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Green Synthesis and Characterization of Molybdenum oxide Microstructures Tuned by *Syzygium cumini* Extract.

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Abstract

Molybdenum Oxide arose as one of the significant material due to its superior semiconducting and optical properties. In this present work, Molybdenum Oxide (MoO_3) nanopowders were synthesized by ultrasonication-solution combustion method by using extract of *Syzygium cumini* as a surfactant to tune the morphology of the samples. While preparing, source of MoO_3 used was ammonium heptamolybdate tetrahydrate which was converted into molybdenum nitrate using concentrated nitric acid. Microstructures of the as synthesized samples with different concentrations (5ml, 10ml, 15ml and 20ml) of extract was compared with the bulk material of MoO_3. Powder X-ray Diffraction Patterns (PXRD) obtained result confirms the crystallinity of the prepared samples matches with the JCPDS Card number-98-015-2312 and there is no impurities present in the prepared sample. Scanning Electron Microscopy (SEM) provides morphology of the samples shows the microstructural rods with dispersed nanocrystals structural rods. Configuration of the *Syzygium cumini* was categorized using Gas Chromatography Mass Spectroscopy shows many polysaccharides. Covering of these polysaccharides at the time of ultrasonication led to the formation of microstructures was explained using egg box model.

Key words: *Syzygium cumini*, Ammonium heptamolybdate tetrahydrate, Molybdenum Oxide, ultrasonication, SEM, PXRD.

1. **Introduction**:

In recent years, development of nanoscale materials with different morphologies has become a focal area in basic scientific researches due to their unique properties which differ from those of respective bulk materials. Currently, developing new methods for preparing novel chemical structures within the size range of 1–100 nm is a major challenge of the nano technical field **[1].** Among the nanostructured metal oxides MoO_3 is considered as better anode material because of its anisotropy layered structure. Low cost, low environmental impact, eco-friendly **[2].** MoO_3 is normally found in orthorhombic phase α-MoO_3 **[3].** This phase can be described as a layered structure formed by covalent double layer of MoO_6 octahedral running along the (100) and (001) directions, held together by Van der Waals forces in the (010) direction. These lead to the crystal growth by preferentially exposing the [010] face, thus resulting in elongated stab-like crystals. This characteristic yields directly nanostructures of MoO_3.

Molybdenum oxide (MoO_3) is an significant transition-metal oxide that has conventional significant attention in materials science because of its unique structural, optical, electrical, catalytic and electrochemical properties **[4].** Molybdenum oxide (MoO_3) is one of the most widely investigated compounds. It exists in several polymorphic forms, with orthorhombic α-MoO_3 being the most stable phase under ordinary conditions **[5].** The Characteristic properties of molybdenum oxide are strongly dependent on its crystal structure, particle size, morphology, surface area, oxidation state and defect concentration. These characteristics can be tailored by selecting appropriate different synthesis methods. Therefore, the development of the material simple, rapid, and efficient preparation techniques for molybdenum oxide has become an important area of research for advanced functional materials **[6].** The capability to control the morphology and defect structure of MoO_3 is particularly important because molybdenum oxide exhibits variable oxidation states **[7]**. Although Mo^{6+} is dominant in stoichiometric MoO_3, partial reduction can generate Mo^{5+} and oxygen vacancies. These defects can influence the electrical conductivity, optical absorption, catalytic activity and electrochemical behavior of the material **[8]**. Ultrasonication-assisted combustion may provide opportunities to modify these characteristics through control of reaction conditions and subsequent heat treatment.

Ammonium heptamolybdate tetrahydrate is commonly employed for the preparation of molybdenum oxide. AHT is a water-soluble molybdenum compound that provides molybdate ions in aqueous solution **[9].** AHT is a high purity, good solubility and relatively easy handling make it an attractive precursor for laboratory scale synthesis **[10, 11].** The final product Molybdenum oxide and its characteristics depend on parameters such as precursor concentration, heating rate, combustion temperature, reaction atmosphere and duration of heat treatment [**12**]. In recent years, significantly capable of producing fine and homogeneous oxide powders using simpler and faster processing routes using ultrasonication-solution combustion method. In the preparation of molybdenum oxide, ultrasonication of an AHT-based precursor solution can contribute to improved homogeneity and more uniform distribution of the molybdenum-containing species **[13,14]**. The applications of molybdenum oxide have expanded considerably from conventional catalytic and ceramic applications toward modern energy and electronic technologies **[15].** In catalysis, MoO_3 is valued for its redox properties and surface activity and has been investigated in oxidation reactions and as a component of multifunctional catalysts. Its catalytic behavior is strongly related to surface area, crystal orientation, oxidation state and defect concentration **[16].**

The modern application of MoO_3 is in electrochemical energy storage **[17, 18].** MoO_3 has attracted attention as an electrode material for lithium-ion batteries, sodium-ion batteries and other emerging rechargeable battery systems because its layered structure can accommodate mobile ions. The electrochemical performance is affected by particle size, crystallinity, porosity, structural defects and phase composition. Thus, ultrasonication-assisted combustion provides a potentially useful for producing fine and porous MoO_3 powders for energy-storage research **[19,20].** Molybdenum oxide (MoO_3) is also widely investigated for electrochromic devices because its optical properties can reversibly change during ion insertion and extraction. This property makes MoO_3 relevant to smart windows, variable-transmittance optical devices, displays and energy efficient building technologies **[21].** Nanostructured MoO_3 can provide improved ion accessible surface area and facilitate electrochemical switching [**22].**

In modern optoelectronics, molybdenum oxide is also of interest as a functional interfacial and charge-transport material. MoO_3 has been investigated in applications involving organic light-emitting diodes, organic photovoltaic devices and other thin-film electronic architectures **[23].** Its ability to facilitate charge extraction and modify electrode interfaces makes it an important functional oxide in advanced device fabrication. Also, molybdenum oxide has potential applications in photocatalysis, environmental remediation, supercapacitors, solar-energy technologies, and chemical sensing **[24].** Its performance in these applications can be enhanced through nanoscale engineering, composite formation, doping and introduction of oxygen vacancies. Overall, the preparation of molybdenum oxide from ammonium heptamolybdate tetrahydrate using an ultrasonication-assisted combustion method represents a promising approach for producing fine, porous and potentially nanostructured oxide materials **[25].**

In this work, MoO_3 nanopowders have been synthesized by eco-friendly ultrasonication-solution combustion method by using extract of Syzygium *cumini* as a surfactant to tune the morphology of the samples **[26].** While preparing, source of MoO_3 used was ammonium heptamolybdate tetrahydrate which was converted into molybdenum nitrate using concentrated nitric acid. Starting from ammonium heptamolybdate as precursor and using an organic additive, MoO_3 particles were prepared with different morphologies within the range of 40–100 nm. Effects of reaction conditions such as media pH, concentration of metal ion in solution and additive/Mo molar ratio on particles morphology, and size have been precisely investigated **[27].** This approach is an easy and one step process which might be used as a method for mass production of MoO_3 with low costs.

2. Experimental Method:

Molybdenum trioxide (MoO_3) nanopowder was prepared using ammonium heptamolybdate tetrahydrate (AHT), as the molybdenum precursor through an ultrasonication-assisted solution combustion method. A calculated quantity of AHT was dissolved in deionized water under continuous magnetic stirring to obtain a homogeneous precursor solution. Added 5/7 ml of con. nitric acid to the solution mixture and the solution was subjected to ultrasonication for about 30 min to enhance dissolution and promote uniform mixing at the molecular level. The resulting solution was heated at approximately 80° C to evaporate excess water. The combustion product was allowed to cool to room temperature and was gently ground using an agate mortar to obtain a fine powder. The resulting powder was further calcined in air at an appropriate temperature, typically 550° C nearly about 45 minutes to remove residual organic matter and improve the crystallinity of MoO_3. Finally, the calcined material was ground and stored for further characterization. The phase and crystallinity of the prepared MoO_3 nanopowder can be investigated using X-ray diffraction (XRD), while morphology and particle size can be examined using SEM/TEM.

Similarly, a series of mixtures were prepared by dissolving 2.5 g of AHT in 10 mL of distilled water. To this solution, different volumes of Jamun (*Syzygium cumini*) plant extract, namely 5, 10, 15, and 20 ml, were added separately to obtain four different compositions. Each mixture was subjected to continuous stirring using a magnetic stirrer to ensure proper mixing and interaction between AHT and the plant extract. During stirring, the colour of the mixtures gradually changed to dark violet, indicating the formation of a reaction product. After magnetic stirring, 7 ml of concentrated nitric acid (HNO_3) was carefully added to each mixture. The resulting solutions were subsequently subjected to ultrasonication to promote uniform mixing and facilitate the reaction. Following ultrasonication, the initially dark violet colour changed to pale yellow. The prepared solutions were then considered for further characterization and analysis.

Also, Ammonium Heptamolybdate Tetrahydrate (AHT) of mass 2.245, 1.236, 1.730, 0.7415 g was dispersed in 10 ml of distilled water, followed by the addition of Jamun plant extract (5, 10, 15 and 20 ml) under magnetic stirring. Subsequently, TEOS (Tetra Ethyl Orthosilicate) of volume 0.5, 1.3, 2.1 and 2.9 ml was added along with another 10 ml of distilled water and the corresponding volume of Jamun plant extract5 to 20 ml. The reaction mixture initially exhibited a light blue colour, indicating the formation of the precursor. After ultrasonication, 7 ml of concentrated HNO_3 was added as an acidic catalyst, resulting in a change of colour from light blue to pale yellow. The colour change suggests modification of the reaction medium and progression of hydrolysis and condensation reactions involving TEOS.

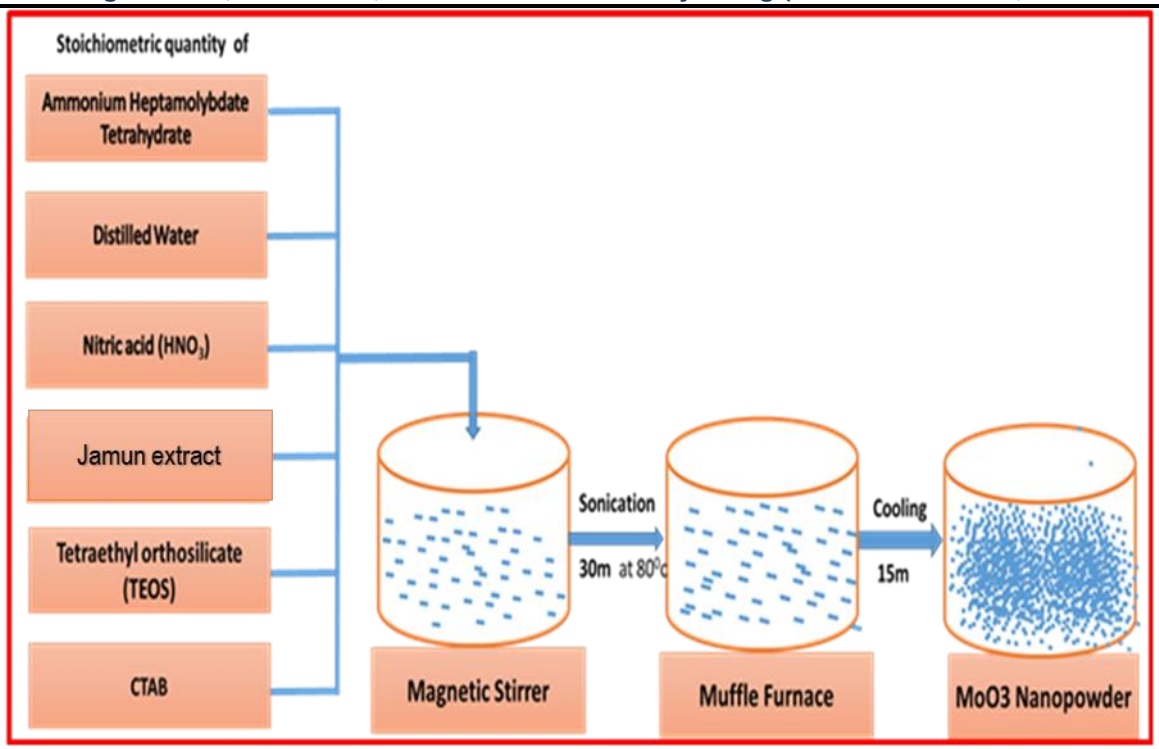


Fig.1.Depicts the preparation of Molybdenum oxide using Ammonium Heptamolybdate Tetrahydrate using ultrasonication-assisted solution combustion method.

3. Result and Discussions:

The reaction mechanism Ammonium heptamolybdate tetrahydrate (AHT) is a big complex molecule which is usually used as a precursor in molybdenum compounds preparation. The XRD results confirm the formation of crystalline molybdenum oxide through the ultrasonication-assisted combustion route using ammonium heptamolybdate tetrahydrate as the precursor and jamun extract as a natural surfactant **[28].** The characteristic diffraction reflections are predominantly consistent with orthorhombic α-MoO_3. The observed peak broadening indicates the formation of relatively small crystallites, which may be associated with the combined effects of ultrasonic irradiation and the phytochemical constituents of jamun extract during nucleation and crystal growth. The absence of significant impurity peaks suggests good phase formation, while the crystallite size can be quantitatively estimated from the FWHM of the major diffraction peak using the Scherrer equation. Thus, the XRD analysis demonstrates that the employed green ultrasonication-assisted combustion method is effective for producing crystalline MoO_3 with potentially controlled structural characteristics **[29].**

Figure.1. shows PXRD patterns for synthesized MoO_3nanostructures using different molar concentration by using Jamun plant extract as a fuel. for synthesis of 5 ml and 10 ml of molar concentration of MoO_3nano powder some amorphous impurities peaks are appeared, for increasing molar concentration of 15 ml, impurities peaks are disappeared and only pure crystallineMoO_3peaks appeared and The PXRD confirms the formation of MoO_3 nanostructures crystallinity JCPDS No. 13–0128 **[30]**. Further, inversing the molar concentration of 20 ml, some amorphous impurities peaks are appeared. 15 ml of molar concentration of Jamun plant extract as optimize for synthesis of crystalline MoO_3 nanopowder.

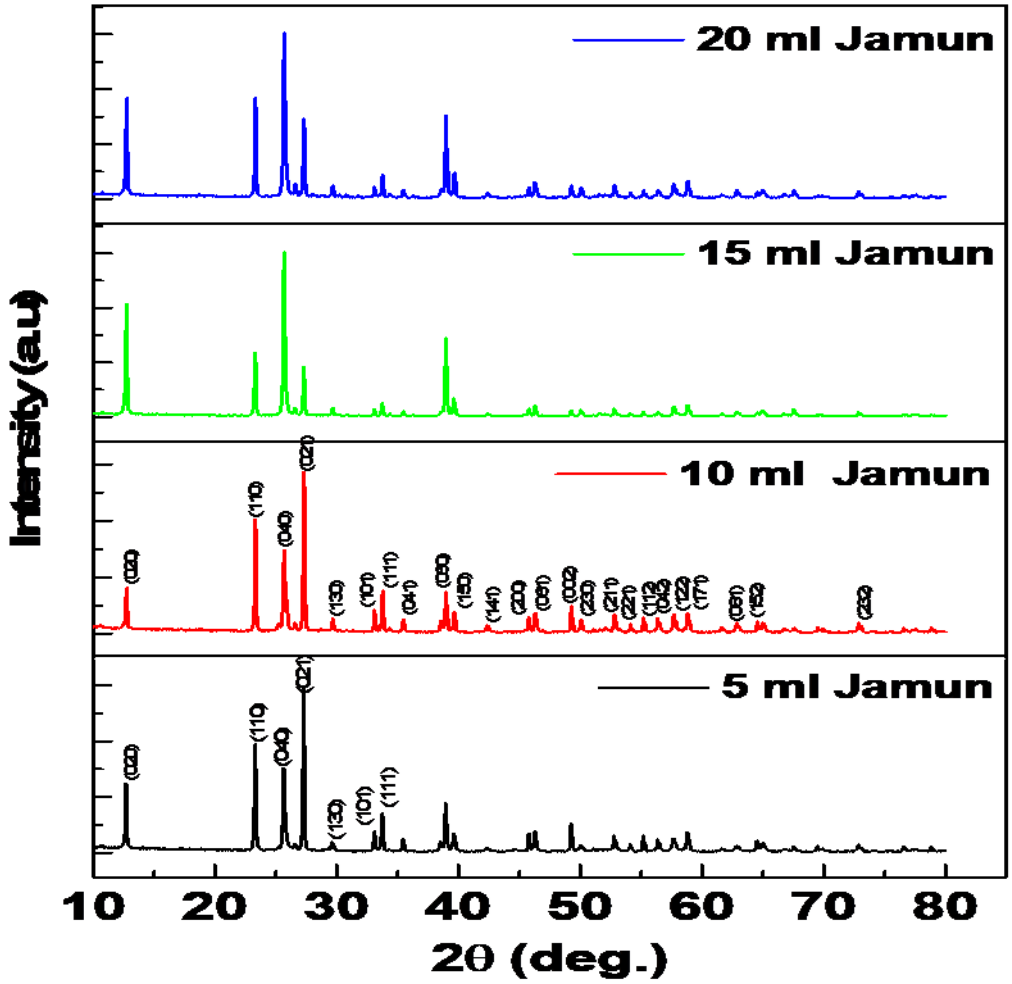


Fig.2 . PXRD patterns for MoO_3 nanostructures obtained by using Jamun plant extract as a fuel for different molar concentration for synthesis of 5 ml, 10 ml, 15 ml and20 ml.

The XRD pattern of MoO_3 is that the addition of 15 mL Jamun extract with TEOS sample shows the characteristic diffraction peaks of AHT, confirming the presence of its crystalline structure **[31].** The addition of Jamun extract causes changes in peak intensity and broadening due to interactions between its phytochemicals and AHT. The incorporation of TEOS contributes a broad amorphous attributed to the formation of silica (SiO_2) through hydrolysis and condensation **[32].** A decrease in peak intensity or increased peak broadening indicates reduced crystallinity and increased structural disorder as shown in figure 2.

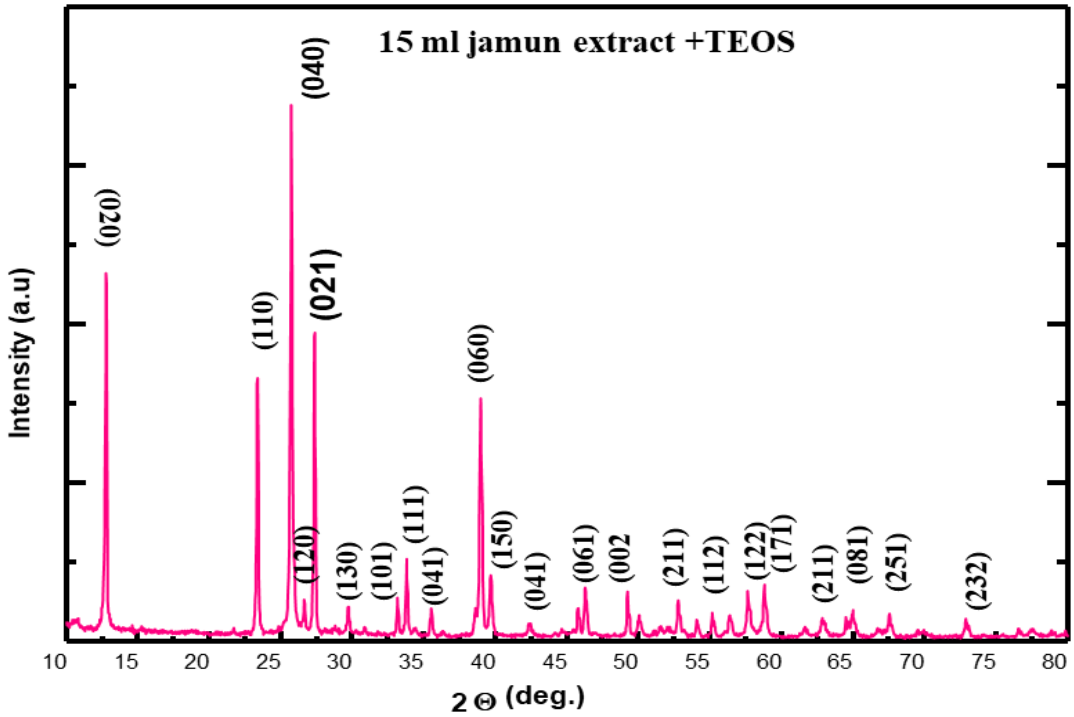


Fig.3. Shows PXRD patterns for synthesized MoO_3 nanostructures using 15 ml of molar concentration by using Jamun plant extract as a fuel, TEOS as surfactants.

The XRD pattern of the AHT with 15 mL Jamun extract and the TEOS–CTAB mixture shows the characteristic crystalline peaks of AHT, confirming the retention of its crystalline structure **[33].** The addition of Jamun extract may cause changes in peak intensity and broadening due to interactions between phytochemicals and AHT. TEOS (Hexadecyl Trimethyl Ammonium Bromide) contributes to the formation of an amorphous silica network, while CTAB acts as a structure-directing agent during silica formation. The observed peak broadening and reduced intensity may indicate decreased crystallinity and increased structural disorder. General, the XRD pattern confirms the formation of a modified Ammonium Heptamolybdate Tetrahydrate with the addition of Jamun extract and the composites of TEOS (Tetra Ethyl Orthosilicate) /CTAB (Hexadecyl Trimethyl Ammonium Bromide) as shown in figure 3.

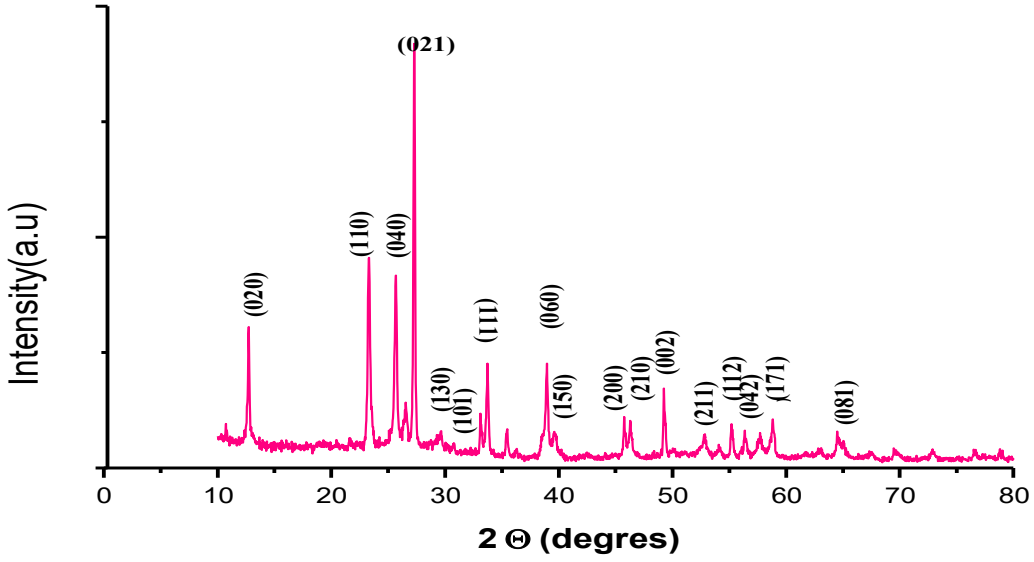


Fig.4. PXRD patterns for synthesized MoO_3 nanostructures using 15 ml of molar concentration by using Jamun plant extract as a fuel, AHT and TEOS as surfactants.

Various additives were examined in similar reaction conditions to find the most effective template for MoO_3 nanoparticles growth. Jamun plant extract as a fuel, AHT and TEOS as surfactants were utilized in a solution with 15 mole/L concentration of Mo and an additive/Mo molar ratio of 1. pH of the reaction media was about 5 in the presence of all the additives. The synthesis temperature was kept at 80^0 C. The SEM images of related samples are shown in Fig. 4. It may be seen that each additive leads to a special morphology and particle size. For example, MoO_3 nanoparticles growth Jamun plant extract as a fuel leads to formation of the rod like structures with agglomerationMoO_3 nanoparticles (Fig. 4a), addition of Jamun plant extract as a fuel, TEOS as surfactants results in larger submicron and nanoparticles with less rod shape and agglomeration (Fig. 4b) **[34].** On the other hand, when Jamun plant extract as a fuel, AHT and TEOS as surfactants were used and completely different morphologies of micro and nanorods obtained (Fig. 4c,). This leads one to conclude that dissimilar structures of additives might cause different chemical and physical interactions between templating structure and ammonium heptamolybdate complex **[35]**. EDX confirms the purity of the samples without any traces of impurities as shown in Fig.5. These differences might in turn affect the rate of nuclei formation and growth.

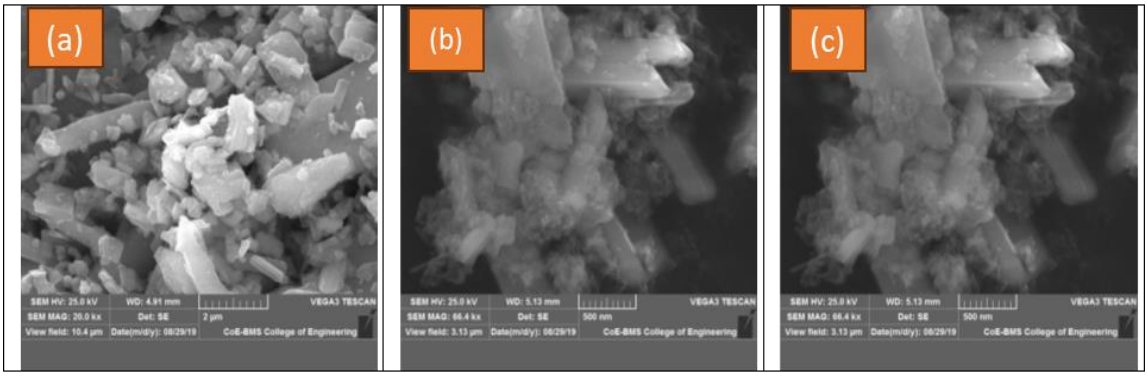


Fig. 5. SEM images of samples grown at same conditions with different additives (a). Jamun plant extract as a fuel of 15 ml, (b). Jamun plant extract as a fuel, TEOS as surfactants of 15 ml, (c). Jamun plant extract as a fuel, AHT and TEOS as surfactants of 15 ml concentration.

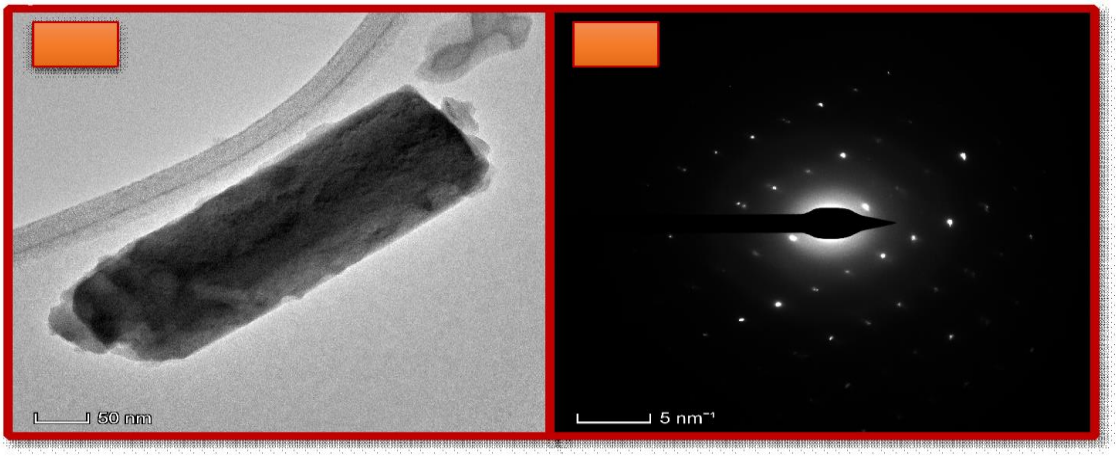


Fig.6 TEM images of a) AHT with 15 ml of jamun extract as a fuel and b) Its SAED Pattern.

Figure (a) presents the TEM micrograph of AHT synthesized using 15 mL of Jamun extract. The sample exhibits predominantly elongated/rod-like nanoparticles with some degree of agglomeration. The particle dimensions are in the nanometer range, with the observed particles size extending to approximately 200–250 nm **[36].** The morphology suggests that the bioactive phytoconstituents of Jamun extract may participate in nucleation, stabilization, and directional growth of the AHT particles. Figure (b) shows the corresponding SAED pattern, consisting of distinct concentric diffraction rings along with bright diffraction

spots, confirming the crystalline/polycrystalline nature of the synthesized AHT. The observed diffraction rings correspond to the crystallographic planes of the material.

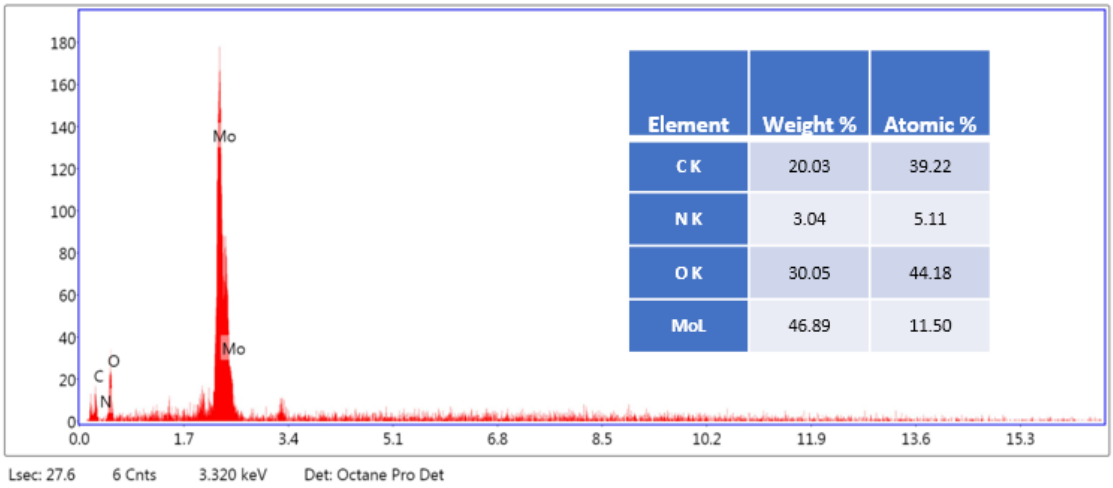


Fig.7. EDX graph of MoO_3 nanoparticles samples.

Ultrasonication reactions occur within the interfacial region, forming nanocrystals due to the very high quenching rate experienced by the products. Several factors influence sonochemical yield and the acoustic cavitation such as low vapor pressure, high surface tension and high sound speed. GCMS studies shows the contents of polysaccharides in plant extracts. Major carbohydrates noticed were glucose (59.2 wt%), Xylose (14.8%), Galactose (6%), Arabinose and Mannose (6.5 % each) as shown in Fig.8. Some traces of disaccharides such as cellobiose were also observed **[37].** All these polysaccharides wrap into complex chains along with the help of protein chains of the extract makes the preparation of the material similar to the egg box model leading to the formation of nanostructures /superstructures of the nanocompounds.

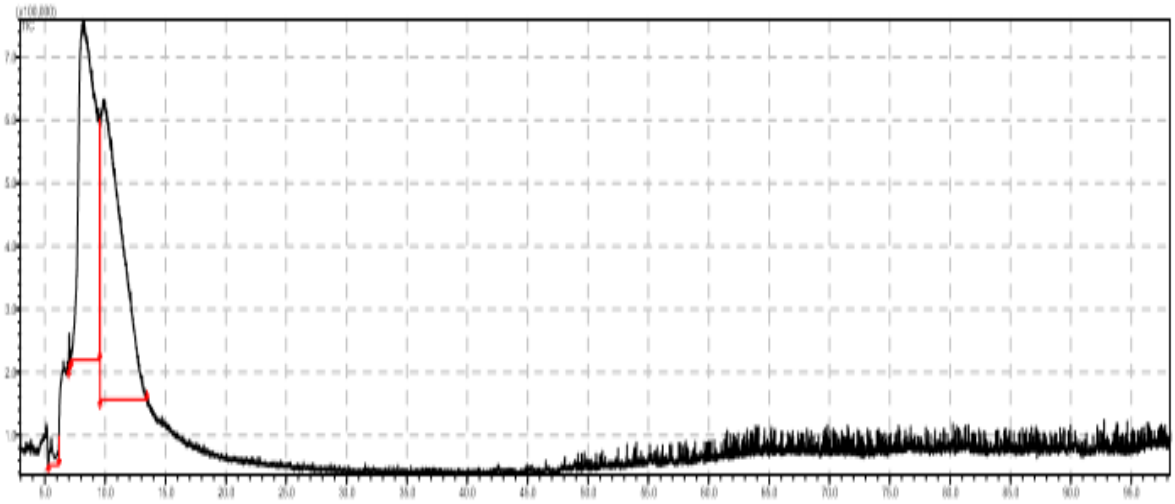


Fig.8. GCMS studies of MoO_3 nanoparticles samples.

4. Conclusions:

Nanostructures of molybdenum oxide were synthesized by successfully prepared the MoO_3 Nanopowders by using plant extract as a fuel, AHT and TEOS as surfactants. Different reaction conditions were examined in order to obtain the best criteria for producing nanoparticles and nanorods of MoO_3, the size of which through obtained PXRD spectra and SEM images were determined to be in the range of 10–100 nm. It was demonstrated that this size may be controlled through such synthesis parameters as additive/Mo molar ratio, media temperature, the solution pH, and concentration of Mo ions in the solution. In other words, by tuning these parameters one may control the size and morphology of materials synthesized. The PXRD confirms the formation of MoO_3 nanostructures and SEM images show the rod like structures with agglomeration. EDX confirms the purity of the samples without any traces of impurities. Ultrasonication reactions occur within the

interfacial region, forming nanocrystals due to the very high quenching rate experienced by the products. GCMS studies shows the contents of

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