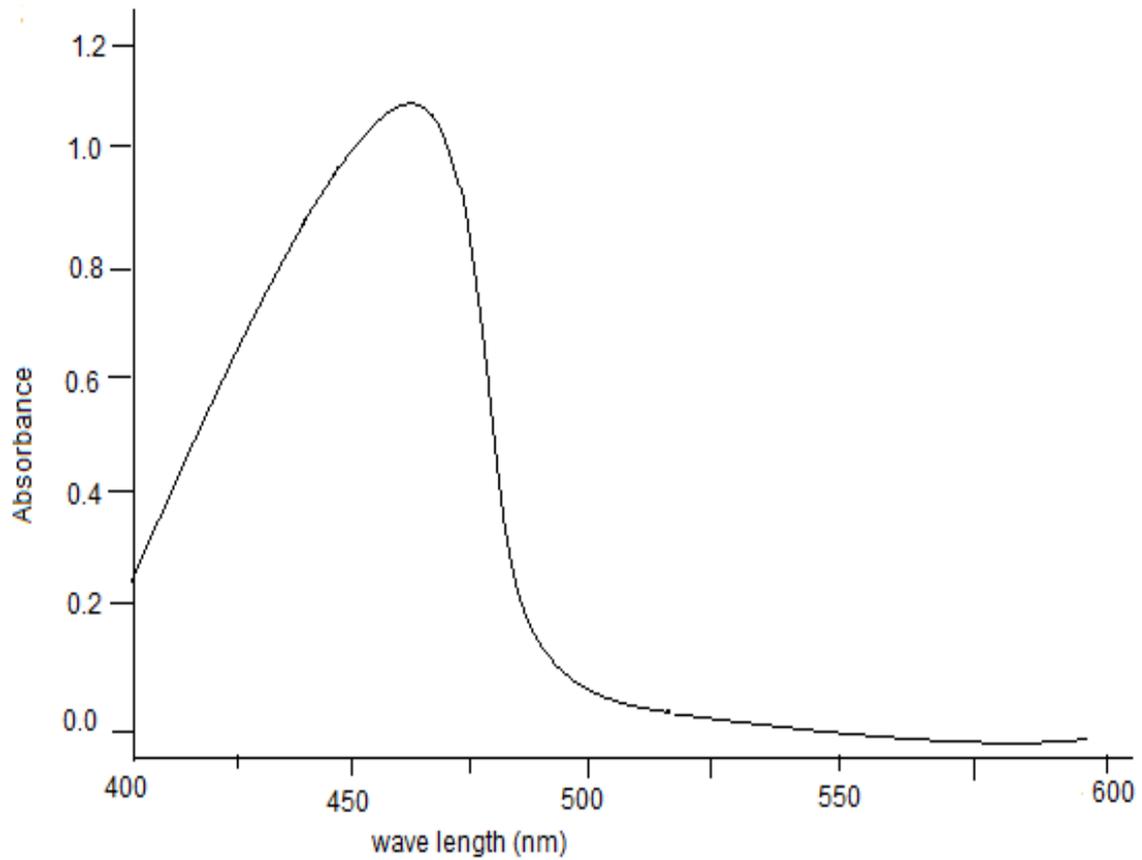


Figure.2. UV-Vis Spectra of Green Synthesized Chromium Oxide nanoparticles

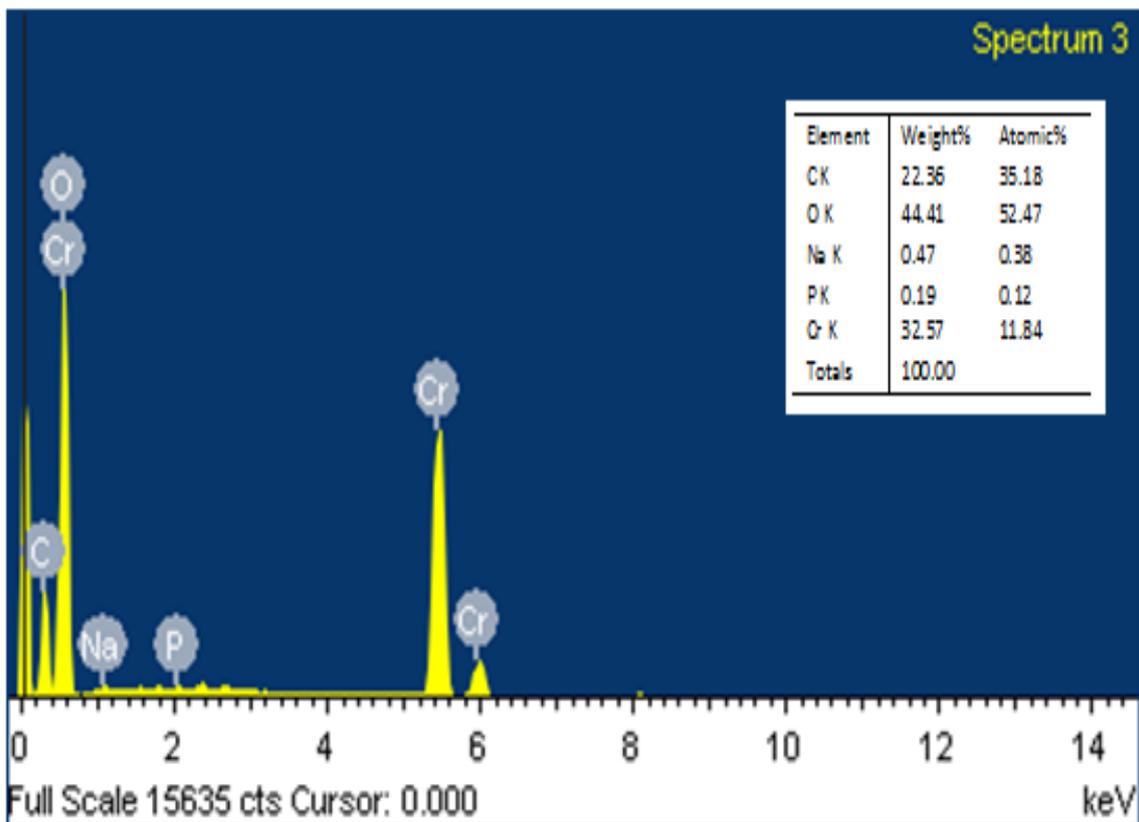


Figure. 3. EDX Spectra of Green Synthesized Chromium Oxide nanoparticles

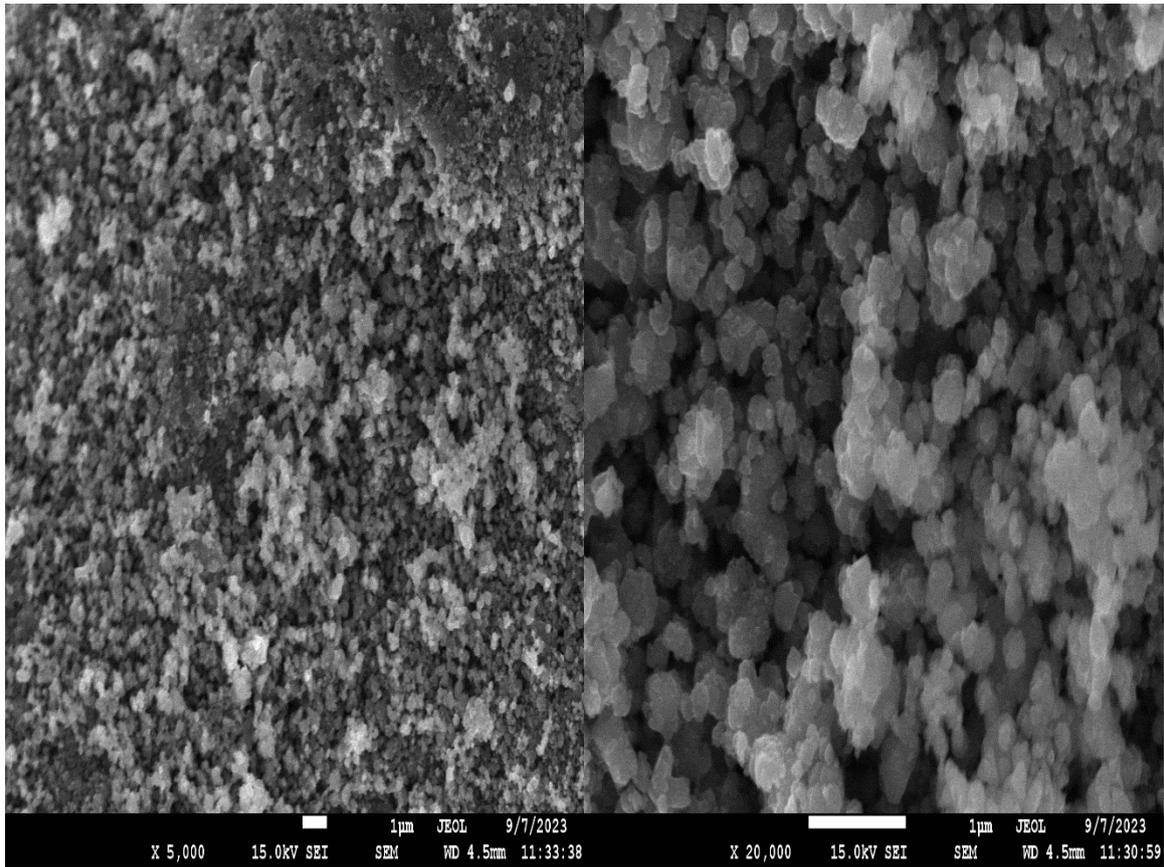


Figure.4. SEM Images of Green Synthesized Chromium Oxide nanoparticles.

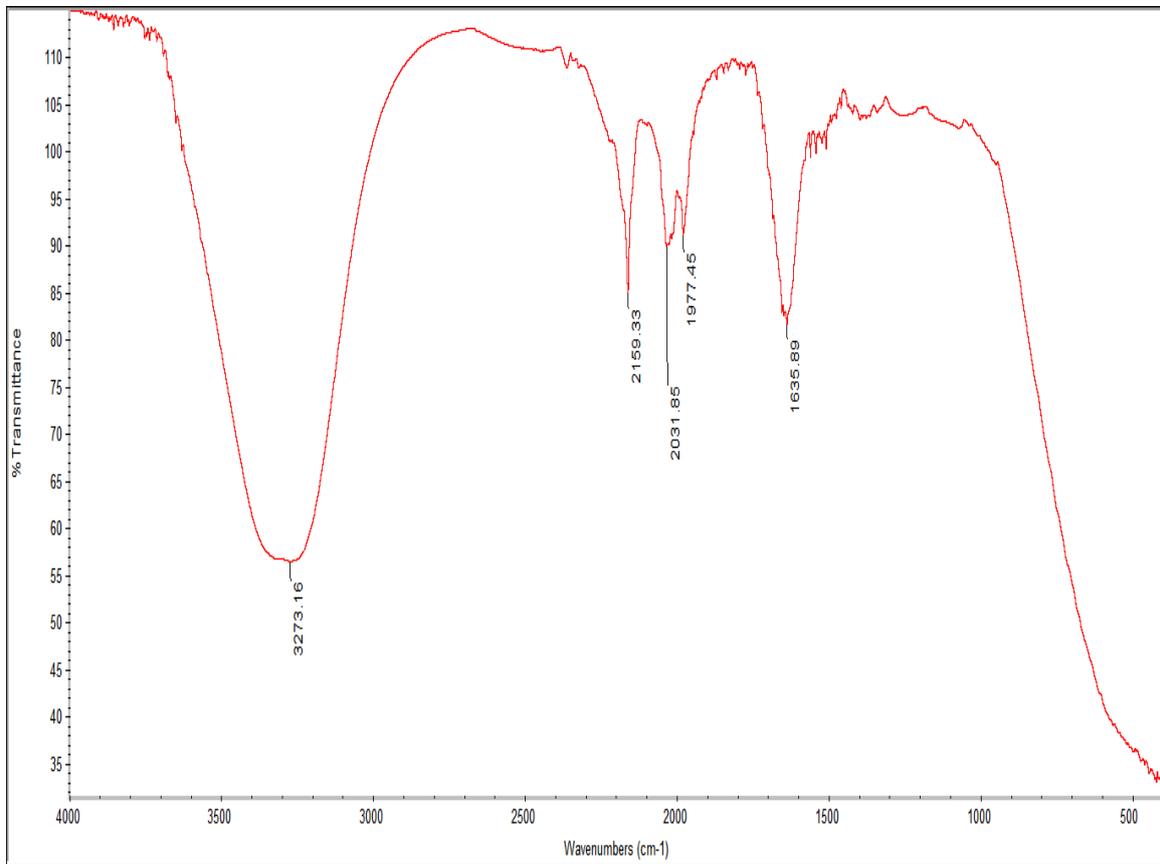


Figure 5. (a) IR spectra of *Mentha Pulegium* Plant Extract

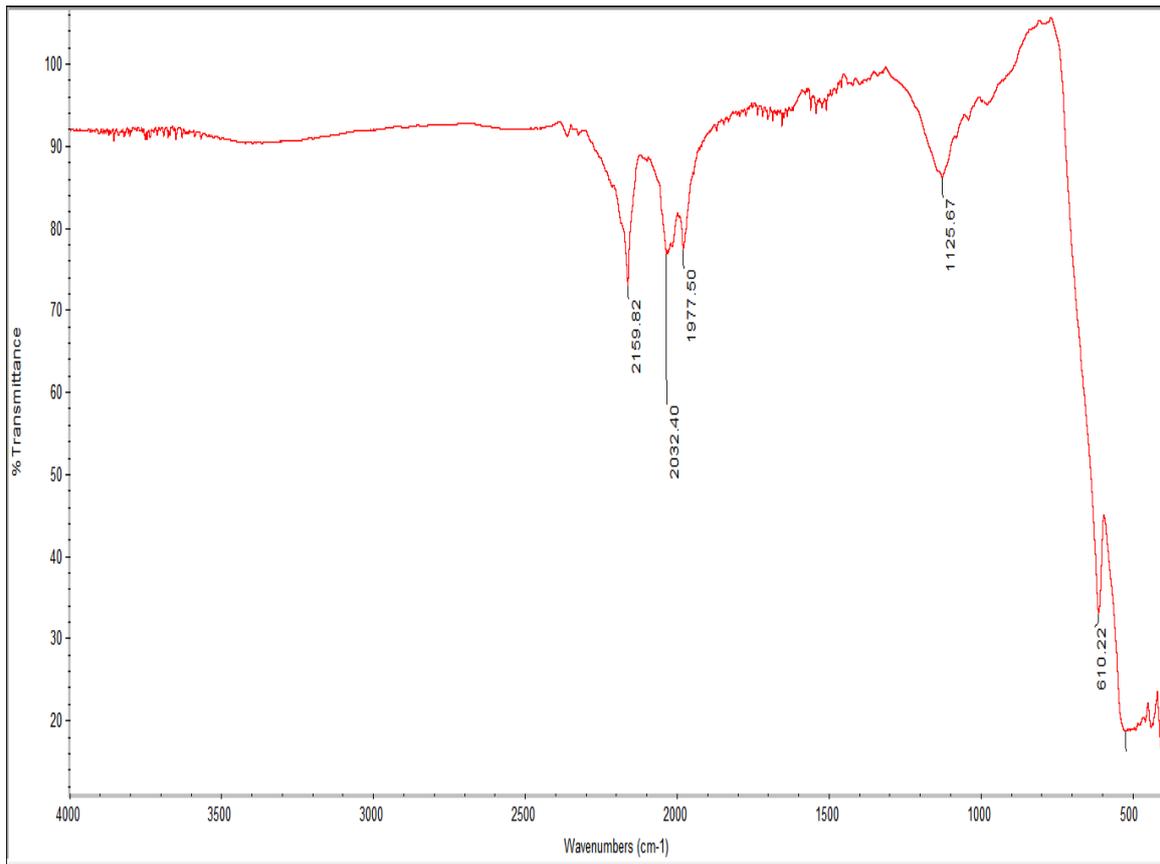


Figure. 5.(b) IR spectra of Green Synthesized Cr₂O₃ nanoparticles

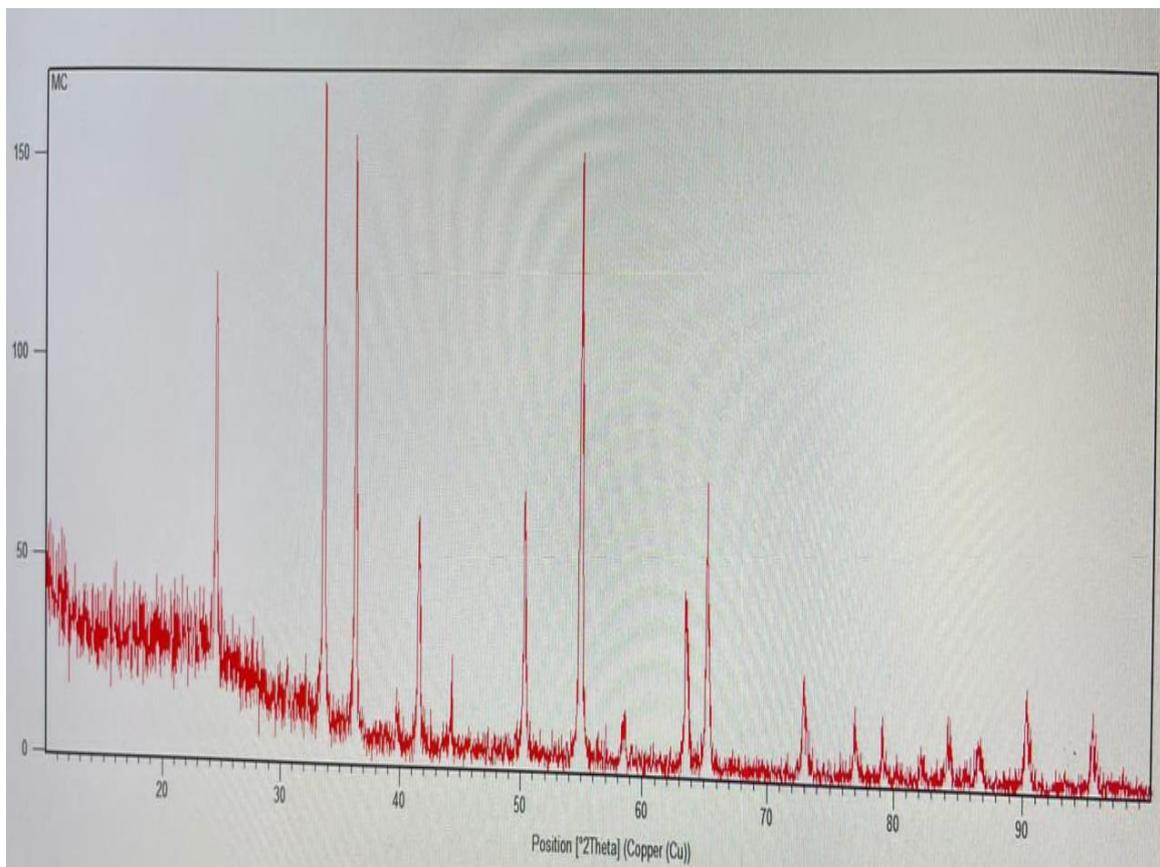


Figure.6. XRD Spectrum of the Green Synthesized Chromium Oxide nanoparticles.

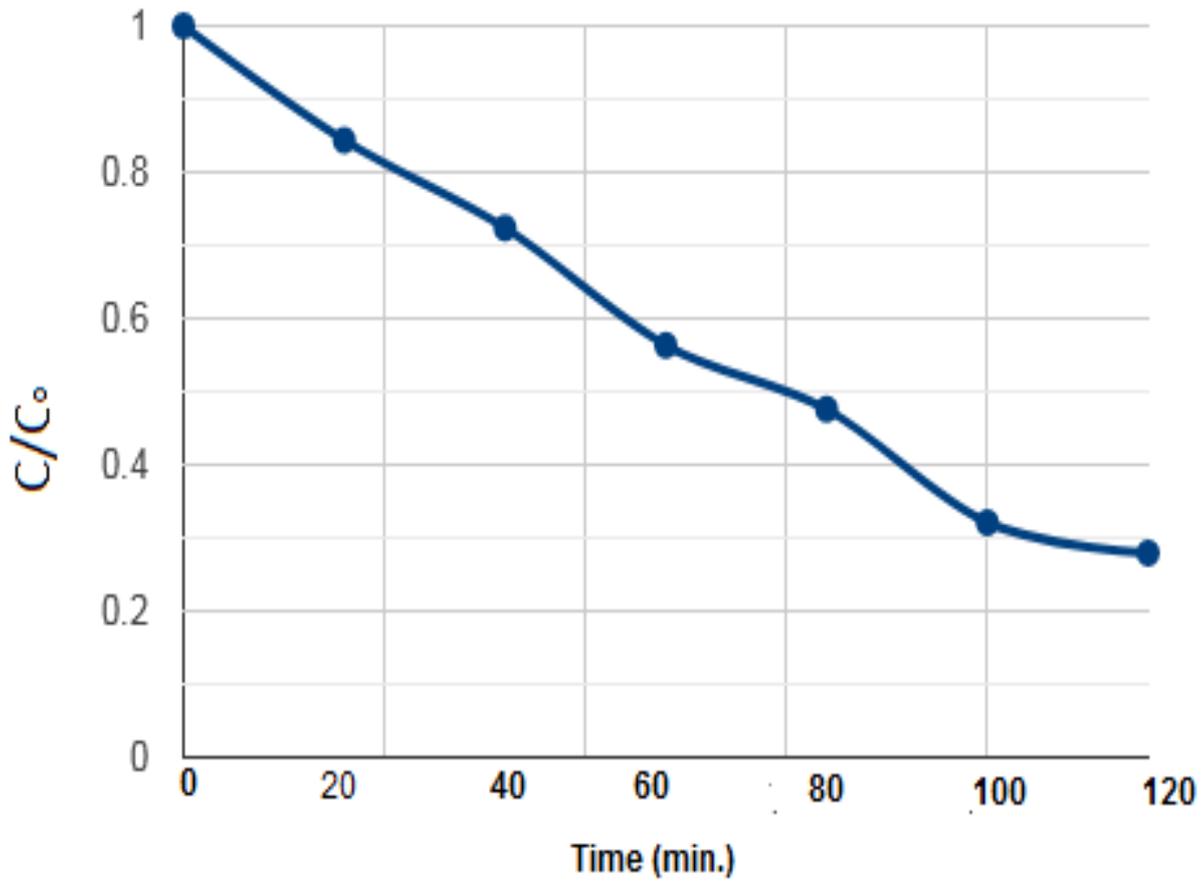


Figure 7. Graph Explaining Photo catalytic degrading of methyl orange using Cr_2O_3 nanoparticles.

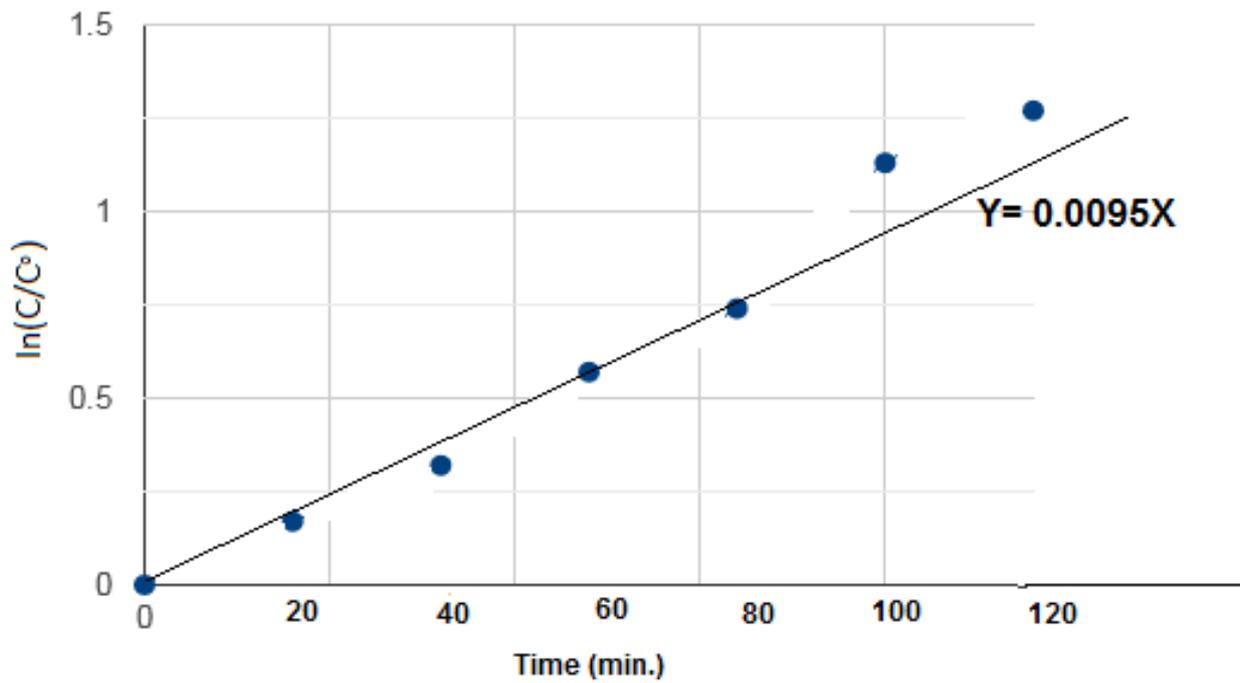


Figure 8. Plot of $\ln(C/C_0)$ Vs. Time for the degradation of methyl orange on Cr_2O_3 nanoparticles.

4. Conclusions

In this work, Cr₂O₃ nanoparticles have been successfully green synthesized using the leaf extract of *Mentha pulegium* plant as capping and a reducing agent. The green prepared Cr₂O₃ nanoparticles have been characterized by Scanning Electron Microscope (SEM), X-Ray Diffraction (XRD), EDX, FTIR, and Ultra Violet /Visible spectroscopy. The particle average size of the prepared Chromium nanoparticles were found to be about 58 nm. Green prepared Cr₂O₃ nanoparticles displayed excellent photocatalytic degradation of methyl orange dye from water. Green prepared Cr₂O₃ nanoparticles enhanced photocatalytic activities may be due to photochemical properties of phytomolecules on their surface. Current study gives fresh insight into development and production of plant conducted effective nanostructure with coming uses in waste-water treatment.

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Conflict of Interest

The authors declare that there is no conflict of interests regarding the publication of this article.

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