

CHARACTERIZATION TECHNIQUES OF CYPERMETHRIN NANOPARTICLES

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Abstract

The nanostructures have been characterized for their structural, morphological, and chemical properties. The techniques employed for the characterization of cypermethrin nanoparticles (CypNPs) were Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM), and UV-Vis Spectroscopy. The characterization of materials such as elemental composition, estimation of trace impurities, structural analysis, morphological analysis, magnetic analysis, identification of crystalline phases and information on crystal defects were performed to understand the toxicity potential of CypNPs. A clean, rapid and eco-friendly CypNPs (Cypermethrin Nanoparticles) can be developed to enhance the properties and to overcome the side effects of conventional cypermethrin (Cyp). Majority of the CypNPs (Cypermethrin Nanoparticles) are spherical and irregular in shape and ranged from 29.23 nm to 49.28nm in size. SEM pattern revealed the irregular surface of CypNPs and monodispersity is confirmed by TEM analysis. These technologies promise to revolutionize the whole range of novel fundamental and applied frontiers in nanobiotechnology, applied microbiology and agricultural practices

Keywords: Cypermethrin nanoparticles, Transmission Electron Microscopy, Scanning Electron Microscopy (SEM), UV-Vis Spectroscopy.

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Experimental and results-

Scanning Electron Microscope (SEM)

In SEM technique, accelerated electrons are generated due to applying a high voltage across the anode and cathode terminal of filament; carry a significant amount of kinetic energy. The kinetic energy of these electrons is dissipated after interaction with the sample and produced several significant signals. These signals that derive from electron-sample interactions are responsible for the formation of images in three dimensions (Hayat *et al.*, 2012). These signals include secondary electrons (that produce SEM images), backscattered electrons (BSE), diffracted backscattered electrons (EBSD that are used to determine the crystal structures and orientations of minerals, photons (characteristic X-rays that are used for elemental analysis), visible light (cathodoluminescence-CL) and heat. Secondary and backscattered electrons are commonly used for imaging samples, secondary electrons are most valuable for showing morphology and topography of samples and backscattered electrons are most valuable for illustrating contrasts in the composition in multiphase samples

(i.e. for rapid phase discrimination), Fig. 1 shows the schematic of electron beam sample interaction.





X-ray generation is produced by inelastic collisions of the incident electrons with electrons in discrete orbital's (shells) of atoms in the sample. As the excited electrons return to lower energy states, they yield X-rays that are of a fixed wavelength (that is related to the difference in energy levels of electrons in different shells for a given element). Thus, characteristic X-rays are produced for each element in a mineral that is "excited" by the electron beam. SEM analysis is considered to be "non-destructive"; that is, X-rays generated by electron interactions do not lead to volume loss of the sample, so it is possible to analyze the same materials repeatedly.



Figure. 2: Schematic of Scanning Electron Microscopy

Transmission Electron Microscopy (TEM)

TEM is used for characterizing and measuring the electric, thermodynamic and mechanical properties of individual nanostructures, from which the structural property relationship can be registered with a specific nanoparticles structure (Wang, 2000; Nikkuni *et al.*, 2014). TEMs are capable of imaging at a significantly higher resolution than light microscopes, owing to the small de Broglie wavelength of electrons. This enables the instrument's user to examine fine detail, even as small as a single column of atoms, which is tens of thousands times smaller than the smallest resolvable object in a light microscope. TEM forms a major analysis method in a range of scientific fields, in both physical and biological sciences. At smaller magnifications TEM image contrast is due to absorption of electrons in the material due to the thickness and composition of the material. At higher magnifications complex wave interactions modulate the intensity of the image, requiring expert analysis of observed images. Alternate modes allow TEM to observe modulations in chemical identity, crystal orientation, electronic and structure.

TEM is an advanced technique which is used to examine the composition, structure, and properties of specimens in submicron level. This technique is based on the principle of

interaction of a beam of transmitted electrons through an ultra-thin specimen. As a result of electron interaction, an image is formed which is magnified and focused onto an imaging device to obtain the image. TEM used electron as "light source" and their much lower wavelength makes it possible to get a resolution a thousand times better than with a light microscope. Due to its major contribution in the characterization of nanoparticles, it has gained the prime importance in nanotechnology applications. The most important application of TEM is the atomic-resolution real-space imaging of nanoparticles.

TEM is a microscopy technique whereby a beam of electrons is transmitted through an ultrathin specimen and electrons are interacting with the specimen as it passes through. An image is formed from the interaction of the electrons transmitted through the specimen; the image is magnified and focused onto an imaging device such as a fluorescent screen or to be detected by a sensor such as a CCD camera. The TEM is composed of various components which include a vacuum system in which the electrons travel an electron emission source for generation of the electron stream, a series of electromagnetic lenses, and electrostatic plates (Fig. 3).



Figure. 3: Schematic of Transmission Electron Microscopy

Ultraviolet-Visible Spectroscopy

Ultraviolet–visible (UV-Vis) spectroscopy refers to absorption or reflectance spectroscopy in the ultraviolet to visible spectral region (300 nm – 900 nm). UV-Vis spectroscopy is a type of absorption spectroscopy in which light of the ultra-violet region (200 nm – 400 nm) is absorbed by the molecule; Fig. 4 shows the schematic representation of UV-Vis Spectroscopy. Molecules containing π -electrons or non-bonding electrons (n-electrons) can absorb the energy in the form of ultraviolet or visible light to excite these electrons to higher anti-bonding molecular orbitals. This is commonly used to quantitatively analyses of the elements and compounds for structural elucidation of matter at the atomic and molecular levels (Chu *et al.*, 2011). Absorption of energy leads to a transition of an electron from the ground state to an excited state. UV-V is spectrophotometer principle which follows the Beer-Lambert Law. This law states that whenever a beam of monochromatic light is passed through a solution with an absorbing substance, the decreasing rate of the radiation intensity along with the thickness of the absorbing solution is actually proportional to the concentration of the solution and the incident radiation.



Figure. 4: Schematic of UV-Vis Spectroscopy

Conclusion

A clean, rapid and eco-friendly CypNPs (*Cypermethrin Nanoparticles*) can be developed to enhance the properties and to overcome the side effects of conventional cypermethrin (Cyp). Majority of the CypNPs (*Cypermethrin Nanoparticles*) are spherical and irregular in shape and ranged from 29.23 nm to 49.28nm in size. SEM pattern revealed the irregular surface of CypNPs and monodispersity is confirmed by TEM analysis.

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