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A Sustainable Approach Towards Synthesis of MoO_3 Nanoparticles using Citrus Limetta Pith Extract

Abhimanyu Kanneganti, Manasa, Prathyusha Doddapaneni

Abstract*— Our research focused on a sustainable approach for synthesis of molybdenum trioxide nanoparticles, which can be utilized in oxidation catalyst and electrochemical devices research. This study reports the exploit of pith, generally considered as waste, to prepare an aqueous extract of citrus limetta as an eco-friendly agent for synthesis of MoO_3 nanoparticles. The obtained product was subjected to X-ray diffraction analysis to confirm the formation of MoO_3 nanoparticles by comparing the peak values with JCPDS software which matched with JCPDS card no.35-0609. The transmission electron microscope micrographs were analyzed to study the size and morphology of the nanoparticles. The absorbance spectrum of MoO_3 nanoparticles was studied using the UV-spectroscopy and the absorbance peak was observed at 257nm. The functional groups present in the final product were studied using Fourier transform infrared spectroscopy.

Keywords*— sustainable approach, molybdenum trioxide nanoparticles, green synthesis, citrus limetta.

I. INTRODUCTION

Molybdenum trioxide is an oxide of molybdenum metal; each molecule has one molybdenum atom and three oxygen atoms. It is bluish gray or light yellow in color. Of all the various compounds of molybdenum metal, molybdenum trioxide is the compound whose production is highest. The use of MoO_3 in electrochemical devices and oxidation catalysts are the area of interest of researchers. Though not much research was done in pharmaceutical applications of MoO_3, it is believed to be a potential anti-bacterial agent. All the applications of MoO_3 like oxidation catalyst, electrochemical devices and anti-bacterial agent are directly or indirectly related to either its band gap or surface area; so there is a huge chance of penetration for MoO_3 nanoparticles in those fields replacing the bulk MoO_3. Sustainable approaches are of high necessity in a finite planet like ours. The sustainable approaches unlike incineration, involve the recycling of one or a mixture of bio-degradable or non-bio-degradable wastes, in production or as fuel for production. This not only has environmental benefits but also economic benefits. It plays an important role in the field of waste management. Employing sustainable approaches for production of energy or goods helps in reducing the carbon footprint and in delivering a greener planet and cleaner approaches to the next generation. With the increase in sustainable approaches, our dependency on depleting fossil fuels fades away.

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Sustainable approach in the field of chemistry and material science involves the use of bio-degradable wastes as catalysts or synthesis of materials. The green chemistry, for synthesis of nanomaterials not only makes use of bio-degradable wastes as a replacement for chemicals but also has added advantages like need of lesser temperature during synthesis, no release of toxic by-products and size control of the nanomaterials. Having such brilliant advantages, sustainable chemistry is what all the present researchers have their eyes on.

II. MATERIALS AND METHODS

A. Materials Used

Ammonium heptamolybdate was purchased from MERCK chemicals and two commercially available citrus limetta fruits were used. Double distilled water was used throughout the process.

B. Preparation of Citrus Limetta Pith Extract

Two fresh citrus limetta fruits were taken and washed with distilled water to remove any dust or organic debris on the surface. The fruits were then cut in two halves each. The interior of citrus limetta fruit is slushy and contains seeds. The edible slushy part along with the seeds was scraped of carefully using a penknife. The isolated pith was then minced into small pieces and taken in a beaker, to this 100ml of double distilled water was added. This solution was heated at 60oC for 60mins. The solution turned from transparent to light yellowish green in color. The solution was filtered using a whatman no.1 filter paper.

C. Experiment Procedure

A homogeneous solution of ammonium heptamolybdate was formed by adding 0.1mM ammonium heptamolybdate in 100ml of distilled water and stirring the solution for 15mins. To the ammonium heptamolybdate solution 5ml of filtered citrus limetta pith extract was added drop by drop under perpetual stirring. The solution was then heated at 70oC till the supernant got evaporated. The final dry product left over was dark grayish brown in color. The obtained product was calcinated in at muffle furnace at 700oC for 2hrs at 5oC rise in temperature per minute. During calcination the product slowly turned from dark grayish brown to light bluish gray in color. The product after calcination was finely crushed and stored in air tight vials.

III. RESULTS AND DISCUSSION

The obtained product was subjected to XRD, TEM, UV and FTIR characterizations.

A. X-ray Diffractometer

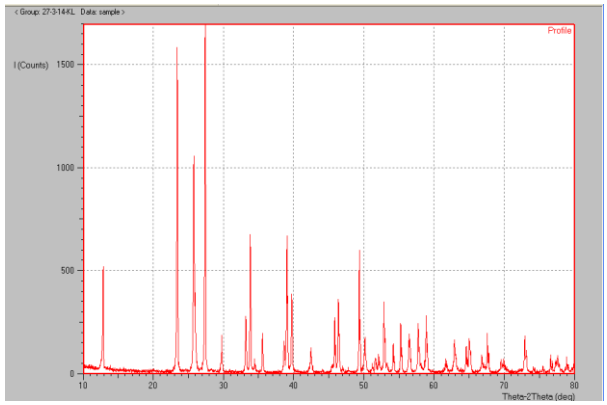

Fig 1. XRD plot of MoO_3 nanoparticles

The product was subjected to XRD analysis using Bruker D-scan X-ray crystallography equipment. A Cu-k-alpha radiation (1.54 Aº) was used, operated at 40Kv and 30mA, with 2Ɵ ranging from 10º-80º. The **Fig 1.** Shows that the XRD peaks were observed at 12.8º, 23.5º, 25.7º, 27.3º, 29.8º, 33.6º, 39.1º, 49.3º, 58.9º and 64.5º. The XRD analysis confirmed that the obtained product was MoO_3 nanoparticles using JCPDS software, when the peak vales were compared they matched with the JCPDS card no.35-0609. The XRD analysis revealed that the MoO_3 nanoparticles had an orthorhombic structure.

B. Transmission Electron Microscope

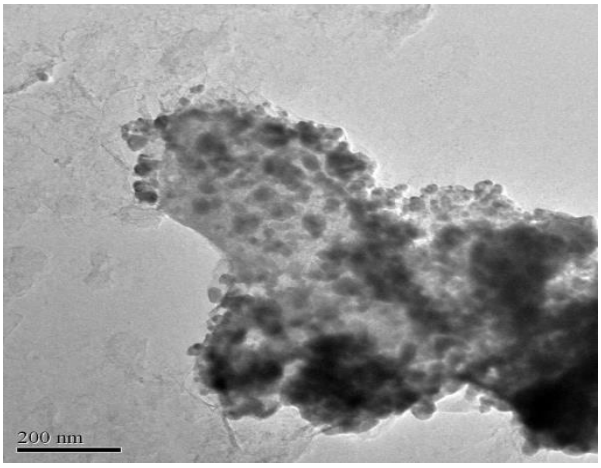

Fig 2. TEM micrograph of MoO_3 nanoparticles at 200nm scale

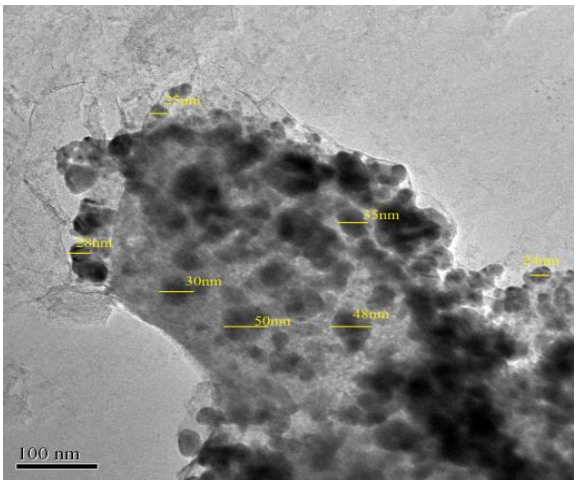

Fig 3. TEM micrograph of MoO_3 nanoparticles at 100nm scale

The MoO_3 nanoparticles have undergone TEM analysis at 200nm and 100nm range. The TEM micrographs reveal that the size distribution of the nanoparticles was pretty uniform. The size of the particles was ranging from 24nm to 50nm. The average size was accounted to be around 34.2nm. The morphological studies revealed that the shape of the particles formed was nearly spherical and uniform. Very less to zero agglomeration of nanoparticles was observed.

A. UV Spectroscopy

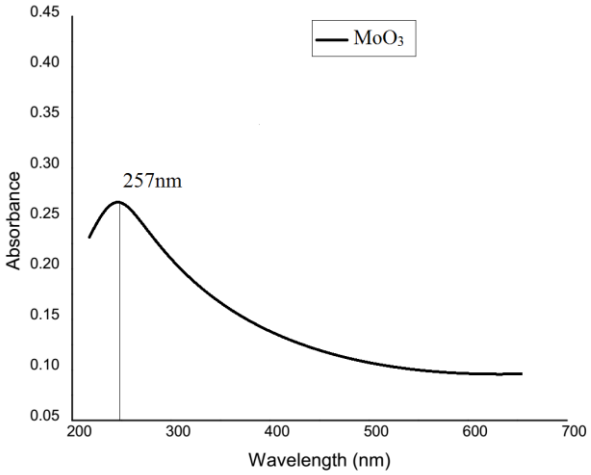

Fig 4. UV spectrum of MoO_3 nanoparticles

The green synthesized molybdenum trioxide nanoparticles were subjected to UV spectroscopy to study the absorption spectroscopy. The absorption was seen maximum at 257nm, which falls under the UV-C region. The UV-C region is called as the short wave ultra violet region, which ranges from 200-280nm. This study indicates that MoO_3 nanoparticles absorb UV-C rays unlike other metal oxides nanoparticles like ZnO or TiO_2 which absorb only UV-A or UV-B rays.

B. FTIR Spectroscopy

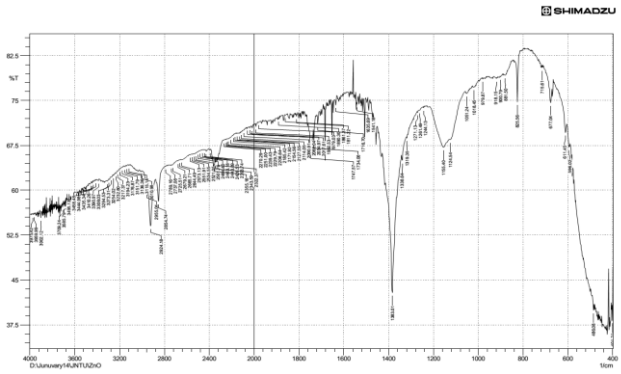

Fig 5. FTIR plot of MoO_3 nanoparticles

The FTIR spectroscopy was done to study the functional groups and bonds present in the compound. In the **Fig 5.** the peaks were observed at 486 cm^{-1}, 1155.4 cm^{-1}, 1383 cm^{-1}, 1747.5 cm^{-1}, 2355.5 cm^{-1} and 2924.1 cm^{-1}. The peak at 486 cm^{-1} is very broad and is the only significant peak below 1000 cm^{-1}. It is well known that in the FTIR spectrum the inorganic stretch peak is seen between 400 cm^{-1} and 1000 cm^{-1}, so the peak at 486 cm^{-1} is expected to be the Mo-O stretch.

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The remaining peaks other than 486 cm^{-1} correspond to bonds (functional group), =C-H bend (alkenes), C-H rock (alkanes), C=O stretch (carboxylic acids), -C=C- stretch (alkynes) and C-H stretch (alkanes) respectively. These stretches indicating the presence of organic compound were observed due to citric acid ($C_6H_8O_7$) which is the key constituent of citrus limetta.

IV. CONCLUSION

A sustainable approach for synthesis of MoO_3 nanoparticles using citrus limetta was studied and was found to be successful. The product was conformation was done by comparing the XRD peak values with the JCPDS software. The product was confirmed as MoO_3 as the peak values matched with JCPDS card no.35-0609. The TEM analysis was done which revealed that the nanoparticles were almost spherical in shape with an average diameter of 34.2nm. The TEM analysis showcased the unique size controlling advantageous ability of green synthesis. The absorbance spectrum of MoO_3 nanoparticles was studied using UV spectroscopy, the absorbance peak was observed at 257nm which falls in the UV-C region.

V. ACKNOWLEDGMENT

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