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A Facile Synthesis and Characterisation of CdS Nanoparticles at Low Temperature Using Aegle Marmelos Extract

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Abstract. CdS NPs have become found applications across diverse domains in the field of nanobiotechnology because of their established biomedical characteristics. The dimensions and the materials used for coating CdS NPs are pivotal factors influencing their effectiveness in biomedical applications like cancer treatment, bacterial inhibition, bioimaging and biosensors. In this study, we succesfully produced CdS NPs by ecofriendly approach using *aegle marmelos* extract. The synthesized CdS NPs was subjected to characterization using X-Ray Diffraction spectroscopy (XRD), Ultraviolet-Visible spectroscopy (UV-VIS), Fourier Transform Infrared spectroscopy (FT-IR), Dynamic Light Scattering (DLS), Scanning Electron Microscope (SEM) with EDX and Transmission Electron Microscope (TEM). The research delineates a straightforward, economically viable environmentally friendly approach to synthesizing CdS nanoparticles, which is suitable for extensive production. Additionally, it presents a tactic to regulate both the size and distribution of nanoparticles using eco-friendly biomolecules, enhancing their potential for various applications. Based on the results obtained from the characterization studies, it was proposed to the further process for biological applications.

Keywords: Green Synthesis, CdS nanoparticles, *aegle marmelos*, Characterisation

1. Introduction

Due to recent advancements in nanotechnology, a broad array of substances, with a minimum of one dimension falling within ranging between one and hundred nanometers, can now be engineered. These materials, known as nanomaterials (NMs), are artificially created substances with dimensions at the nanometer scale. They are, in at least one dimension, less than 100nm rendering them imperceptible to the naked eye, while conventional bulk substances exceed hundred nanometers in every aspect and can be observed using basic microscopes. Nanoparticles, in contrary to their bulk counterparts, exhibit physical properties that are contingent on their morphology and dimensions rather than being dimension-independent [1]. The utilisation of non-toxic chemicals throughout the entire synthesis process makes the production of nano-sized particles through "green" techniques both cost-effective and environmentally sustainable. The underlying principle of "green chemistry" plays a pivotal role in this context, where extracts derived from fruits, plant stems, seeds, roots, and vegetables are utilized. By

employing natural reducing agents and stabilisers, this approach reduces the reliance on potentially hazardous chemical reagents [2].

Nanoparticle synthesis methods fall into two primary categories, each with distinct approaches. The first approach is known as the bottom-up method, which involves chemical techniques such as co-precipitation, electrochemical, hydrothermal, photochemical, sonochemical, etc. This method involves combining individual atoms or molecules to produce nanoparticles. The second approach, known as the top-down method, relies on physical techniques like arc discharge, ball milling, electro-deposition, evaporation-condensation, lithography, sonication, etc. In this case, external forces are applied to break down solid bulk materials into smaller particles [3]. Chemical methods involving the use of hazardous and toxic compounds for particle reduction and stabilisation not only pose environmental risks but are also expensive and time-consuming, requiring high energy inputs. On the other hand, biological and physical methods stand out as ideal techniques for nanoparticle synthesis due to their simplicity, safety, eco-friendliness, and effectiveness [4].

Similarly, various methods have been employed to synthesis CdS NPs. In this particular study, CdS NPs are synthesised through chemical precipitation techniques chosen for their simplicity, cost-effectiveness, and minimal reliance on specialised equipment and organic solvents [5]. Cadmium (Cd) possesses unique attributes, including high electrical conductivity, malleability, flexibility, and corrosion resistance [6]. Additionally, cadmium sulfide (CdS) exhibits fluorescence, recognised for its outstanding optical and electrical properties, photocatalytic activity, and diminished harmfulness in comparison to Cd [7]. Various types of Cadmium sulfide NPs are generated through different physical, chemical and biological approaches. In order for CdS NPs to be useful, they need to have certain properties, like dimensions, morphology and surface charge which are all controlled by the way they were made [8]. In the medical sciences, CdS NPs synthesised through biological methods are more prevalent due to their reduced harmfulness and superior suitability with biological systems [9]. The synthesised nanoparticles underwent a comprehensive physicochemical assessment, encompassing UV-Vis, FTIR, XRD, DLS, SEM, and TEM analyses. These analyses confirmed the successful synthesis of CdS NPs with the desired size and morphology. Additionally, the UV-Vis analysis revealed the characteristic absorption peak of CdS NPs, indicating their optical properties are suitable for various applications in biomedical research and technology.

2. Materials and methods

2.1. Chemicals

The entirety of synthesis reagents was acquired from SRL Chemicals Pvt. Ltd. and were of analytical quality. Double-distilled water had been utilised throughout the entire reaction.

2.2. *Preparation of Aegle Marmelos* extract:

Ariyalur District in Tamil Nadu, India, is where the Aegle Marmelos leaf was gathered. The Aegle Marmelos leaf was picked from the plant and given a thorough rinsing to get rid of any remaining dirt. The Aegle Marmelos leaf was subsequently, it was left to air dry for a duration of twenty days before being chopped into bits and ground. With constant stirring, 10 grammes of this powder have been heated in hundred milliliters of double-distilled water in a beaker. It became filtered and cooled to room temperature after 1 hour. After filtering, the extract was put away for further synthesis.

2.3. Green synthesis of Cadmium sulfide nanoparticles

To 50 milliliters of double-distilled water, comprising 0.1 M cadmium acetate dihydrate, 10 ml of Aegle Marmelos extract was added. The resultant aqueous solution of sodium sulfide (50 ml, 0.1 M) was also added very slowly. There was an instant shift in colour, and the final solution looked anywhere from pale brown to orange. At 80–90 °C, this solution had been stirred for 10 h. The final colour, orange, showed that CdS nanoparticles had been formed as shown in the Figure 1. The accumulated CdS nanoparticles obtained during synthesis were subsequently frozen for use in future research.

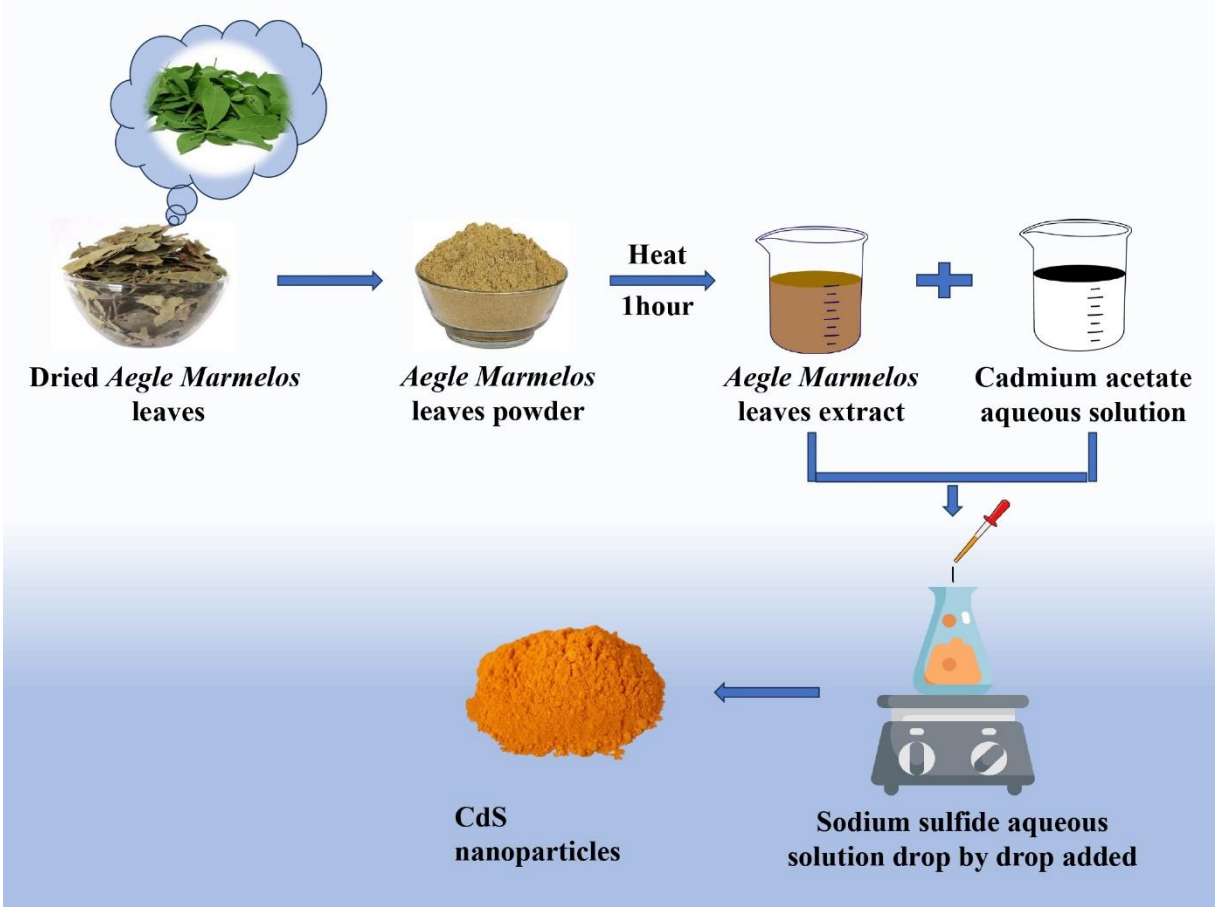

Figure 1 - Bio-synthesis of Cadmium sulfide nanoparticles utilizing *Aegle Marmelos* leaves extract.

Table 1 - Synthesis methods and characterization techniques for cadmium sulfide nanoparticles

Sr. No	Characterization	Morphology	Size(nm)	Ref
1	UV–vis, SEM, EDX, XRD	Spherical	50–180	10
2	UV–vis, TEM, EDX, FTIR	Crystalline	200–250	11
3	UV–vis, DLS, zeta potential	-	95.6	12
4	SEM, EDX, FTIR, XRD	Triangular	40–80	13
5	UV–vis, FT-IR, SEM, EDX, XRD, TEM	Spherical	95.1	present

2.4. Characterisation

We used XRD, UV-Vis, FT-IR, FE-SEM with EDX and HR-TEM to study the CdS nanoparticles that were made from Aegle Marmelos leaf extract. These methods of characterization yielded important details regarding the structural, morphological and optical characteristics of the Cadmium sulfide NPs. The NPs' crystal structure and phase purity were discovered by XRD analysis, and their optical characteristics were discovered by UV-VIS spectroscopy. The functional groups in the leaf extract that could have aided in the formation of CdS NPs were found using FT-IR spectroscopy. Particle size and form might be examined using FE-SEM and EDX.

3. Result and discussion

XRD were utilised to evaluate the crystalline structures and phases of the nanoparticles. The particle size, also known as the grain dimension, was calculated using the Scherrer formula. The XRD pattern of CdS NPs made from Aegle Marmelos leaf extract can be seen in Figure 2. It shows three intensity peaks in the 2θ¸ range, with values of 26.507 °, 30.808 °, and 52.134 degrees. According to JCPDS card number 00-010-0454, these peaks are the (111), (220), and (311) crystallographic planes. They help make the spherical shape of CdS NPs. The presence of these wide peaks suggests that the granules exhibit either a high degree of amorphousness or partial crystallinity. The widening of the XRD peaks indicates a small particle size and a non-uniform distribution of particle sizes [14].

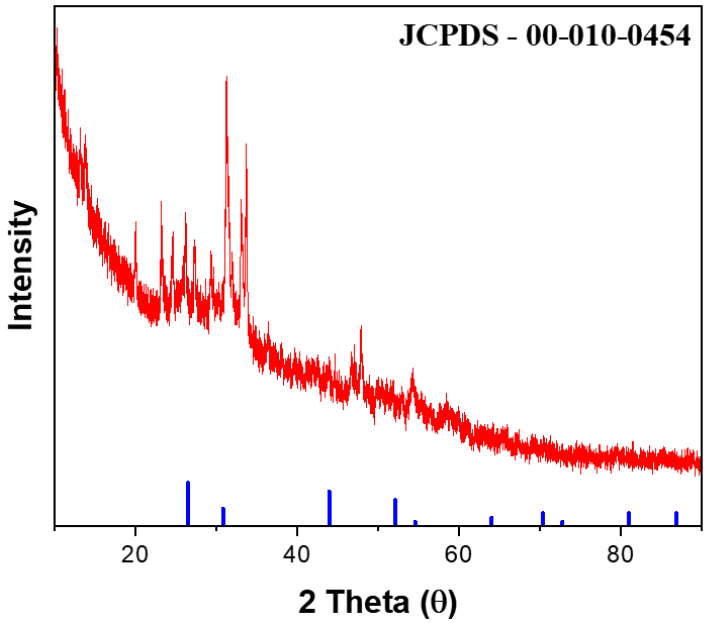

Figure 2 – XRD diffraction pattern with JCPDS card number.

As depicted in Figure 3, UV-Vis spectroscopy were utilized to evaluate the optical properties of the CdS NPs. Notably strong absorptions were observed in the ultraviolet-visible range, spanning from approximately 300 to 490 nm in the graph. The CdS NPs that were studied had UV-Vis absorption peaks within the spectrum of wavelengths of 273.00 to 431.00 nm, which corresponds to absorbance values of 1.306 and 0.395. These peak positions are directly linked to the average diameter of the particles, with smaller diameters corresponding to shorter wavelengths. The findings showed that there was a covering substance, which was Aegle Marmelos leaf extract. This substance stopped the nanoparticles from sticking together to form a solid mass. This provided concrete evidence of the influence of quantum confinement, also known as the quantum size effect (QSE). The UV-Vis blue-shift, which couldn't be explained before, can be explained by the QSE in the case of direct gap semiconductor nanocrystals. This includes things like the optical absorption edge moving towards higher energies as the size decreases [15]. The positions of the peaks in the absorption spectrum can indeed be correlated with the mean diameter of particles. Smaller particle diameters correspond to absorption peaks at shorter wavelengths, representing higher energy transitions. The Quantum Size Effect (QSE) observed in direct-gap semiconductor nanocrystals results in a shift of the optical absorption edge towards higher energies as the size decreases. This phenomenon accounts for the apparent UV–vis red-shift effect. The band gap of the CdS NPs in this study was determined to be 4.56 eV using the formula $E = hc/\lambda$, where E represents the energy gap of the material, h is Planck's constant, c denotes the velocity of light, and λ is the wavelength or onset absorption wavelength. [16].

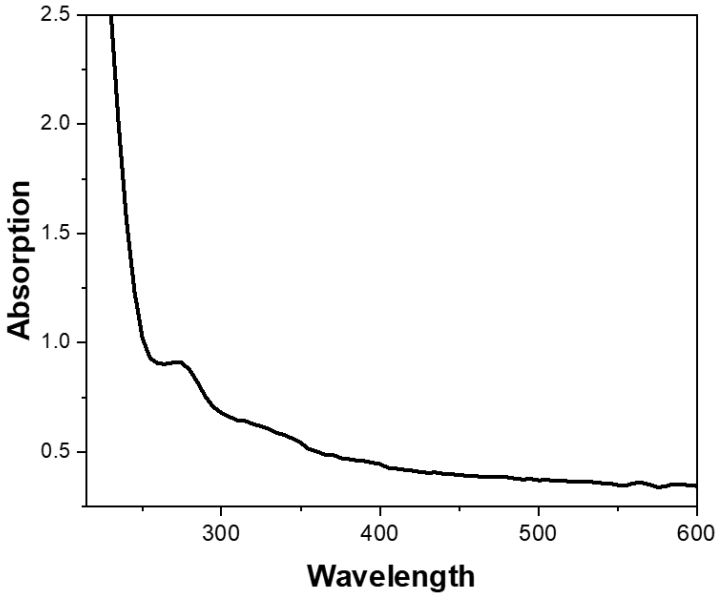

Figure 3 – Uv-visible spectroscopy synthesised CdS NPs

FT-IR spectra of CdS NPs occur presented in Figure 4, displaying intricate spectral lines with maxima at 3443, 2964, 2930, 2875, 1625, 1451, 1385, 1195, 1134, 1112, 950, and 565 cm^{-1}. The peak at 3443 cm^{-1}, corresponding to O-H stretching, is in accordance with the observation of the O-H stretching at 3434 cm^{-1}. However, the latter peak appears considerably broader than expected due to the presence of adsorbed moisture in the synthesized complex.

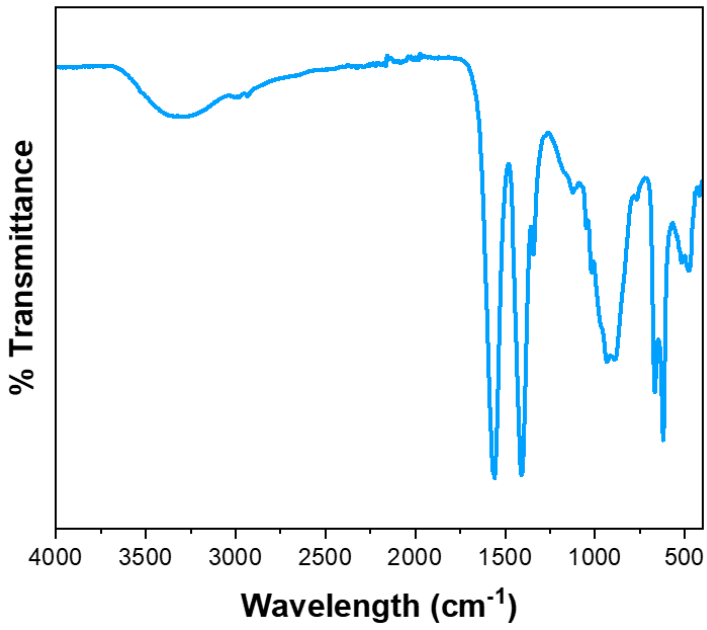

Figure 4 – FT-IR spectroscopy for synthesised CdS nanoparticles

The presence of a signal at 2921 cm^{-1} signifies that an alkyl group has acted as a cap for the newly formed CdS. In addition, the appearance of a peak at 1115 cm^{-1} suggests the formation of propyl chains, serving as capping agents for the CdS [16].

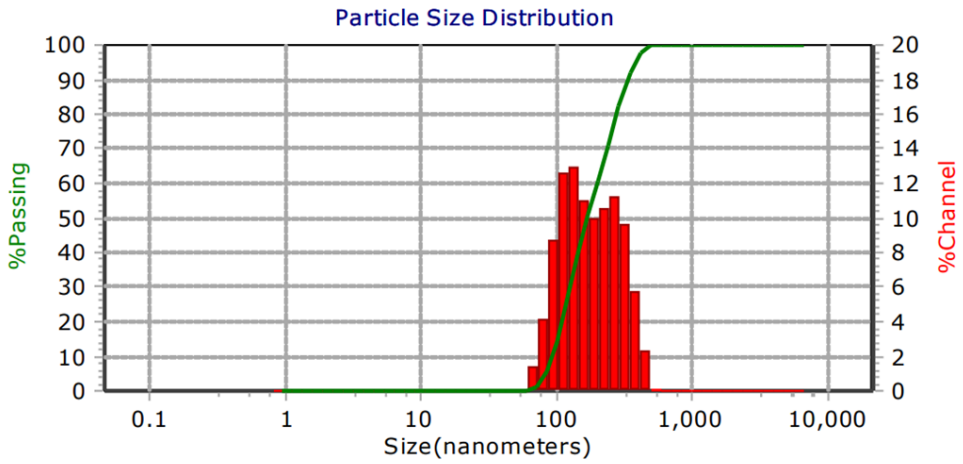

Figure 5 – Particle size analysis for CdS nanoparticles

Figure 5 presents the size distribution of nanomaterial particles determined using dynamic light scattering (DLS). Notably, this distribution differs from the one observed for CdS NPs at DSC 95.10 and 373.0 nm. The size distribution exhibits a width of 59.3 nm, a volume percentage of 50.6%, and an average diameter of 119.3 nm. It's crucial to emphasize that due to the aggregation of nanoparticles, the actual size exceeds the expected mean particle size. Larger particles in the sample have an impact on DLS measurements and tend to produce broader size distributions. The presence of larger-than-expected particle sizes suggests that the nanomaterials in the sample have a propensity to agglomerate. This consideration is crucial for the accurate interpretation of DLS data for these CdS NPs [18].

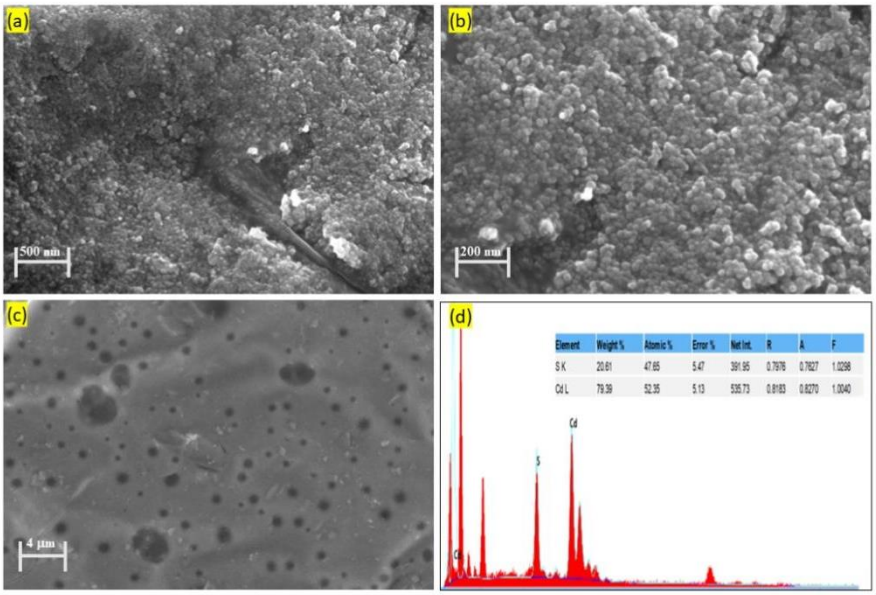

Figure 6 – FE-SEM images for prepared CdS nanoparticles with EDX

FE-SEM was used to look at the biosynthesized CdS NPs' sizes and shapes. FE-SEM was employed to look at the size and shape of the CdS nanoparticles made from Aegle Marmelos leaf extract. The CdS NPs are shown in FE-SEM images in Figures 6(a), (b), and (c), taken at different magnifications. The CdS NPs show well-crystallized, irregularly shaped particles in these SEM pictures. It is clear that the particle sizes have a tendency to form agglomerates and to exhibit a spherical appearance as they gradually rise in size. These findings imply that the biosynthesized CdS NPs have a tendency to group together and develop into bigger, spherical particles. The uneven shape of the particles suggests that the CdS NPs may not have grown and encapsulated uniformly during the synthesis process. It was also

confirmed that the CdS NPs were made by looking at the EDX spectrum. EDX spectroscopy serves as a robust tool for verifying nanoparticle composition. In Figure 6(d), we can observe the typical EDAX spectrum of the CdS NPs. The spectra demonstrated that the sample exclusively contained Cd and S, typically absorbing at 3–4 keV due to the surface plasmon resonance characteristic of metallic CdS NPs. The elemental peaks in the EDX spectra were always attributed to cadmium and sulphur, and their atomic weight fractions in the biosynthesized forms were very close to being 1:1 [19].
Furthermore, HR-TEM was used to look at the surface morphological features of the man-made CdS nanoparticles. TEM examination stands as the most precise method for assessing nanomaterial size. It provides insights into various morphologies, stabilisations, and dimensions of CdS NPs. The study's goal was to find out the size and shape of CdS nanoparticles made outside of cells using extract from Aegle marmelos leaves by using TEM. The transmission electron micrograph (Figure 5) clearly reveals that these particles exhibit a spherical morphology. Additionally, the TEM measurements indicate an average size of 95.10 nm. The TEM analysis shows that these CdS nanoparticles have a narrow size range, which suggests that they were made in the same way every time. The spherical morphology of these CdS nanoparticles is crucial for their applications in various fields. This particular form enables an elevated surface area-to-volume proportion, which amplifies their catalytic effectiveness and electrical conductivity. Moreover, the narrow size range ensures consistent performance and reproducibility, making them ideal candidates for targeted drug delivery systems in medicine [20].

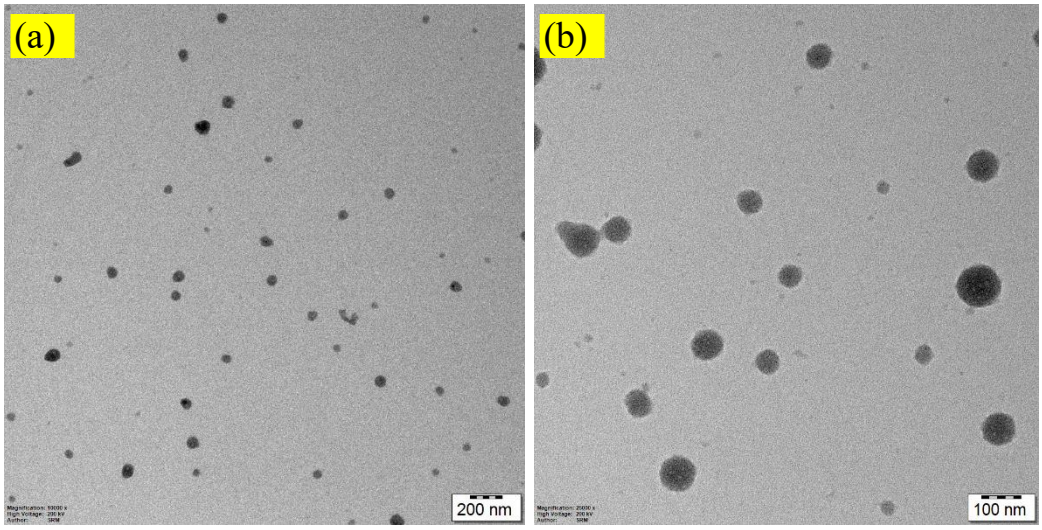

Figure 7 - HR-TEM images for synthesised CdS nanoparticles with SAED pattern.

4. Conclusion

This study's environmentally friendly technique of producing CdS nanoparticles involved employing Aegle marmelos leaf extract as a stabilising agent to reduce the size of the cadmium sulphide particles. Using Aegle marmelos leaves as a binding agent has simplified, reduced costs, and improved environmental safety while producing cadmium sulphide nanoparticles. While SEM data may be used to evaluate nanoparticle size, XRD data on nanoparticles can be used to ascertain the size of the crystal. Crucially, the ecologically benign procedure of producing these cadmium sulphide nanoparticles in manufacturing processes is ensured by the absence of poisonous or dangerous sealing agents.
Additionally, it is crucial to bear in mind that alterations in the capping agent's concentration could impact the size and dispersion of the crystals. A thorough examination of both crystal and nanoparticle sizes is also possible with the use of nanoparticle XRD and SEM data, leading to a more precise characterization of the nanoparticles. Furthermore, to maximise the synthesis process and get the

appropriate nanoparticle characteristics, it is essential to comprehend that the concentration of the capping agent affects crystal size and dispersion.

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