



# A Review on Nanotechnology: Analytical Techniques Use and Applications

Tassew Belete Bahru<sup>1\*</sup> and Eyasu Gebrie Ajebe<sup>1</sup>

<sup>1</sup>Department of Chemistry (Analytical Chemistry), College of Natural and Computational Sciences, Wolkite University, Wolkite, Ethiopia.

## Authors' contributions

This work was carried out in collaboration among all authors. All authors read and approved the final manuscript.

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## ABSTRACT

The combination of nanotechnology with molecular biology, information technology and instrumentation, is opening the door to a new industrial age. The aim of this review article is to summarize the current knowledge of nanotechnology in synthesizing, identifying and characterization of nanomaterials using analytical techniques. Physical and chemical approach synthesis of nanomaterials befalls challenges in the development of analytical techniques used to characterize them. The major techniques include: Transmission Electron Microscopy, Scanning Electron Microscopy, Atomic Force Microscopy, Dynamic Light Scattering, X ray Photoelectron Spectroscopy, X-ray Diffraction, Single Phase Inductively Coupled Plasma Mass Spectroscopy, X ray Fluorescence Spectroscopy, Auger Electron Spectroscopy, X ray Absorption Fine Structure, Capillary Electrophoretic Separations, Magnetic nano particles coupled HPLC and Dynamic light scattering. Nanomaterials have been characterized for the extensive potential applications in optics, electronics, magnetism, and catalysts; chemical sensing, biomedicine, micro reactor, and they have been applied in food, biological, environmental and pharmaceuticals uses. In spite of the extended use of nano particles in diverse consumer products, there is a great concern over the unexpected impact or effects on humans due to exposure.

\*Corresponding author: E-mail: [tasiyye@gmail.com](mailto:tasiyye@gmail.com);

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## 1. INTRODUCTION

Over the last decade, nano particles [1,2] synthesis became most active areas due to their unique properties [3,4,5] and applicability in optics, electronics, magnetics, catalysts, chemical sensing, biomedicine, micro reactor etc. [6,7,8]. They have discrete functional parts with one or more dimensions of 100 nm or less [9,10]. The creation of nano particle falls within material chemistry today [4]. They can be synthesized using “bottom up” or “top down” approaches [11,12,13]. Literature indicates that, techniques such as chemical vapour condensation, pulse electron deposition, plasma synthesis, crystallization of amorphous solid, severe plastic deformation, and consolidation of mechanically alloyed or cryomilled powders are used to synthesize nanomaterials [14]. Super critical fluid technology offers an effective production of polymeric nano particles by avoiding the use of organic solvents [15,16].

Nanotechnology has been applied in different areas. For example, researchers investigated that, the food agriculture through the effectiveness of pesticide in a case if very small amounts are enclosed in nano capsules [17]. On the other hand, there is DNA detection assay, biomarker discovery, cancer diagnosis and detection of infectious microorganisms [18]. Recently, reviewer [19] suggested that, the specific interactions of nano particles with metabolites or bio macromolecules help metabolomics spectra by molecular characterization or in improving the ionization efficiency of mass spectrometry or reveal relationships between spectral signals that belong to the same molecule [20]. Other application are in separation science and miniaturized techniques. Some of them is, ultra-dilute polymer solutions (for long DNA) in microchip electrophoresis, gold nano particles (stationary phases) in gas chromatography with monolayer protected nano particles [20], EAG, 2007.

As reviewed by Stefanos et al. [21] nanostructures have attracted huge interest as a rapidly growing class of materials for many applications. Analytical techniques have been used to characterize the size, crystal structure, elemental composition and a variety of other physical properties of nano particles. In several cases, there are physical properties that can be evaluated by more than one technique. Different

strengths and limitations of each technique complicate the choice of the most suitable method, while often a combinatorial characterization approach is needed [21] As indicated in some findings of the researchers, the development of analytical tools for nanotechnology are in progress and contrasting to considerable context of the trusted data [22]. As reviewed by Enisa and Mirjana, there have been varieties of biological and toxicological interactions of nanomaterials *in vitro* and *in vivo* experimental systems [23,18].

A large body of data concerning the development of physicochemical characterization of nano material is a fundamental issue for coming years [24] because; sophisticated analytical techniques (instruments) have being emerged. Researchers have been directed nanomaterials to pro inflammatory and inflammatory markers since existing knowledge on the health effects of ambient fine particulates, which identified a central role for oxidative stress and inflammation in the toxicological mode of action [24]. There can be problems and issues faced during various *in vitro* and *in vivo* studies concerning nano materials. Researchers had tried to identify a solution as well as becoming alert thus, saving time and effort [25].

The objectives of this review article are to mention the analytical techniques employed in nanotechnology, to summarize the current knowledge of nanotechnology, to explain the synthesizing method of nanomaterials and to identify the applications of nanotechnology in different areas.

## 2. ANALYTICAL TECHNIQUES EMPLOYED IN NANOTECHNOLOGY

For characterization of nano materials, a number of researches have been conducted. Among them [1], summarizes the Engineered Nano Materials (ENMs) in foods and the suitability of existing instrumentation for identification and quantification [26]. The recent advances in the development of electrochemical sensors and biosensors are based on the click chemistry fictionalization [27]. There is development of novel drug delivery systems using nano particles [16]. The nano-based analytical methods have been developed to control analytes of interest in foods. As studied by Enisa and Mirjana [28,23]. Modern techniques were developed to meet the

different requirements in food inspection Furthermore, (Kanchi et al. 2014) reported that, there is novel routes in the development of new nano materials which are used for the detection of organic and inorganic pollutants [29].

The small size and large surface area to volume ratio of nanomaterials results in increased rates of oxidation and subsequent dissolution compared to larger-scale forms of silver [30,31]. Researchers had assessed the problem of selecting the suitable method for the preparation of nano particles [32]. Nano sensors contribute to the specificity, sensitivity, and performance of the methods and improved by using nano materials for their construction [23,33]. The nanotechnology based biosensor or nano biosensor technology is revolutionizing the health care industry such as the nano biosensor technology which is used in the measurement of metabolites, monitoring of diabetes forensic medicine, homeland security [34]. Additionally, methodologies used to characterize the composition, morphology and biological properties of synthesized nano particles by multiple techniques have been presented [35].

Different researchers reviewed that, analytical tools are deleveraged and applied [3,36] to generate predictive modeling determinations during development. Techniques for predictive in vivo information include those that measure surface chemistry at nano, micro, and macro scales for both inorganic and organic particles [37,38]. Structural characterization is essential for nanomaterials research [39]. Posth et al. [40] have assessed common magnetic nano particle analytical techniques under various criteria in order to define the methods that can be used as either standard technique for magnetic particle or those that can be used to obtain a comprehensive picture of a magnetic nano particle system [40].

## **2.1 Transmission Electron Microscopy (TEM)**

Materials that have dimensions small enough to be electron transparent, such as powders or nano tubes, can be quickly produced by the deposition of a dilute sample containing the specimen onto support grids [41,42]. The electron microscope uses electrostatic and electromagnetic "lenses" to control the electron beam and focus it to form an image [43].

Researchers have been used a technique whereby a beam of electrons is transmitted

through an ultra thin specimen and interacts as passes through the sample. An image is formed from the electrons transmitted through the specimen, magnified and focused by an objective lens and appears on imaging screen. It is sensitive to extended crystal lattice defects. The specimens must be prepared as a thin foil so that the electron beam can penetrate.

## **2.2 Scanning Electron Microscopy (SEM)**

This technique creates magnified images using electrons instead of light waves [41,44,31,12]. When the beam of electrons interacts with the atoms of the sample, signals in the form of secondary electrons, back scattered electrons and characteristic X- rays are generated that contain information about the sample's surface topography, composition etc. It can also produce very high-resolution images of a sample surface, revealing details about 1-5 nm in size in its primary detection mode i.e. secondary electron imaging. Detectors collect these X rays, backscattered electrons, and secondary electrons and convert them into a signal that is sent to a screen similar to a television screen.

## **2.3 Auger Electron Spectroscopy (AES)**

In this technique, after an inner shell excitation which can be caused by a collision with a primary electron that has an atom in an energy level above its ground state. The atom returns to its ground state by filling the empty level created, with an electron from a higher energy level and this leads to the release of energy and momentum. It uses a primary electron beam to probe the surface of a sample. The electron impact results in a disturbance of the sample in a region of about 1- 3 $\mu$ m depth, leading to the emission of secondary electrons, backscattered electrons, Auger electrons and characteristic X-rays. It focuses only on the small part of secondary electrons that are emitted because of the auger process. The identity and quantity of the elements are determined from the kinetic energy and intensity of the auger peaks, which occur at specific kinetic energies in the auger-spectrum. To separate and locate the auger peaks from the intense background of scattered electrons the signal is deviated [45,46]. It is used to quantify and qualify the elements.

## **2.4 X-ray Photoelectron Spectroscopy**

XPS is used to determine the elements and the quality of those elements that are present within ~ 10 nm of the sample surface. It also detects

the contamination, if any, exists in the surface or the bulk of the sample. If the material is free of excessive surface contamination, XPS can generate empirical formula of the sample and the chemical state of one or more of the elements can be identified. It can be also used to determine the thickness of one or more thin layers (1-8 nm) of different materials within the top 10 nm of the surface. It can also measure the uniformity of elemental composition of textile surfaces after nano level etching, finishing, or coating of the surfaces [31].

## 2.5 X-ray Fluorescence Spectroscopy

It is similar to XPS in terms of the excitation mechanism but differs in its detection mechanism. While XPS detects photoelectrons, XFS detects "secondary" or "fluorescent" X-rays from a material that has been excited by high-energy X-rays (or sometimes  $\gamma$ -rays) (EPA, 2015). The principle behind XFS is relatively straightforward. When a material is exposed to high energy X-rays, ionization or electron ejection can take place if the X-ray photon energy is greater than its ionization energy. Due to the high energy of X-rays or  $\gamma$ -rays, tightly bound electrons in the inner, low energy orbitals of the atom in the material can be expelled. The resulting ionised atom is not unstable and electrons in outer, higher energy orbitals may fall or make a transition into the lower orbital to fill the hole left behind. In doing so, energy may be released in the form of a photon usually with energy in the X-ray region still with energy equal to the energy difference of the two orbitals involved. The X-rays penetrates up to a depth of about 1-10  $\mu\text{m}$ . Only photo-ionised electrons of the first atomic layers (1-10  $\mu\text{m}$ ) can leave the solid without significance energy loss, pass an electrostatic energy analyzer and reach the electron detector. The measured kinetic energy of these photoelectrons depends on the photon energy and the binding energy of the atomic orbital from which the electrons originate [42].

## 2.6 Atomic Force Microscope (AFM)

AFM is an ideal for quantitatively measuring the nanometer scale surface roughness and for visualizing the surface nano-texture on many types of material surfaces including polymer nano composites and nano finished or nano-coated textiles. In AFM a probe consisting of a sharp tip (nominal tip radius is in the order of 10 nm) located near the end of a cantilever beam is raster scanned across the surface of a specimen

using piezoelectric scanners. Changes in the tip specimen interaction are often monitored using an optical lever detection system, in which a laser is reflected off the cantilever and onto a position sensitive photodiode. AFM can be used to explore the nano structures, properties, surfaces, and interfaces of fibers and fabrics. For example, structural characteristics of nano fiber materials and nano level surface modification [41,44]. For non conductive nano materials, AFM is a better choice for analyses Bennig et al. 1986, [47].

## 2.7 Single-particle ICP-MS

Mitrano et al, [48] reported that, due to its sensitivity, flexibility, and analytical speed, ICP-MS performed in single-particle mode (SP-ICP-MS) is gaining popularity for detecting and measuring inorganic-based engineered nano materials (ENM), as traditional inductively coupled plasma mass spectroscopy (ICP-MS) already does, but with the important distinction that single-particle mode has the ability to detect a difference between ionic and whole-ENM elemental sources (Andrea et al. 2015). In single-particle mode, ENMs are introduced into an ICP and completely ionised, with the resulting ions being detected by a mass spectrometer. To ensure that only a single particle is measured, the sample has to be diluted to have temporal resolution between particles that only a single particle is introduced into the plasma at a time. The mass spectrometer must also be capable of making extremely rapid measurements to ensure ENM detection (data should be acquired at least every 100  $\mu\text{s}$ ) as the transient signal of a 50-nm gold nano particle can vary, on average, between 600 and 800  $\mu\text{s}$  depending on the instrument operating conditions and ion optics design.

## 2.8 X-ray Absorption Fine Structure

XAFS is used to measure the fine structure of an analyte near the absorption edge when subjected to X-ray radiation. It is similar to UV-visible electronic absorption spectroscopy, in principle, except that the spectral range is in the X-ray region and electronic-XAFS focuses on the fine structure specifically since it provides local structural information about specific atoms or ions. EXAFS relates to the details of how X-rays are absorbed by an atom at energies near and above the core-level binding energies of that atom. EXAFS measurements reflect the modulation of an atom's X-ray absorption probability due to the chemical and physical

states of the atom. EXAFS spectra are especially sensitive to the formal oxidation state, coordination chemistry, and the local atomic structure of the selected element. One advantage of EXAFS is that it works for crystalline as well as non crystalline or even highly disordered materials, including solutions (EPA, 2015).

## 2.9 Capillary Electrophoretic Separations

Yang-Wei Lin et al. (2005) had used Capillary electrophoresis (CE) and microchip capillary electrophoresis (MCE). These are two of the most powerful techniques for the analysis of DNA. They reported about DNA separation using chip-based nanostructures and nano materials in CE and MCE. Based on the dependence of the mobility of DNA molecules on the size and shape of nanostructures, several unique chip-based devices have been developed for the separation of DNA, particularly for long DNA molecules. Unlike conventional CE and MCE methods, sieving matrices are not required when using nanostructures [44].

## 2.10 Magnetic Nano Particles and HPLC

Laleh et al. (2017) had used a micro extraction method using Ag modified-magnetic nano particle (Ag-MNPs) coupled with high performance liquid chromatography (HPLC) for determination of cefterixone in plasma. They had synthesized magnetic nano particles via a mild solution route. The prepared nano particles were modified with a thin layer of silver and characterized with different methods such as X-ray diffraction (XRD), transmission electron microscopy (TEM), FTIR and ultraviolet-visible (UV-Vis) spectroscopy. Effect of several parameter such as pH of donor and acceptance phase, amount of magnetic particle and extraction and desorption time were optimized. They reported that, under the optimal condition the enrichment factor was obtained. The detection limit was 0.02 mg/mL and a wide linear range from 0.06 to 40 µg/mL were obtained.

## 2.11 Dynamic Light Scattering (DLS)

Sovan et al. [16] reviewed that currently, the fastest and most popular method of determining particle size is photon-correlation spectroscopy (PCS) or dynamic light scattering (DLS). DLS is widely used to determine the size of Brownian nano particles in colloidal suspensions in the nano and submicron ranges. Shining

monochromatic light (laser) onto a solution of spherical particles in Brownian motion causes a Doppler shift when the light hits the moving particle, changing the wavelength of the incoming light. This change is related to the size of the particle. It is possible to extract the size distribution and give a description of the particle's motion in the medium, measuring the diffusion coefficient of the particle and using the autocorrelation function. The photon correlation spectroscopy (PCS) represent the most frequently used technique for accurate estimation of the particle size and size distribution based on DLS (EPA).

## 3. APPLICATION OF NANOTECHNOLOGY

### 3.1 Nanotechnology in Electronics and Computing

Nanotechnology systems are useful in tailoring the magnetic, optical, and electronic properties of material. The nanomaterials will have an increasing impact on electronics, since the drive for increasingly smaller dimension in electronics translates into the requirement for higher functionality, increased memory density, and higher speed [49]. It is one of the very important technologies to make the electronics into Nano scale, so that the devices will make into more smart system. Since, the Nanotechnology is continually playing a vital role in improving the capability of electronic products. This technology also made the devices very light making the product easy to carry or move and at the same time, it has reduced the power requirement [50].

Magnetic random access memory (MRAM) enabled by nanometer scale magnetic tunnel junctions that can quickly and effectively save even encrypted data during a system shutdown or crash, enable resume play features, and gather vehicle accident data. Displays for many new TVs, laptop computers, cell phones, digital cameras, and other devices incorporate nano structured polymer films known as organic light emitting diodes, or OLEDs. OLED screens offer brighter images in a flat format, as well as wider viewing angles, lighter weight, better picture density, lower power consumption, and longer lifetimes. Other computing and electronic products include Flash memory chips for iPod nanos; Magnetic random access memory (MRAM) enabled by nanometer scale magnetic tunnel junctions that can quickly and effectively save even encrypted data during a system shutdown or crash, enable resume play features, and gather vehicle accident data [51].

### 3.2 Application in Food Analysis

Recently, technology with nano materials is being used repeatedly in a number of areas. Food is the variable on which the life is dependent and it is an exploratory means to analyze them at nano level. Nanotechnology tools are used in the entire food production chain. From cultivation (e.g. Pesticides), industrial processing to packaging foods [9,52,53]. To check the quality of food products at nano level, scientists have been participating in different researches using several analytical techniques. Accordingly, Xiang Zhang et al. [52] have used nanotechnology in selecting nano materials by developing fluorescent sensors for food pH based on nano particles and investigated water activity probes. The results shows that, quinine sensors exhibited blue shifts of emission spectra as pH increased; the ratio of peak intensity or peak area of emission spectra at two different emission wavelengths also decreased dramatically in the range of pH~3.0-5.0. In the study of Ruud JB, [54], food grade TiO<sub>2</sub> materials (E171), food products and personal care products were investigated for their TiO<sub>2</sub> content and the number-based size distribution of TiO<sub>2</sub> particles present in these products [54].

### 3.3 Application in Environmental Analysis

Fadri G, Tian Y, and Bernd N, [27] reviewed that, there are still major knowledge gaps (e.g. on ENM production, application and release) that affect the modeled values. In food safety, photo catalysis could find uses in cleansing the surface of fresh fruits and vegetables of toxic agrochemical residues and in destroying bacteria on such produce [55,48]. The surface-to-volume ratio increases drastically with the reduction of the size of the adsorbent particle from bulk to nano dimensions [56]. Shrivastava, [55] applied nano materials in the area of metallic and organic nano particle synthesis. They used these nano materials based chemistry for waste water treatment for three major types of contaminants: halogenated organics including pesticides, heavy metals and dyes.

Caur and Gupta (2009) applied nano materials for the pre-concentration of trace amounts of Ni(II) in different samples using 1-(2-pyridylazo)-2-naphthol modified SiO<sub>2</sub> nano particles as solid-phase extractant. They had optimized various parameters such as pre-concentration factor, effect of pH, sample volume, shaking time,

elution conditions and effects of interfering ions for the recovery of analyte. They had found adsorption capacity of SiO<sub>2</sub>-PAN nano particles to be 42.81 μmol g<sup>-1</sup> at optimum pH. They found detection limit (3σ) 0.43 μg L<sup>-1</sup>. They obtained the adsorption equilibrium of Ni (II) on SiO<sub>2</sub>-PAN nano particles within 10mins. Adsorbed Ni (II) was easily eluted with 5 mL of 6 molL<sup>-1</sup> HCl.

### 3.4 Applications in Biological Analysis

The unique optical and electrical properties of nanomaterials, such as gold nano particles, nano rods, quantum dots, carbon nanotubes, graphenes, nanopores, and polydiacetylene nanovesicles, are closely associated with their dimensions, which are comparable in scale to those of targeted biomolecules (Min C, and Young R, 2016). The category of biological nano materials is defined as materials of biological origin that are used for nano technological applications. There are a magnitude of different materials and approaches that are being investigated. However, altogether it seems that bio-analytical applications are most developed. Kun-Chan et al. [57] applied nano material by Using Bio functionalized nano particles to probe pathogenic bacteria [58]. Gold nano particle (GN) and embedded silicon nano wire (SiNW) configuration were applied for label-free DNA detection to enhance the sensitivity [31]. Such as antibacterial, antifungal, antiviral, anti-inflammatory, anti-cancer, and anti-angiogenic.

### 3.5 Application in Pharmaceutical Analysis

Nano medicine is the preservation and improvement of human health using molecular tools and molecular knowledge of the human body [7]. Thangaraja et al. [59] applied nano material by successfully synthesizing Polyethylene Glycol (PEG) coated silica nano particles and used to load the drug Ibuprofen. Yao Liu et al. (2003) applied nano material by investigating Poly-lactic-glycolic acid (PLGA) nano particles for sustained drug-release properties. They obtained the results of PLGA particles with 90% or greater efficiency (approx. 200 nm diameters) to incorporate the drug estradiol. Hirsjärvi, S. (2008) [60]. used nano particles by preparing from a biodegradable poly (lactic acid) (PLA) polymer. They studied the effect nano precipitation, a nano particle preparation method, on the physicochemical properties of the polymer and model drugs encapsulated in the nano particles as well as the

effect of the drugs on the polymer by thermo analytical and spectroscopic methods. The antimicrobial activity due to the treatment is found to be acceptable and fast to dry cleaning with loss in strength well within the industrial norms [61].

### 3.6 Agricultural Application

As stated by Waleed, [62], nanotechnology consider a novel key to growing agricultural production through implementing nutrient efficiency and improved plant protection practices, also, nanotechnology may have real solutions for various agriculture problems such as improved crop varieties, plant protection, detect diseases and monitor plant growth. In addition, nanotechnology may have a great future for the development agricultural sector through advanced applications and the probability of products and increased global crops production volume to feed the world population in next decades [62].

### 3.7 Future Opportunities and Challenges

Nanomaterials (NM) open huge prospects for innovation in different fields such as medicine, electronics, cosmetics, and materials. However, their uses raise questions about possible risks to the environment and humans [24]. Economies mostly without a strong science base competitiveness and agility in nanotechnology development demand sustained broad scientific base: such as lack of a strong industry base, lack of ability to translate investments into economic outcomes as industry dominated by multinationals. High nanotechnology costs for acquisition of intellectual property rights, nanotech infrastructure; lack of human and policy capacity; trade barriers, the political instability in constitute further barriers, although these are not unique to nanotech.

Low average incomes and low government spending on investment and even healthcare, in many worsens the scenario for nanotechnology to even be considered in less developed countries. Lack of a comprehensive standards infrastructure, data base or regulatory framework including protocols for risk and life cycle assessment for nanotechnologies globally scares less developed countries from wanting to adopt the innovation. This challenge is further exacerbated by a lack of tools to assure quality, evaluate, and mitigate hazards and risks. There are many technical, challenges in developing the

following techniques. Virus-like systems for intracellular systems, designing of bio mimetic polymers, control of sensitive drugs, functions of active drug targeting, bio responsive triggered systems. Most major and established internal research programs on drug delivery that are formulations and dispersion containing components down to nano sizes [63,57].

## 4. CONCLUSION

Nanotechnology is moving into the centre of worldwide because of its applications. The materials with nano meter size in one or three dimensions have the properties those are neither that of bulk materials nor that of molecular compounds. Synthesizing nano materials play a role for the development of analytical methods, to characterize the nano scale structure. Nanotechnology has been exploited for extensive potential applications to enhance life and sustenance in specific areas such as pharmaceutical developments and drug delivery biosensing, food security, quality assurance and different areas.

## COMPETING INTERESTS

Authors have declared that no competing interests exist.

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