Chapter

Diverse Synthesis and Characterization Techniques of Nanoparticles

Agnes Chinecherem Nkele and Fabian I. Ezema

Abstract

Nanoparticles are small particles that range from 1 to 100 nm in size, exhibit several physical and chemical features. An understanding of nanoparticles would reveal great qualities and potential applications that would aid the diversification of thin film technologies. The synthesis methods employed like top-down, bottom-up, chemical, biological, and mechanical processes have great influence on the properties exhibited by such nanomaterials. This review covers an insight into the knowledge of nanoparticles, their classifications, parameters affecting their efficient performance, synthesis and characterization techniques of nanoparticles. Nanoparticles are also characterized to obtain their morphological, structural, optical, elemental, size, and physiochemical features. The potential applications of nanoparticles have not been left undiscussed.

Keywords: nanoparticles, thin film technologies, synthesis, characterizations, applications

1. Introduction

Nanotechnology involves synthesizing and developing different nanomaterials. The field of nanotechnology allows different nanoparticles of unique features to be produced. Nanoparticles (NPs) are complex material particles that fall within the range of one to hundred nanometers. Their nanometer sizes drive the chemical, optical, physical, and electric features of the nanoparticles [1]. Naturally, nanoparticles can be sourced from geological, biological, meteorological, and cosmological means. However, nanoparticles can be created from liquid and solid materials by breaking down biopolymers, condensing gases, wet chemical process, implantation of ions, hydrothermal process, pyrolysis, radiolysis etc. Nanoparticles are usually viewed with the aid of electron microscopes, can penetrate filters, and have unique mechanical properties that distinguish them from the bulk materials. Nanoparticles exist in various shapes like nanorods, nanostars, nanofibers, nanospheres, nanoflowers, nanoboxes etc. [2].

Nanoparticles comprise a functionalized surface, a shell of different layered materials, and the core/main nanoparticle [3]. The features of materials in their bulk form are different from their nanoparticle forms because of the large area to volume ratio, interfacial layer, affinity to solvents, kind of coating, quantum mechanics effects, rate of diffusion, mechanical, and ferromagnetic features [1]. The large area to volume ratio makes the nanoparticles highly reactive and able to penetrate membranes. The chemical nature of nanoparticles should be studied to enhance their molecular attachment to surfaces.

2. Classification of nanoparticles

Nanoparticles may be metallic, non-metallic [1], anthropogenic, engineered, organic, or inorganic as outlined in **Figure 1**. Metallic nanoparticles include copper,

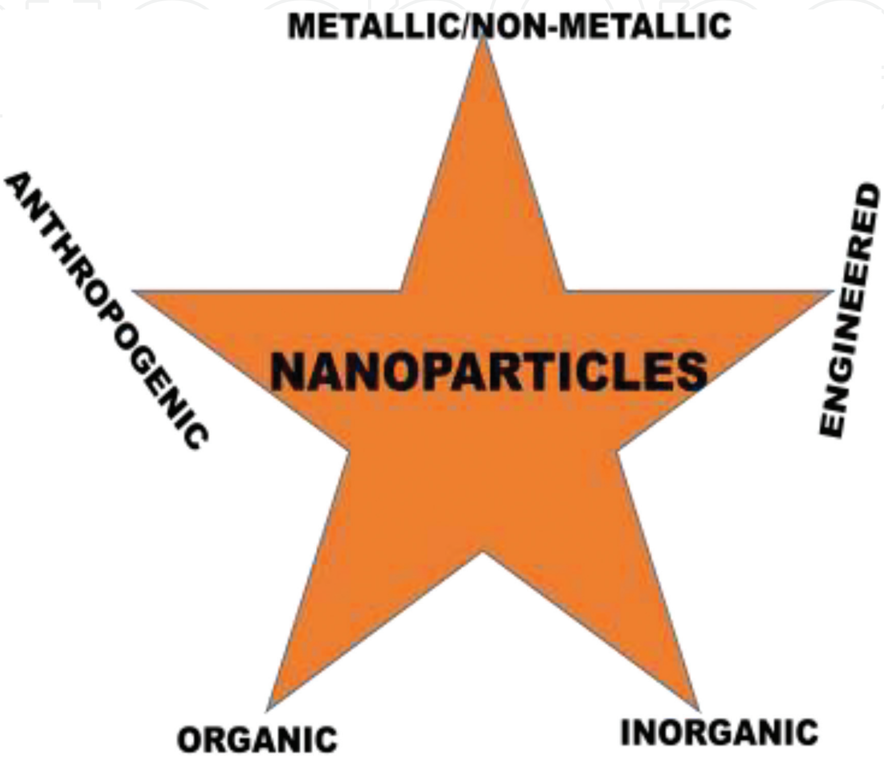

Figure 1.
Schematics on the classifications of nanoparticles.

Nanoparticles	Features
Silver	Very effective, high antimicrobial performance, wide range of usage
Gold	Good for identifying protein interactions, useful in tracing out fingerprints, detects antibiotics and cancerous cells, efficient for cancer diagnosis and other bacteria
Iron	Biocompatible and useful for treating cancer, sorting stem cells, analyzing genes, and drug delivery
Quantum dot	Diameters less than 10 nm, semiconducting nanoparticle, size-dependent
Carbon nanotubes	sp2 hybridized carbon atoms, strong electron bonds, high electrical conductivity, good catalysts.
Copper	Wide absorption spectrum, distinct optical features, yields good quality nanoparticles
Ceramics	Inorganic amorphous solids; could be polycrystalline, porous, amorphous or dense; vastly applied in photocatalysis, imaging devices etc.
Semiconductor	Large and tunable band gap nature, suitable in water splitting and electronic appliances.
Polymeric	Majorly organic components and easily functionalized
Lipid-based	Comprise lipid components, uses surfactants as core stabilizers

Table 1.
Some nanoparticles and their respective features.

magnesium, zinc, gold, titanium, silver etc.; while non-metallic nanoparticles include silica, carbon nanotubes etc. Anthropogenic NPs are by-products obtained from industrial produce while engineered nanoparticles are directly obtained from manufacturing processes.

Some of the nanoparticles and their features [2, 4] have been summarized in **Table 1**.

3. Synthesis techniques of nanoparticles

The techniques applied in synthesizing nanoparticles greatly influence their morphology, size, structure, and performance. The electrochemical, physiochemical, optical, and electrical features of the nanoparticles are also affected. In some occasions, nanoparticles are coated so as to retain their features after precipitating out of suspensions. The synthesis methods for nanoparticles are broadly divided into top-down and bottom-up approaches [4].

3.1 Top-down approach

Top-down method is a destructive method that breaks down large molecules into smaller parts before converting into the relevant nanoparticles. This approach involves some decomposition strategies like chemical vapor deposition (CVD), milling process, and physical vapor deposition (PVD). Milling is used to extract nanoparticles from coconut shells with the crystallite size reducing with increasing time. Nanoparticles of iron oxide, carbon, dichalcogenides, cobalt (III) oxide have been produced using this method.

3.2 Bottom-up approach

This approach involves the formation of nanoparticles from simple materials in a build-up manner. It is environmentally friendly, less poisonous, feasible, and of low cost. The materials used are usually Reduction and sedimentation processes like green synthesis, bio-chemical, spin coating, sol–gel etc. adopt this approach. Nanoparticles of titanium dioxide, gold, bismuth have been synthesized via this approach. The reaction chain for the production of gold nanoparticle has been illustrated in **Figure 2** [5].

Synthesizing nanoparticles could also involve chemical or biological processes [1]. Some chemical synthesis techniques of nanoparticles include sol–gel method, wet chemical synthesis, hydrothermal method, thermal decomposition, microwave method etc. [2]; while the biological means involve enzymes, microorganisms, plant extracts, and fungi.

3.3 Chemical methods

Some chemical methods adopted in synthesizing nanoparticles include sol gel, precipitation, hydrothermal, thermal decomposition, solvothermal, vapor synthesis etc. [6, 7]. Sol–gel method is an easy means of producing nanostructures by homogenously mixing precursors in a solvent to form a gel material which is then heated to produce the required nanoparticle. It begins from preparing a sol which undergoes gelation process to solvent removal. Wet chemical/precipitation method is a fast and easy process for synthesizing large scale nanoparticles. Hydrothermal method utilizes high pressure and temperature to power heterogeneous reactions under aqueous solvents like water. The kind of pressure, pH, and temperature

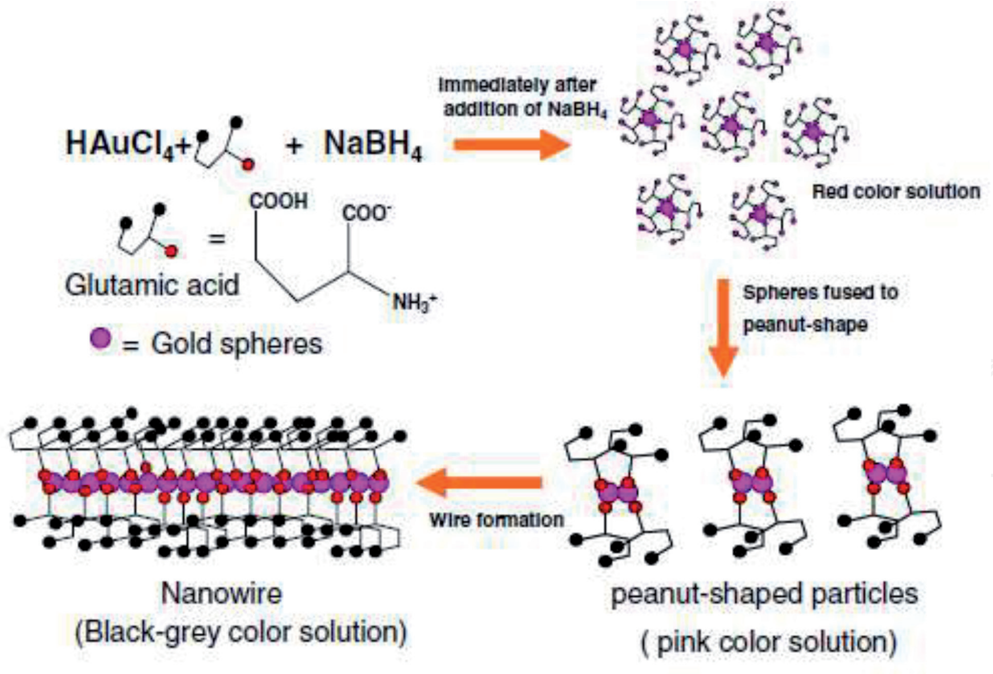

Figure 2.
Formation process of gold nanoparticle.

applied affects the features of the synthesized nanoparticles. Such nanoparticles are suitable for biotechnological use because of their hydrophilic surface nature [8]. Thermal decomposition involves oxidizing a solid material in optimal temperature. Solvothermal method uses a solvent to produce various materials like polymers, semiconductors, or metals at moderate or high pressure [9]. It produces novel and stable nanoparticles with controlled thicknesses and temperature. To synthesize nanodots; the cationic source is dissolved in suitable solvent alongside a surfactant which stabilizes the growth rate. Cadmium selenide, zinc oxide, zinc selenide are producible using this method and can be applied in magnetic and biotech industries [10]. In vapor synthesis, gaseous molecules chemically react to produce a phase which condenses and leads to particle growth. The higher the temperature, the faster the particles are formed. Different means of inducing homogenous nucleation include condensing inert gases, vaporizing a supersaturated material using a pulsed laser, generating a spark discharge by charging electrodes, sputtering the material with unreactive gaseous ions; or through some chemical methods like chemical vapor deposition, photothermal method, flame synthesis, or spray pyrolysis [11]. This method suitably yields nanoparticles of titania, carbon, and silica. Flame synthesis is commonly used to commercially produce silica, carbon black, optical fiber, and titania [12]. Particles produced by converting gases in furnace reactors or hot walls are usually very pure, although it produces agglomerated particles.

3.4 Biological methods

Biological or biosynthesis of nanoparticles is an environmentally-friendly, green, and non-toxic method involving microorganisms [13–15]. Nanoparticles of iron oxide, silver, nickel oxide, copper oxide, zinc ferrite have been synthesized using this method [16–22]. The location of the nanoparticle determines the point of synthesis; whether intracellular or extracellular [1]. Intracellular production of nanoparticles uses enzymes to move ions into the cells of microbes and produces smaller sized nanoparticles in the organism. Extracellular synthesis does not involve

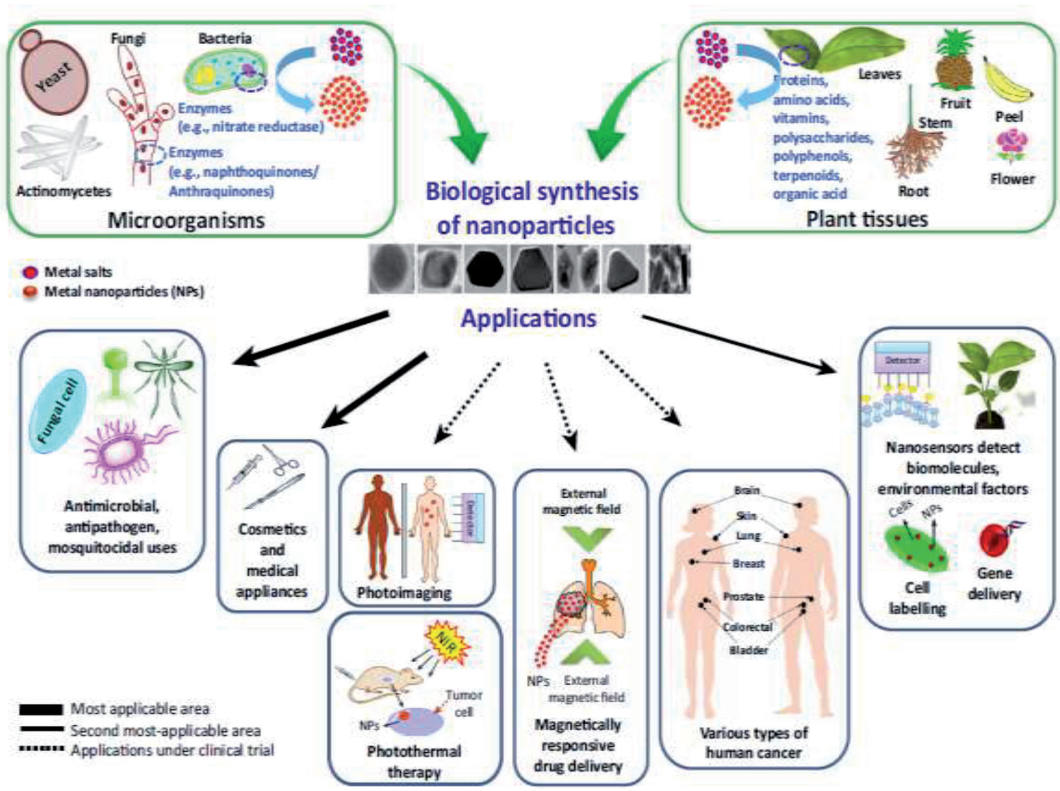

Figure 3.
Diverse bio-development synthesis of nanoparticles and their application areas.

cell components and yields nanoparticles outside the cell, uses fungi with large secretory organs. Microbes like fungi and bacteria are responsible for controlling the synthesis process. Microorganisms are immensely used to produce nanoparticles because of their economical, non-poisonous nature, and detoxification of heavy metal power. Phytonanotechnology is compatible with biological systems, available source materials, high stability, and entails synthesizing nanoparticles from plants [23]. Changes in the pH level of plants alter their binding strength, morphology, and the number of metallic ions available during the synthesis. The different sources, synthesis methods, and areas of application of nanoparticles have been represented in **Figure 3** [23]. Biogenic means of producing nanoparticles are green and cheap; with the involvement of fungi, waste materials, and bacteria [5].

3.5 Mechanical methods

Nanoparticles can also be synthesized by mechanical methods like mechanical alloying, milling, and mechanochemical processes [24]. Milling method regenerates interfacial chemical operations at low temperatures. Mechanochemical technique involves continuous welding operations that adequately select milling materials and minimize agglomerations. For effective production; the stoichiometry of source materials, thermal treatment, paths for reaction to occur, and milling conditions would be carefully considered. Nanoparticles of oxides, iron, nickel, silver, cobalt can be synthesized using these methods.

4. Characterization methods for nanoparticles

Properties of nanoparticles like shape, size, surface morphology, crystalline nature, light absorption etc. need to be completely described using relevant characterization techniques [2]. Some of the methods used to characterize nanoparticles [4] include:

4.1 Morphological features

The morphology of nanoparticles greatly influence the properties exhibited by nanoparticles. Microscopy methods applied on nanoparticles are usually electron microscopy or scanning probe microscopy. Scanning electron microscope (SEM) gives nanoscale and surface information of the dispersion and morphology of nanoparticles. Microscopy techniques are destructive and used for single-particle measurements. Transmission electron microscopy (TEM) uses transmittance of electrons to provide bulk information at high and low magnifications. Optical microscopic technique is not useful for nanoparticles because the size of nanoparticles is smaller than light diffraction limit. Coupling spectroscopic techniques to electron microscopes would enable elemental studies to be carried out.

4.2 Optical studies

Optical methods reveal reflectance, transmittance, photochemical, and luminescence features of nanoparticles. Spectroscopy uses the interaction of particles with electromagnetic radiation to determine the shape, concentration, and size of nanoparticles. Spectroscopic techniques like infrared, ultraviolet–visible, photoluminescence (PL), UV/vis-diffuse reflectance spectrometer (DRS), and magnetic resonance methods are applied to nanoparticles. DRS is specially used to determine the band gap energy of nanoparticles. PL studies reveal the effect of emissivity and absorptivity on the excitation of photons, half-life, and recombining effects of the charges. The sizes of nanoparticles affect their optical features and make it useful in bioimaging devices [4].

4.3 Structural analysis

The structure of nanoparticles gives details about the kind of bond existing between the atoms and the features of the bulk material. Some of the structural techniques used on nanoparticles include BET, X-ray diffractometry (XRD), IR etc. XRD describes the phase, particle size, type of NP, and crystal nature of the nanoparticles.

4.4 Elemental studies

The elemental composition of nanoparticles can be determined using energy dispersive X-ray spectroscopy (EDX), XPS, Raman, FT-IR etc. EDX details the elemental components of bulk particles. Better contrast is obtainable when the obtained spectra are compared with a computer generated model. XPS is a very sensitive spectroscopic method used to obtain the exact compositional ratio of the elements, their bonding nature, depth profile analysis. Raman and FTIR techniques use vibrational methods to show functionalized peaks and particle information.

4.5 Size estimation

Sizes of nanoparticles can be estimated using scanning electron microscope, transmission electron microscope, X-ray diffractometer, atomic force microscope etc. The sizes of the nanoparticles are obtained using size distribution profiles and give more precise results when used alongside digital models. The surface area can be estimated using BET via adsorption and desorption processes.

4.6 Physiochemical characteristics

Mechanical properties, optical activity, surface area, and chemical reactions of nanoparticles are physiochemical characteristics obtainable from nanoparticles. Free surface electrons on nanoparticles are very mobile and are not scattered upon light illumination. The magnetic features of NPs are manifested at small nanoscales due to their uneven distribution, influenced by the synthesis technique adopted, and find vast application in biomedicine, resonance imaging, and catalytic devices. Mechanical characteristics of nanoparticles like stress, surface coatings, hardness, strain, friction, adhesiveness etc. aid an understanding of NPs and greatly affect the quality of the surface. Nanoparticles have great conduction to heat especially on the surface.

5. Application areas of nanoparticles

Generally, nanoparticles have been applied in various areas including anticancer drugs, vaccines, disease treatment, cancer diagnosis, mechanical factories, electronics, optical devices, energy harvesters, manufacturing processes, cell imaging, and delivery systems due to their unique features [4]. NPs also aid water contaminants to be absorbed on the surface during water purification, serve as environmental sensors, and protect materials from harmful substances. Some of the application areas of the nanoparticles [2, 23] have been summarized in **Table 2**.

Despite the numerous applications of NPs; they suffer from poisonous and harmful body effects which inhaled, ingested, or transferred to the ground and surrounding environs. Nanoparticles are also affected by organic materials which lead to agglomeration. The poisonous effects associated with NP synthesis can be curtailed by adopting green synthesis methods especially in the synthesis of silver,

Nanoparticles	Application areas
Nickel oxide	Dye sensitized solar cells, supercapacitors, batteries, water treatment and catalytic systems, gas sensing devices.
Carbon nanotubes	Integrated circuits, electronic components, textile, construction, cosmetics, medicine
Cerium oxide	Biomedical equipments, electronic appliances, energy devices
Titanium dioxide	Coatings, water purifiers, paints
Silver	Clothing, textile industries, food packaging companies, agriculture, automotives, electronics, medicine, and fitness centers.
Iron	Optical devices, water purifier
Calcium	Agriculture, automotives, food
Zinc oxide	Agriculture, automotives, cosmetics, home appliances, food
Gold	Cosmetics, environmental products, food, medicine
Palladium	Automotive, electronic appliances, food

Table 2.
Application areas of some nanoparticles.

iron, copper, gold nanoparticles amongst others [25]. The synthesis process for silver nanoparticles is as shown in **Figure 4** [25].

Green synthesis involves different capping substances like biomolecules and polysaccharides. Green methods are non-poisonous, environmentally friendly, involve toxic-free solvents, compatible in biological systems, and utilize reagents like sugars, polymers, vitamins, plant extracts [26]. Plant-based extracts like latex, leaf, seed, root, or stem are more suited for bioprocesses as they are cheap, non-complex, easily reproduced, and highly stable. Other sources of waste materials useful for nanoparticle production have been outlined in **Figure 5**. Models can be developed to minimize the difficulties associated with distributing the size of the particles and NPs synthesis by computing the rates at which the particles get nucleated [11].

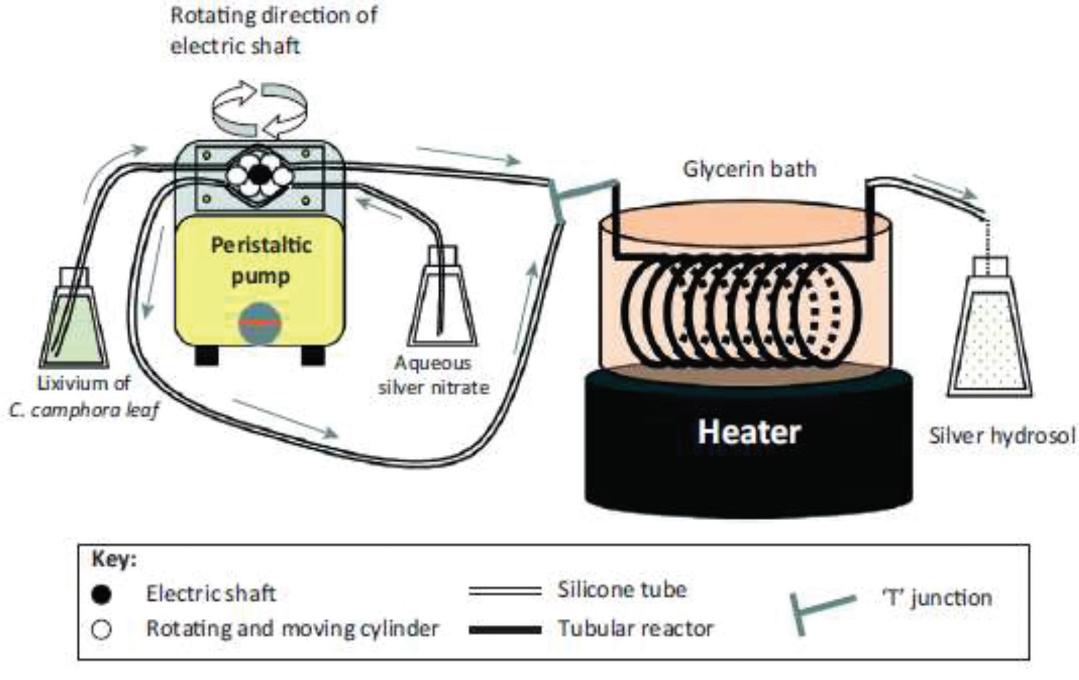

Figure 4.
Experimental diagram for the bioproduction of silver nanoparticles.

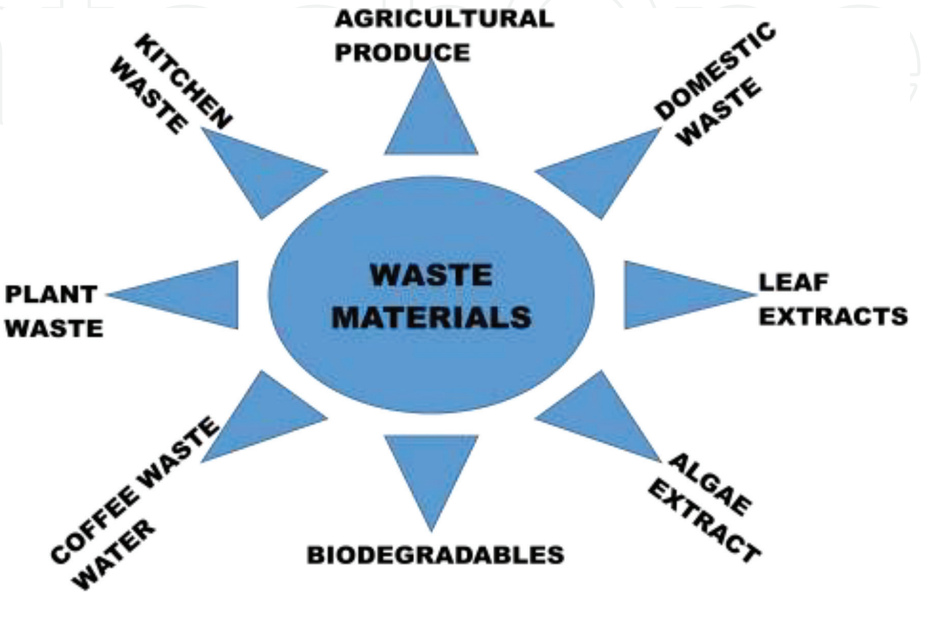

Figure 5.
Schematic showing different sources of waste material.

6. Conclusion and future perspective

The need for environmentally-friendly and stable nanomaterial that would be compatible with biological systems have prompted researchers into the production of nanoparticles. This chapter gives general knowledge on nanoparticles, their classification, merits and demerits, several synthesis and characterization techniques. Nanoparticles have economical and simple manufacturing processes that are classified into top-down method, bottom-up approach, chemical synthesis, biological method, and mechanical process. Several characterization methods of nanoparticles are geared towards understanding the morphological, structural, optical, size, mechanical, and physiochemical features. Each property is obtainable from different machines and using different techniques. The synthesis and characterization methods employed greatly influence the obtained features of the nanoparticles. Nanoparticles find useful application in medicine, drug delivery, cosmetics, optical devices, electronics, solar cell devices etc.

Author details

Agnes Chinecherem Nkele[1*] and Fabian I. Ezema[1,2,3,4]

1 Department of Physics and Astronomy, University of Nigeria, Nsukka, Enugu, Nigeria

2 Nanosciences African Network (NANOAFNET), iThemba LABS-National Research Foundation, 1 Old Faure Road, Somerset West 7129, P.O. Box 722, Somerset West, Western Cape Province, South Africa

3 UNESCO-UNISA Africa Chair in Nanosciences/Nanotechnology, College of Graduate Studies, University of South Africa (UNISA), Muckleneuk Ridge, P.O. Box 392, Pretoria, South Africa

4 Department of Physics, Faculty of Natural and Applied Sciences, Coal City University, Enugu, Nigeria

*Address all correspondence to: nechenkele@gmail.com

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