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Eco-Environmentally Friendly Green Synthesis and Characterization of Aluminum Oxide Nanoparticles Using Leaf Extract of *Mentha Pulegium*

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Abstract

In recent years, the nanoparticle synthesis methods conducted by plants replaced the traditional chemical procedures of nanoparticles preparations. This study demonstrates the environmental kindly preparations of aluminum oxide nano particles (Al_2O_3-NPs) using leaf extract of *Mentha pulegium* plant. The obtained Al_2O_3-NPs characterized by Ultra Violet /Visible spectroscopy, Fourier Transform Infrared (FTIR), Scanning Electron Microscopy (SEM), Energy-Dispersive X-ray (EDX) and X-ray diffraction (XRD) studies. The presence of the sharp peak at 262 nm in the Ultra Violet /Visible spectrum and the color changed by surface plasma resonance explains that the Al_2O_3-NPs formed. The Energy-Dispersive X-ray (EDX) spectrum of Al_2O_3 nanoparticles reported the elemental composition of our sample, which found to consist of 30.49% Al and 56.28% O, confirming the high purity of the Al_2O_3-NPs powder.FTIR analysis explains that bioreduction of Al^{+3} ions and nanoparticle stabilization probably occurred by interactions between the biofunctional groups of *Mentha pulegium* extract and aluminum. The Scanning Electron Microscopy (SEM) analysis reported loosely agglomerated nanoparticles with each other, resulting inrough, sticky, an irregular and porous morphology shape. In addition, X-ray diffraction (XRD) studies suggested their crystalline feature by the characteristic peaks at 2θ= 21.2°, 36.5°, 46.3°, 61° and 66.5°, corresponds to the lattice planes (220), (311),(400), (511) and (440) respectively. The average crystallite size for Al_2O_3-NPswas found to be 35 nm. It is found that Al_2O_3-NPs prepared by leaf extract are stable. This procedure of preparation is less toxic and cost of preparation is low as compared to other chemical and physical procedures.

Keywords: Aluminum oxide nanoparticles, Green Synthesis, Mentha Extract, Characterization.

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1. Introduction

Nanotechnology is developing area in biomedical sciences which can be used intensively in health care sciences [1]. Nanotechnology is a rapid extending field that employing nanomaterials for treatment and diagnosis aims [2]. The advancement of nanomaterials is most one hopeful advances for the therapy of a variety of illnesses, such as fungal and bacterial infections, as well as different types of cancer [3-4]. Nanoparticles (NPs) have a noticeable physicochemical properties, 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, used in a different type of applications, which includes 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 photo catalysis [9] and adsorption [10].

The green preparations of nanoparticles has grown-up as an alternative to traditional chemical and physical procedures and has the potential to mitigate some of their damaging impacts [11]. Green preparation is an exciting procedure for preparing nanoparticles since it is successfully, an environmentally friendly and have low cost [12]. Biological prepared metallic NPs are cancer-fighting cytotoxic agents [13]. Compared to the preparation ofnanoparticles by fungus and bacteria, the preparation ofnanoparticles using extracts of plant is easy andstraight forward approach for producing nanoparticles on a large scale [14]. Prior to this study, Several studies used extracts of plant for the green predation of Al_2O_3- NPs, for instance, the extract of leaf of *Muntingia calabura*[15], the extract of leaf of Rosa [16], the extract *of Citrus aurantium* peel [17], and the extract of grapefruit [18], and *Phoenix dactylifera* seed extract[19]. The 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 [20-22]. These plants have phytochemicals compounds, which is a useful factor for reducing metal ions. The extract of *Mentha pulegium* was mediated for the green preparations of NPs, such as silver NPs [23] Ag/Fe_3O_4nanocomposite[24], zinc oxide NPs [25], iron oxide NPs [26], and copper oxide NPs[27].Green preparations of Al_2O_3-NPs is depended on either extract of plant or microorganisms in their reduction and stabilization process. The biological preparation of Al_2O_3-NPs take place by plant extracts, which is friendly environmentally and allows the regulation of preparations of NPs with fine-defined form and size [18-28]. In the present studies green environmentally friendly preparation of nanoparticles of aluminum oxide (Al_2O_3-NPs) using extract of leaf of *Mentha pulegium* plant were employed and characterized by Ultra Violet /Visible spectroscopy. Fourier Transform Infrared (FTIR), Scanning Electron Microscopy (SEM), Energy-Dispersive X-ray (EDX) and X-ray diffraction (XRD) techniques.

2. Materials and Methods

The present research work was performed in the chemistry laboratory, Department of Chemistry, Faculty of Science, Albaha University. All the chemicals used were of analytical grade and available commercially. In this research work, the chemicals came from Merck (Germany, Darmstadt), and Sigma Chemicals Co. (St. Louis, MS, USA). The chemicals used in this work are 0.5 Molar of $AlCl_3.6H_2O$, 1M NaOH and deionized water (DI).

2.1. Plant Material Collection

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

2.2. Preparation of Plant Extract

A total of 10 g of fresh leaves of *Mentha pulegium* were taken. The leaves of *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 cm^3 of deionized water, and heated with stirring at 65 C° for half an hour until the solution color changed to dark red, then filtered through Whatman filter paper no.1, and maintained at 4 C° for further studied.

2.3. Green Synthesis of Aluminum Oxide (Al_2O_3) Nanoparticles

For the green synthesis of Al_2O_3 nanoparticles, 50 ml of 0.5 Molar of $AlCl_3.6H_2O$ was added drop wise to 20 ml of *Mentha pulegium* extract in a 500 ml beaker and 1M NaOH was also added drop wise with continuous stirring for 2 hours at temperature of 80ºC. A sharp change in color from red to dark-white colored was observed when the solution of aluminum chloride was added, due to surface plasmon resonance indicating the formation of the required Al_2O_3 nanoparticles. The solution then centrifuged; the precipitate washed several times with deionized water and ethanol to remove the impurities and dried in the oven for two hours. It finally calcined in the Muffle furnace at 500℃ for 3 hours and white powder colored Al_2O_3 nanoparticles obtained. Finally, the obtained nanoparticles were stored in an airtight container for characterization. Schematic production processes for the These phytomolecules can play as ligands and chelating employed and characterized by Ultra Violet /Visible Al_2O_3nanoparticle and its characterization have been shown in Fig.1.

3. Results and discussion

3.1. Characterization

Mentha pulegium leaf extract was employed a as capping and a reducing agent for the preparation of Al_2O_3 nanoparticles. The Al_2O_3 nanoparticles preparation checked visibly by identifying the change of the color during the addition of metal salt solution in extract of the leaf. The formation of our nanoparticles can checked by the change of the color of reaction from red to white. This transition of the color happened because of the resonance of surface Plasmon (SPR) phenomenon on the nanoparticle's surface. *Mentha pulegium* leaf extract is a rich source of biological active phytomolecules such as, polyphenols, flavonoids, terpenoids, alkaloids, etc[29].With different type of metal ions to stabilize and reduce the ions to nano shape [30]. The aluminum Chloride ($AlCl_3.6H_2O$), have free moving ion upon dissolved in water. The freely moving Al^{3+} ions due to electron-deficiency centre which can be attracted towards the phytomolecules polyphenols, etc.).The chelate complex was formed between plant's phytomolecules and metal ions, upon transferring electrons (donor—acceptor process) from oxygen to Al^{3+}[30-31]. The polyphenols, flavonoids, etc, can oxidize and change into keto-form. Prior to this process, Al^{3+} is reduced to zero valent Al^0 specie and stabilized spontaneously by the plant's phytomolecules (flavonoids, alkaloids, polyphenols, etc.) found in their area. They easily oxidized and changed into Al_2O_3-NPS capped with phytomolecules of *Mentha pulegium* leaf extract during calcinations and air-drying [30-32]. The same green synthesis mechanism was also reported to synthesize Al_2O_3-NPs by using leaf extract of the neem[33].

3.1.2. Ultraviolet/Visible Study

Ultraviolet/Visible spectroscopy utilized to identify the preparation of Al_2O_3-NPs. Fig.2, explains the spectra of ultraviolet/Visible of Al_2O_3-NPs prepared from *Mentha pulegium* extract. There was a prominent peak in the visible spectrum at around 262 nm. The position of these peaks explains that the extract of the plant merely decreased the amount of aluminum salt into aluminum-oxide nanoparticles. The position of absorption of Al_2O_3-NPs is depended on the size of particle and shape [34]. We can observe from the literature, that the position of absorption depends on the sources utilized for the preparation of Al_2O_3-NPs, for example, extract of macroalgae *Sargassum ilicifolium* (227 nm) [35], extract *of Citrus aurantium* L (322 nm) [17], and the waste of Al foil (237 nm) [34].

3.1.3 Elemental composition analysis

Energy-Dispersive X-ray (EDX) analysis utilized to identify which elements are in a compound or material. Fig.3, explains the EDX spectrum of aluminum oxide nanoparticles prepared from the extract of *Mentha pulegium*, and shows that the elemental composition found to be 56.28% oxygen and 30.49% aluminum in our synthesized Al_2O_3-NPs.

3.1.4. Scanning Electron Microscope (SEM) Analysis

Figure 4. Shows the Scanning Electron Microscope images of the green prepared aluminum oxide nanoparticles from extract of *Mentha pulegium*, at two different magnification.

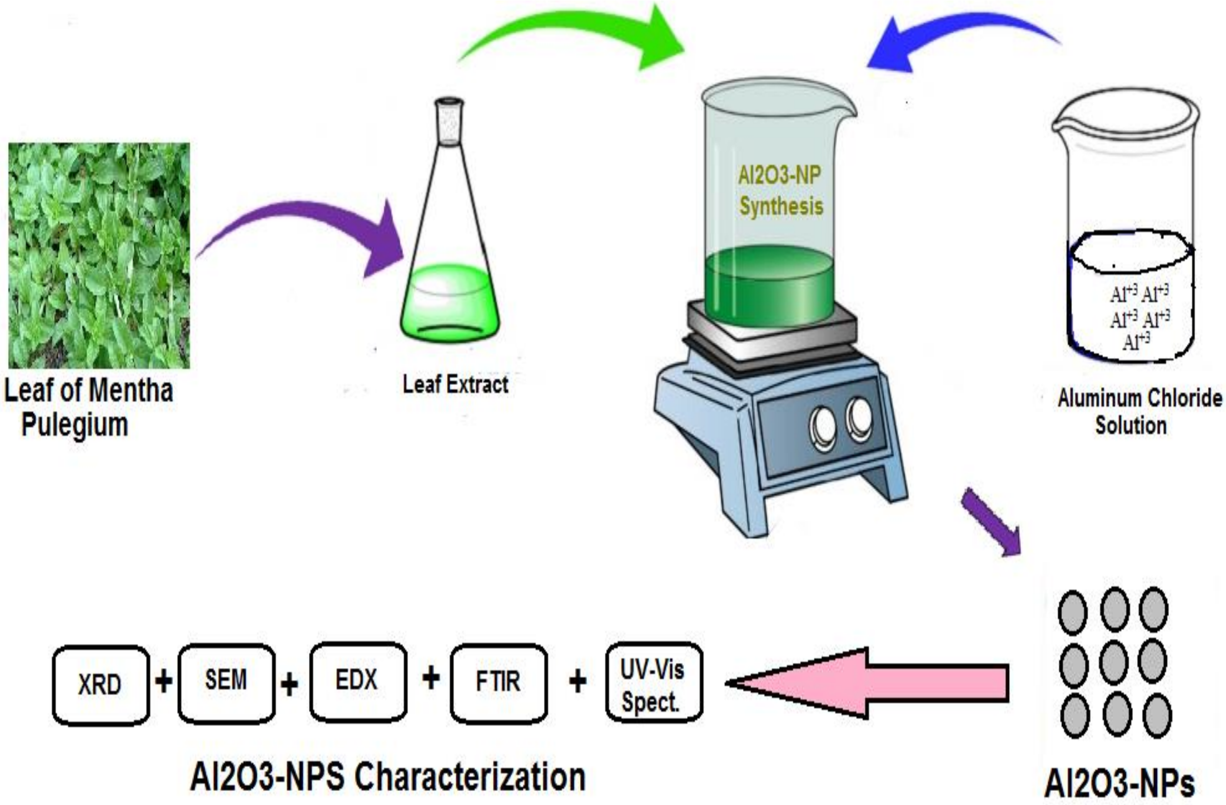

Figure 1. Representation of the experimental stages involved in the Al_2O_3-NPS preparation and characterization

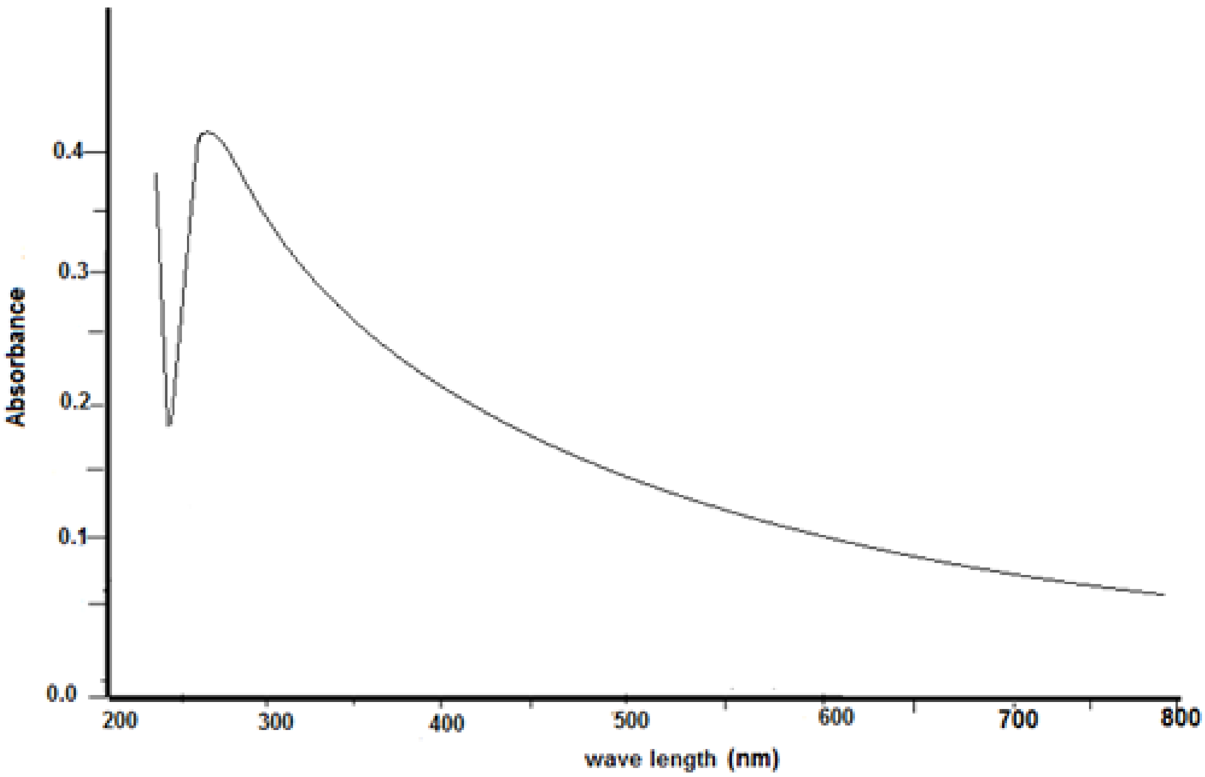

Figure 2. Ultraviolet/Visible Spectra of Green Synthesized Al_2O_3-NPs

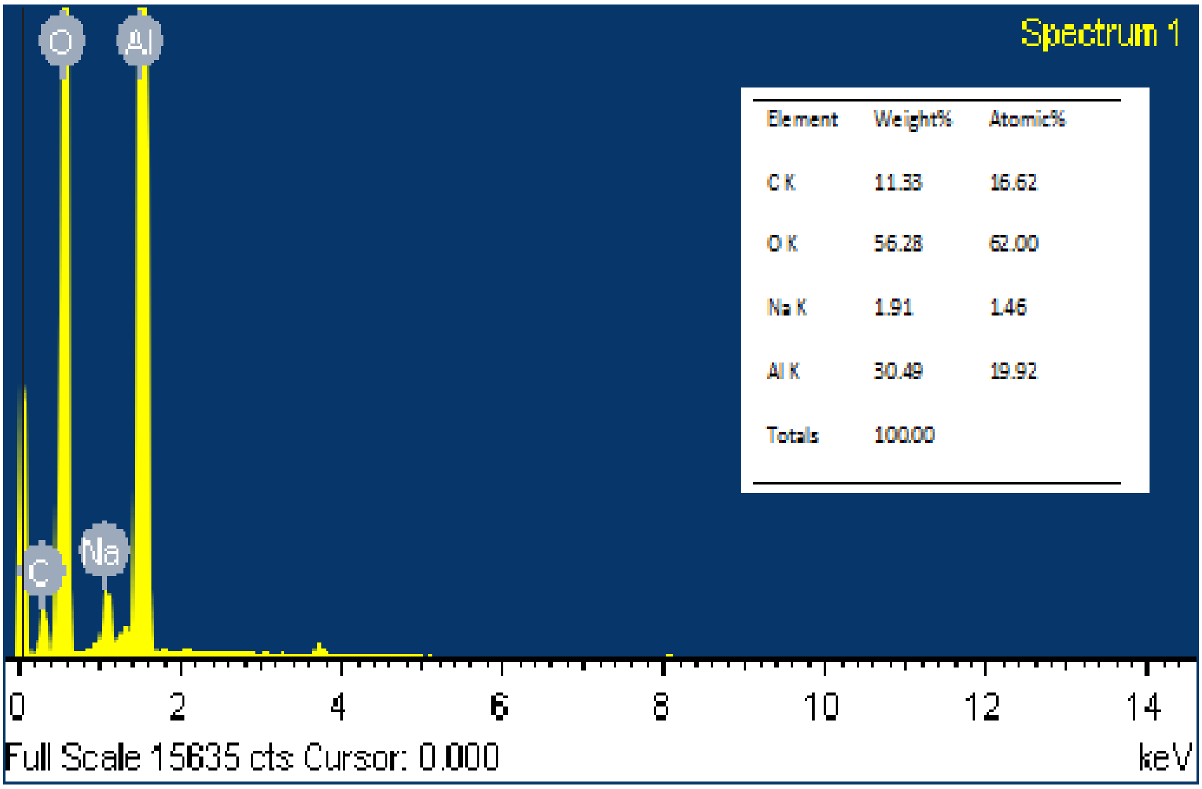

Figure 3. Energy-Dispersive X-ray (EDX) Spectra of Green Synthesized Al_2O_3-NPs

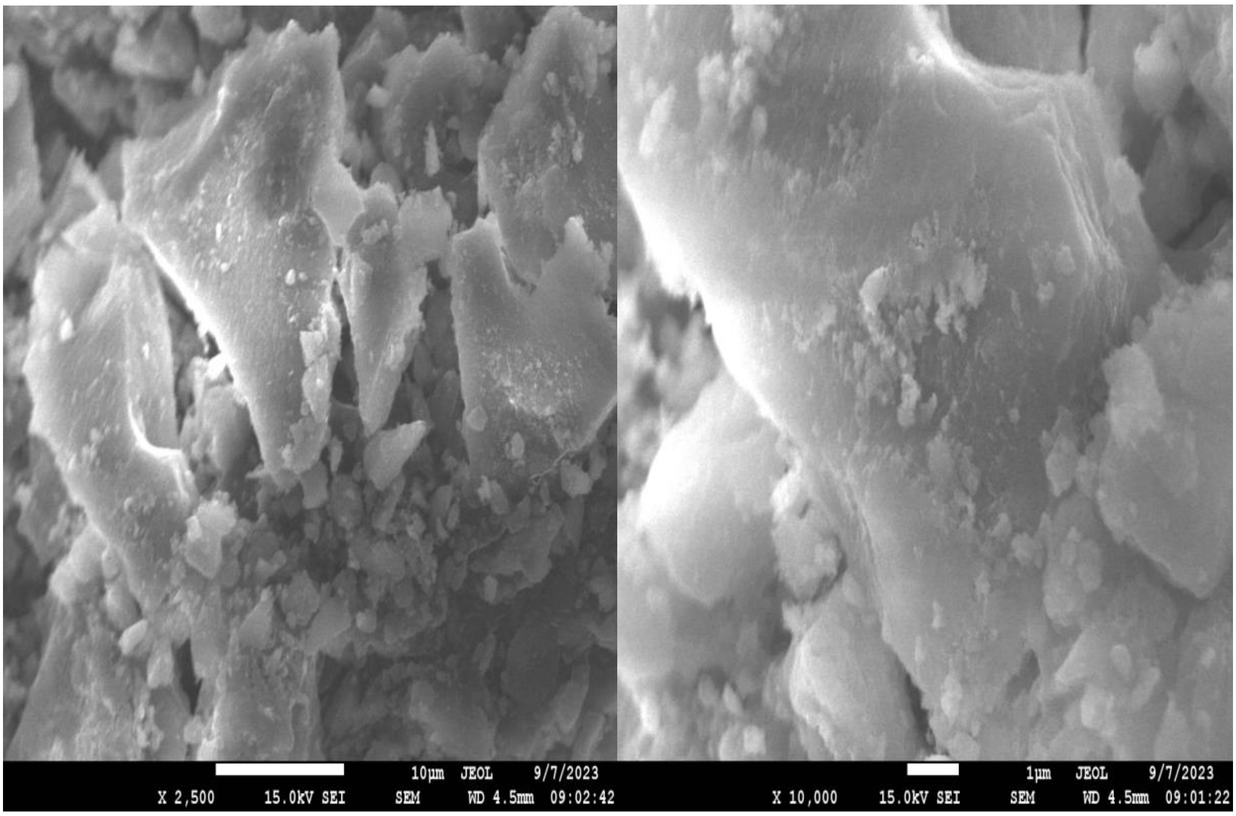

Figure 4. SEM Imagesof Green Synthesized AluminumOxide nanoparticles.

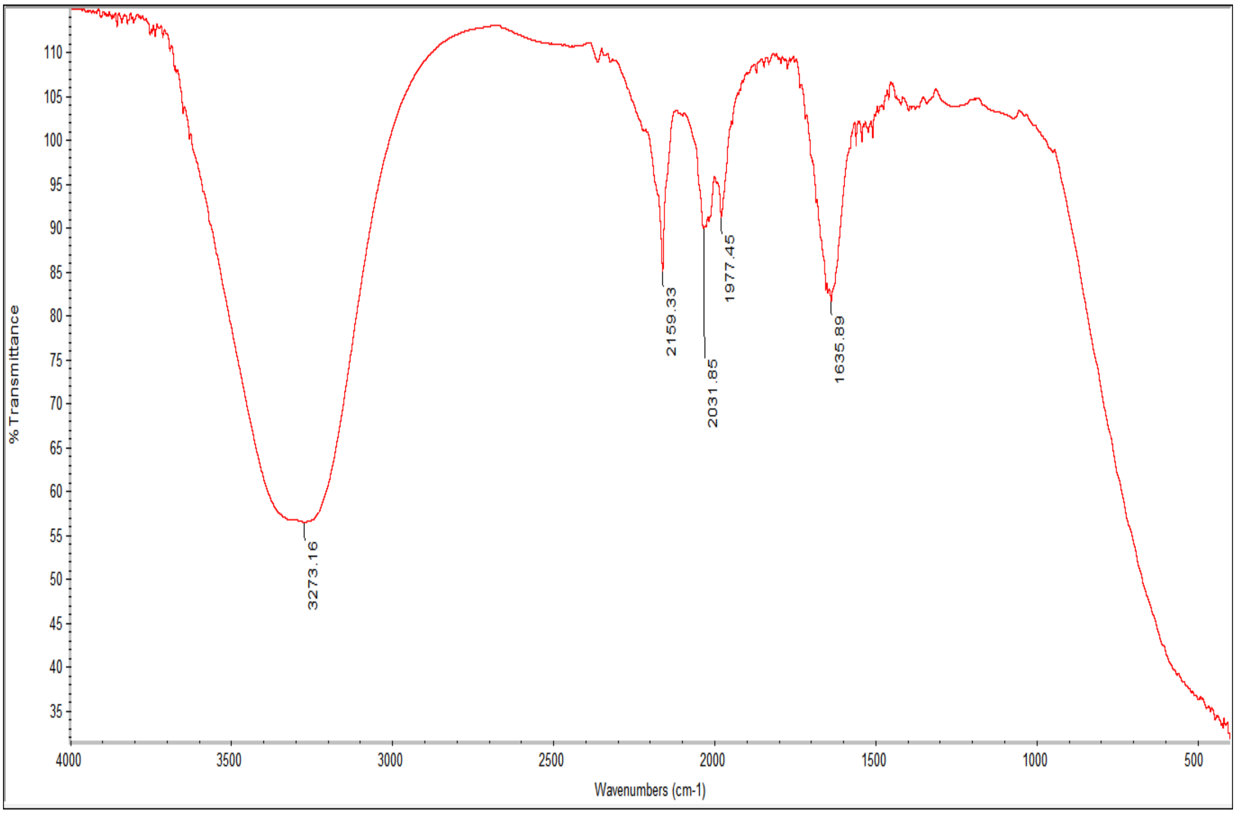

Figure 5. (a) IR spectra of *Mentha Pulegium* Plant Extract

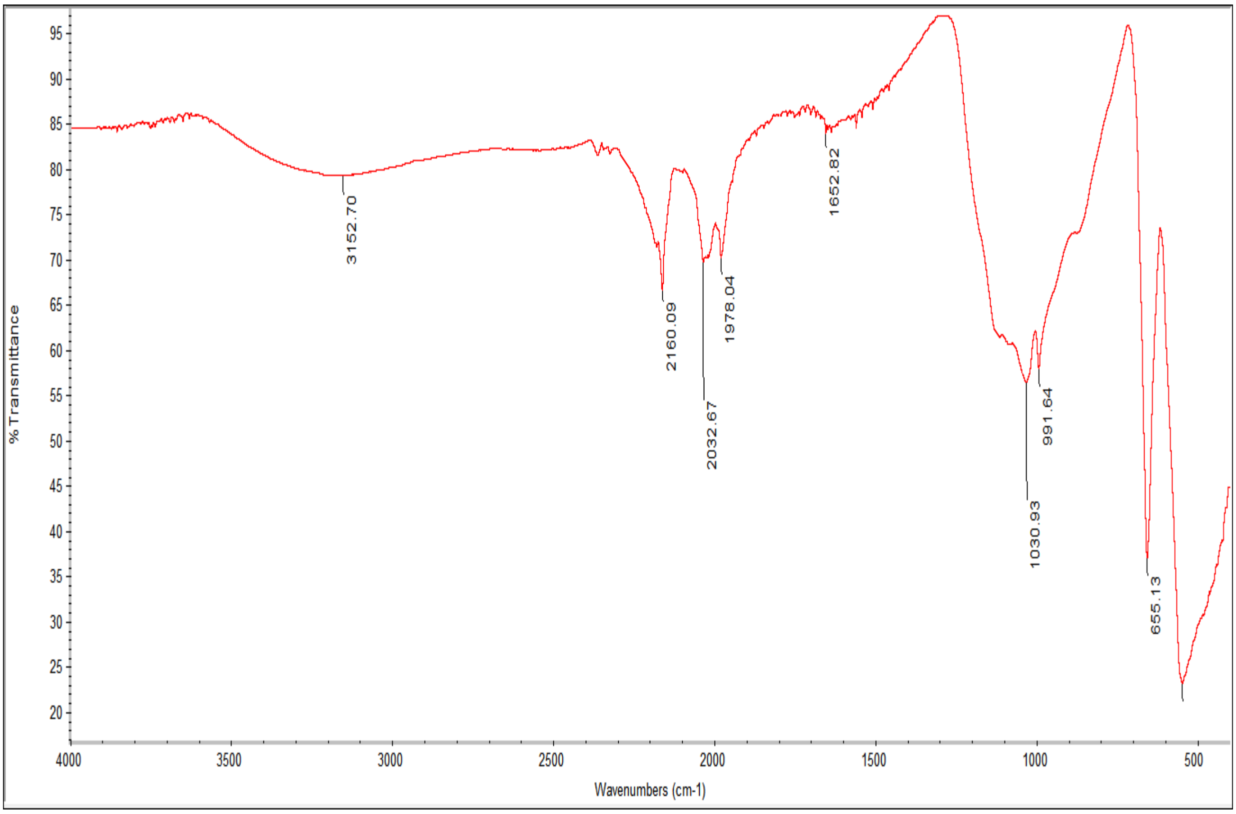

Figure 5. (b) IR spectra of Green Synthesized Al_2O_3 nanoparticles

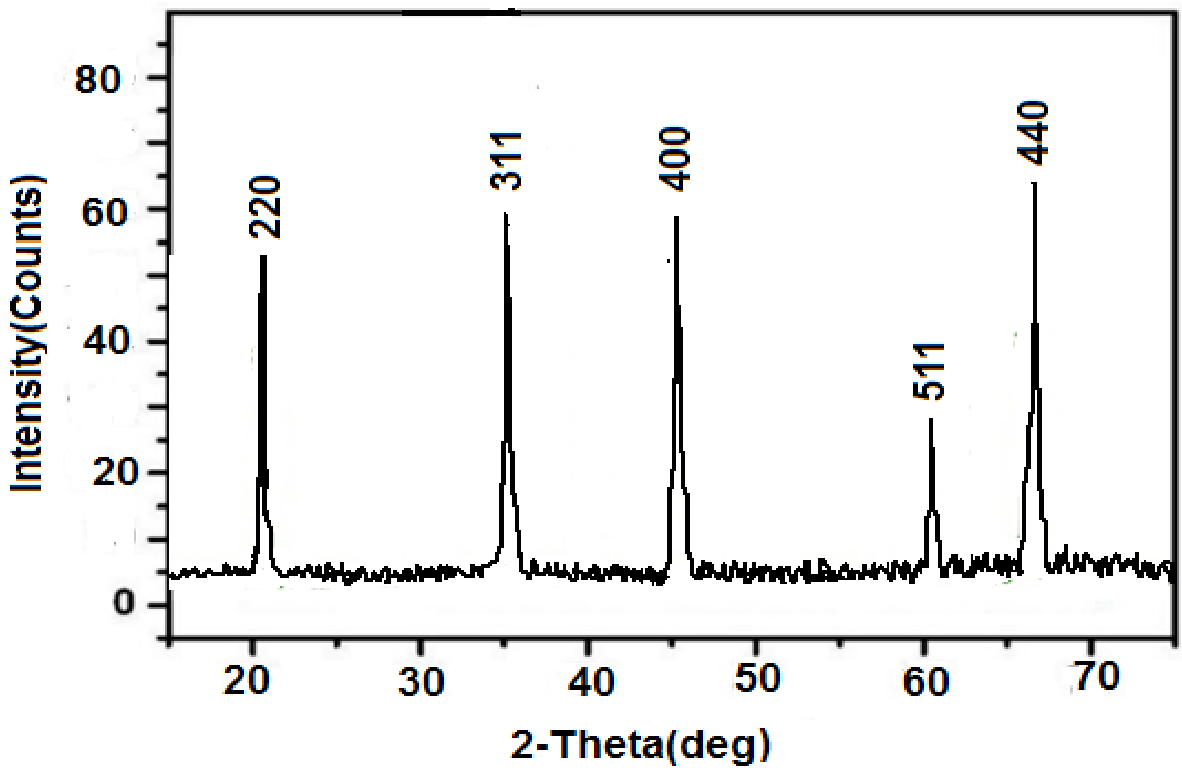

Figure 6.XRD Spectrum of the Green Synthesized Aluminum Oxide nanoparticles

It is clear from lower magnification image1(10 μm) that the prepared nanoparticles are clusters in a large-scale area and have approximately uniform morphologies. Image2(1μm) shows the higher magnification image of such morphologies. The general structure of prepared Al_2O_3-NPs shows little variation. The Scanning Electron Microscope imaged displayed that the prepared nanoparticles agglomerated loosely with each other, resulting in irregular, porous and rough sticky structure, may be due to the green synthesis technique adopted. The primary particles of Al_2O_3-NPs are in a nano-structure and have variety kinds of aggregation, the same results of the green preparation procedure have been reported by Bhutto et al. [36]

3.1.5 Infrared Spectroscopy

The characterization of prepared aluminum oxide nanoparticles done by using Perkin Elmer, Fourier Transform Infra-Red (FTIR) spectrometer. The Fourier Transform Infra-Red spectra of the extract of *Mentha pulegium* plant and aluminum 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, terpenoids, alkaloids, 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 Aluminum oxide nanoparticles. Peaks at 2160 cm^{-1} in both spectra indicate the stretching of alkynes. The peak at ~2032cm^{-1} and ~1978 cm^{-1} in both spectra , represent aromatic ring and C=C alkene bond of flavonoids and terpenoids. The sharp peaks at 1635 cm^{-1} in fig.5(a) corresponds 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 aluminum oxide nanoparticles. The sharp peaks at 550 cm^{-1} and 655 cm^{-1} in figure 5. (b) Compared to figure 5. (a) Is due to the presence of Al-O and Al-O-Al bonds, which explains the presence of aluminum oxide in Al_2O_3-NPs. The presence of the above-mentioned groups indicates that the stabilization of aluminum oxide nanoparticles enhanced by phytoconstituents. Also, it distinguished the peaks around at 991 cm^{-1} and 1030 cm^{-1} in fig.5(b) compare to fig.5 (a) related to the symmetric bending of the (Al–O–H) bond, this agrees with the previous study [37].

3.1.6 X-Ray diffraction Analysis

The crystallinity of the green prepared Al_2O_3 nanoparticles was determined by X-ray diffraction studying, and the results are presented in Fig.6,The X-ray diffraction spectrum of synthesized Aluminum oxide nanoparticles revealed five different Bragg's diffraction peaks at 2θ= 21.2°, 36.5°, 46.3°, 61° and 66.5°, corresponds to the lattice planes (220), (311), (400), (511) and (440) respectively. The observed relative intensities and lattice parameters of the diffraction peaks found closely match the database of the Joint Committee on Powder Diffraction Standards (JCPDS) card file No10-174-072. Thus, explaining the crystalline character of our prepared Al_2O_3-NPs (Fig.5). Imosobomeh L. and Agnes C., et al, also reported the similar results of Al_2O_3-nanoparticles prepared by green methods from neem extract [33]. 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), (λ = 1.5406 A), β is width of the peak of the 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. The value of β in 2θ axis of diffraction profile must be in radians. Scherrer equation, $d = 0.9\lambda/ (\beta \cos \theta)$ was used to estimate grain average sizes of crystallites. The calculated average of crystallite size for Al2O3-NPs was evaluated by the sharpest peak at 2θ = 66.50° which was found to be 35 nm.

4.Conclusions

In this work Al_2O_3 nanoparticles have been successfully prepared by an environmentally friendly, rapid, and low-cost green synthesis method, using the leaf extract of *Mentha pulegium* plant as capping and a reducing agent. The green synthesized Al_2O_3-nanoparticles were successfully characterized using UV-VIS spectroscopy, FTIR, SEM, EDX, and XRD analysis.The particle average size of the prepared Aluminum nanoparticles were found to be about 35 nm. Through the characterization of the resulting particles prepared in this method, boifunctional groups found in Al2O3-NPs come from phytoconstituents, could possibly enhance the stabilization of our prepared nanoparticles. The prepared Al_2O_3 nanoparticles agglomerated loosely with each other, which resulting in irregular, porous and rough sticky structure, and may be due to the green synthesis technique adopted. In short, this represents a quick, simplified description of the resulting nanoparticles, which can benefit from one of their applications in the biological or industrial field.

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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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