Nanoparticles: Synthesis, characteristics, and applications in analytical and other sciences

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Abstract

Nanoparticles (NPs) are widely employed in different research areas, ranging from analytical chemistry and environmental science to medicine, the agriculture and pharmaceutical industry. This is mainly due to the unique characteristics of NPs and the novelty they introduce in such applications. In analytical chemistry, the role of NPs can differ depending on the nature of the steps involved in analytical process. NPs are probably most useful for detection, but sample preparation has also profited from them. For instance, NPs can advantageously replace conventional sorbents for solid-phase extraction. Moreover, NPs are being increasingly used as stationary phases in gas and liquid chromatography or electrochromatography. In this review, a brief summary on the classification, synthesis methods, and properties of NPs is given. Moreover, the examples of applications in different research area are shortly presented. However, the merits of this work are to present the use of NPs in analytical chemistry field.

Keywords

Analytical applications; Nanoparticles; Chromatographic columns; Gas chromatography; Liquid chromatography; Capillary electrophoresis

1. Introduction

“Nanoparticle” has been defined in different ways in the literature. According to ASTM 2456-06 Standard Terminology Relating to Nanotechnology it is defined as “a particle with lengths in two or three dimensions greater than 1 nm and smaller than 100 nm and which may or may not exhibit a size-related intensive property”.

NPs are also defined as zero dimensional nanomaterials distinguishing them from one- and two-dimensional nanomaterials that have either one or two dimensions larger than nanoscale respectively. They are differentiated from their bulk counterparts in terms of size, chemical reactivity, mobility, energy absorption etc. [1].

The selection of suitable synthesis approach is very critical for synthesizing application-oriented NPs [2]. Numerous techniques relying on bottom up and top down strategies have been developed over the time with each giving a certain degree of freedom to the researchers for having NPs with the desired features. NPs can be classified in various types based on the material they are synthesized from. In broader sense, they can be listed under inorganic and organic NPs. Inorganic NPs include carbon-based, metal and metal oxide, semiconducting, and ceramics NPs while organic particles include polymeric and biomolecules derived NPs. Different types of NPs have distinct properties and target applications arising from the nature of the parent material. The general properties of the NPs such as size, shape, and surface area are dependent on the synthesis strategy as well as experimental conditions. The shape and size-controlled NPs can be obtained by manipulating the synthesis conditions [3]. It has been observed that the NPs with certain morphologies are preferable in many applications, thus the concept of shape-controlled synthesis has been extensively studied [4,5]. On the other hand, some NPs possess optical, magnetic, or antimicrobial characteristics that are specifically associated with them but not all types of NPs and such NPs have showed exceptional applications in various fields [6–8]. NPs have been widely used in many scientific areas [9]. The use of NPs in the field of analytical chemistry has exponentially increased in the past decades. The unique properties of NPs make them useful for different analytical applications. It needs to be mentioned, that these uses allow extrapolations for their application in other fields as well. In the field of analytical chemistry, NPs play two main roles. Firstly, as target analytes in the realm of the analysis of the nanoworld, and secondly tools to improve analytical processes.

In this article, we briefly review the basics of the NPs, their types, synthesis methods, general and specific properties and applications. This will help the beginners as well as researchers working in analytical research areas to understand the current state of the science of NPs in analytical science and perspective dimensions. Under no circumstances do we think that our view on this matter is flawless and the only one possible. This is an attempt to cover a broader topic in a best possible way, covering only a few publications, which is due to the limitations of our review but not the quality of other published literature.

2. Synthesis of NPs

NPs are synthesized by a variety of methods, which include physical, chemical, and biological methods, and these methods can be broadly classified into bottom up and top down approaches.

The primary characteristics of NPs are dictated by the synthesis conditions. These characteristics include but not limited to size and size distribution, crystallinity, shape, directional properties, mutual alignment.   The main category of NPs synthesis methods are bottom up approaches and top down approaches. Brief information on both modes are given in Table 1.

Table 1. Information on the bottom up and top-down approaches for NPs synthesis.

Synthesis approach	**Remarks**	**Advantages**	**Disadvantages**	**Methods used for syntheses**
Bottom up approach	The building blocks are added onto each other to form NPs. The atoms, molecules and even smaller particles can be used as the building blocks for the assembling of required nanostructures. The assembling the atoms onto each other leads to crystal planes, crystal planes further stack onto each other, resulting in the synthesis of the NPs or nanostructures. The convenient size of the building blocks relies on the properties to be engineered. This approach usually starts from the homogenous solution or gaseous phase to build-up the NPs. It usually involves a kind of chemical reaction that leads to desired product. In the everyday life it can be resembled with building a house from the bricks.	Provides a better chance of producing NPs with less defects, enhanced homogenous chemical composition, and improved short- and long-range ordering. Advantages in terms of cost, scalability, and uniformity.	The requirement of compatible surfaces and molecules. There are only fewer opportunities to manipulate the atoms and molecules.	Chemical reduction; Electrochemical reduction or oxidation; Photochemical Synthesis; Sonochemical Synthesis; Hydro/solvothermal Synthesis; Thermolysis; Biological methods (bacteria, yeast, fungi, plant extracts, etc.,); Co-precipitation; Microemulsions; Interfacial methods of Synthesis; Solvated metal atoms dispersion; Microwave-assisted Synthesis; Arrested precipitation; Atomic layer deposition; Sol gel fabrication; Vapor phase chemical deposition
Top-down approach	It is a process of miniaturizing or breaking down bulk materials while retaining the original integrity. A top-down approach usually starts from the solid material. Top-down approaches are suitable for nano-fabrication and well-developed instrumentation is available.	Simple method.	The main issue with the top-down approach is the imperfection of surface structure and substantial crystallographic impairment to the processed patterns. Though these imperfections create additional challenges in the application and fabrication of NPs, this approach is suitable for the bulk production of NPs. It requires large and expensive instruments.	Lithography (photo, electron beam, soft, nanosphere, nanoimprint, block copolymer, scanning probe, etc.,); Micromachining; Ball milling; Wire explosion; Arc discharge; Laser ablation; Ion-sputtering; Inert-gas condensation .

2.1. Bottom up approaches

The chemical reduction is the widely used method for synthesis of NPs. It is simple and gives liberty in selection of molar concentration of the reactant, dispersant, and the feed rate of the reactant to acquire NPs with desired size, shape, and size distribution. [10].

Electrochemical synthesis of NPs on a substrate is also an interesting method as it is simple and cost effective. The NPs of various shapes and sizes can be obtained by simply varying electrochemical parameters. This method is applicable for a wide variety of ions. Bottom-up electrochemical approaches involve layer by layer formation of atoms [11].

The electrochemical reduction method is used for the synthesis of hybrid NPs such as graphene–AuNPs. For example, for graphene–AuNPs, the classical version involves deposition of graphene sheets onto an electrode, then immersion of electrode in an electrolytic solution of metallic precursors, and the application of an electrochemical potential.

Another way of NPs synthesis is the photochemical method which involves the application of photochemical source of strongly reducing radicals for generation of NPs. It provides spatiotemporal control of NP generation where light intensity can be used to control particle size. These particles can exhibit excellent stability without the use of stabilizing agents [12].

High intensity ultrasound provide a unique route for synthesis of NPs without requiring bulk high temperatures, high pressures, or long reaction times [13]. The several theories deal to explain with the breaking of bonds using 20 kHz ultrasonic irradiation. Generally, it is linked with acoustic cavitation which involves formation, growth, and collapsing of bubbles. This leads to very high local temperatures, pressures, and cooling and heating rates resulting in high energy chemistry [14]. The other advantages of sonochemical synthesis include energy and time efficiency along with homogeneity in synthesis [15].

Another route of synthesis is hydrothermal approach which rely on synthesis in the solution phase. In other words, it is a method of synthesis from room temperature to high temperature solutions. It can provide a control of the morphologies of the resulting NPs by applying low or high pressures depending on the vapor pressure of the material in the solution. This method can be used to synthesize the NPs from the materials which are by themselves not stable at high temperatures [16] [17].

Co-precipitation methods are also widely employed for the synthesis of NPs [18–20]. They are based on the reactions that allow simultaneous nucleation, growth, and/or agglomeration processes to take place. The insoluble products are obtained under supersaturated conditions. Co-precipitation allows the formation of large number of small sized particles. The secondary reactions may cause changes in particle size, morphology, and aggregation. The simplicity and speedy synthesis are the major advantage of co-precipitation.

Microemulsions have shown some interesting applications due to their extremely low interfacial tensions, large interfacial areas, high thermodynamic stability, ability to solubilize otherwise immiscible liquids. They have also been used for the synthesis of various NPs [21]. Microemulsions generally consist of two immiscible liquids such as oil and water and a surfactant. The surfactant is employed to stabilize the droplets of oil in water or water in oil when employed in small quantities [22].

Solvated metal atoms dispersion method for synthesis of NPs relies on a procedure based on cryochemistry. In this method, metal elements or semiconductors are vaporized to generate free atoms (e.g. Au atoms) or high-temperature reactive molecules (e.g. CdTe molecules). Then they are co-condensed with relatively unreactive solvents (e.g. toluene, pentane, or acetone). This is followed by controlled heating for the production of NPs [23]. The advantages of this method include easy scale up, excellent reproducibility, and prevention of tedious purification procedures [24].

Microwave assisted synthesis is another popular way of NP synthesis [25–27]. Basically, microwave is a replacement of conventional heating and energy sources.

Atomic layer deposition (ALD) has also attracted a great deal of attention [28] in synthesis of NPs due to its following unique features of:

i. Achieving better material growth with atomic level precision;
ii. Freedom of nanostructures manipulation and variability of material composition;
iii. Providing desired crystallinity;
iv. Uniform film growth;
v. Applicability to thermally sensitive substrates.

ALD is a technique of generating thin films on planer substrates through self-limiting chemical reactions between the gaseous phase and solid substrate with control at the atomic scale. The resulting NPs can be employed for wide range of applications [29].

Sol-gel method is another famous procedure for the synthesis of the NPs. It relies on hydrolysis and condensation of metal alkoxide or metal oxide solution. A colloidal solution is formed in hydrolysis step which is known as sol and it is finally changed into a semi-solid phase through the condensation process known as scenariosgel. This gel is then subjected to high-temperature drying to get the desired NPs. [30–32]. The simple procedure, low-cost, homogeneity and high purity of resulting NPs are the main advantages of sol-gel method.

Chemical vapor deposition (CVD**)** is used for the synthesis of inorganic NPs on the surface of suitable substrates. It generally happens in three steps. First, a volatile precursor is introduced into a reaction chamber containing the substrate through a carrier gas. The vapors adsorb over the surface of the substrate leading to some intermediate products. In the last step, the decomposition of the intermediate products is carried out to form solid grains and NPs [33].

Several researchers emphasized that chemical methods of synthesis of NPs are not in accordance with recently emerging concepts of green chemistry. The green chemistry suggests developing the methods that are eco-friendly and do not impact the environment through hazardous effects. This has led the chemists to search for alternative methods that are sustainable and environmentally friendly. The biological organisms and plant extracts have been widely employed for synthesis of various types of NPs [34]. The toxicity of NPs is not a well-established subject; thus, the use of green materials and reagents can decrease the potential toxicity of the NPs and other byproducts. In greener procedures, non-toxic solvents, closed reaction systems, the greener energy sources like microwave and ultrasound, and gentle temperature and pressure conditions are preferably selected. The green procedures have been widely adopted to synthesize zero-valent metal, metal oxide, and salt NPs. The advantages of greener synthesis methods are listed in Figure 1.

Figure 1. Advantages of the greener methods for synthesis of NPs.

2.2. Top-down approaches

Top-down approaches are characteristically simpler and rely either on the removal or division of bulk material to form NPs. Commonly used methods for top-down syntheses are shortly described below.

Ball milling is one of the ways of preparing NPs or dispersion of metals to other materials to form alloys. The powdered material is subjected to high energy collisions with the balls. This is a popular method due to its simplicity, low-cost, and easy of applicability to almost all kinds of materials [35], [36].

The electrical wire explosion is also used as a destructive technique for synthesis of nanopowders and NPs. When a high-density current pulse, passes through a wire, it exceeds the density of the energy in the wire than the binding energy due to excessive energy and expansion lag of the heated material. Consequently, the wire boils up in a burst with a flash of intense light, leading to the production of a superheated vapor and boiling droplets of the exploding wire material and scattering to the ambient atmosphere [37]. This method is highly productive but consumes excessive energy.

Merits and demerits of some bottom-up and top-down techniques are summarized in a comprehensive review on this topic that can be consulted for further reading [38].

3. Types of nanoparticles

NPs can be classified based on their properties such as shape, size, activity, and type of the materials they are made of. The role of size, shape, and other properties will be discussed in other sections. Here, the classification of NPs will be shortly described based on the materials they are made of. NPs can be broadly classified into inorganic and organic particles. The inorganic NPs further include: carbon-based NPs, metal and metal oxide NPs, semiconducting NPs, ceramic NPs; while the organic NPs can be classified into two categories: polymeric NPs and biomolecules derived NPs.

3.1. Carbon-based NPs

Carbon NPs (CNPs) cannot be strictly confined to spherical particles having a diameter less than 100 nm. Indeed, all types of carbon nanomaterials are generally classified under carbon NPs. Based on extensive literature review, it has been noted that there is no consensus on this issue. Thus, single-walled and multiwalled carbon nanotubes (CNTs), graphene, fluorescent carbon quantum dots (CQDs), carbon dots, and others are considered as carbon NPs. They have been widely employed in many fields due to exceptional physical, chemical, mechanical, and thermal properties [39][40].

CNPs have been widely adopted in many scientific and technological applications due to the high surface area, good biocompatibility, low-toxicity, and low cost as well as greener synthesis routes. These properties along with excellent optical features enabled their applications in biological imaging, biomedical, photocatalysis, optical and chemical sensing [39]. Different forms of carbon nanostructures are shown in **Figure 2**.

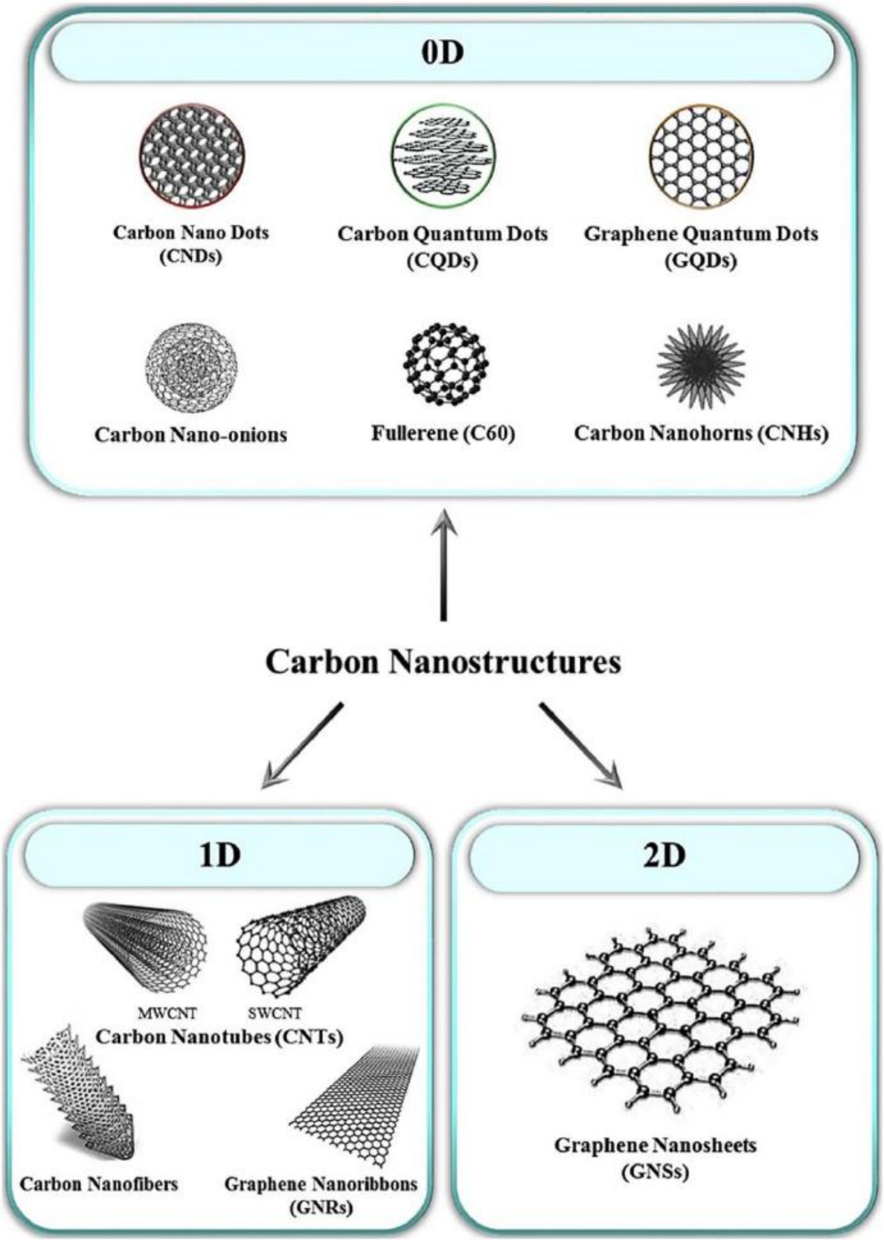

Figure 2. Different types of carbon nanostructures in zero, one and two dimensions. Reprinted with permission from [39]. Copyright 2019 Elsevier B.V.

3.2. Metal and metal oxide NPs

Metal and metal oxide NPs possess a unique and broad range of physicochemical properties. They possess enhanced chemical, electrical, optical, thermal, mechanical, electromagnetic and surface properties compared to their bulk materials. Moreover, they offer large surface areas, controllable size and morphology, and simple surface modification. Introduction and applications of metallic NPs is provided in a review [41].

This is the reason they have been employed in a variety of applications [42] such as biomedical, catalysis, environmental remediation, energy harvesting, molecular sensing, etc. They have been synthesized both by top-down and bottom-up approaches. Au, Ag, Pt, Pd, Cu, CuO, Ni, NiO, Zn, ZnO, Iron oxide, titanium dioxide, cerium oxide is among commonly used metal and metal oxide NPs.

3.3. Semiconducting NPs and QDs

Semiconductor nanoparticles or quantum dots (QDs) have shown excellent applications in labeling of DNA, cells, and proteins. They offer tunable emission spectra that can be tuned throughout the ultraviolet, visible, near-infrared, and mid-infrared spectral ranges [43]. Moreover, they have high photo stability as well as resistance against photo bleaching, and manipulatable surface features. Another advantage of semiconductor QDs is the “quantum confinement effect”. The emission spectra depend on the size of QDs. They are better alternative to natural fluorophores and their optical properties are controlled by many factors such as shape, size, doping, and the surrounding environment [44]. QDs have many advantages over the dyes. The issues of photo degradation under laser excitation, hydrophobicity of some dyes, and solvent dependent quantum yields can be addressed by their replacement with QDs in bioassays.

Some well-known semiconductor QDs are cadmium selenide (CdSe), zinc sulfide (ZnS), cadmium telluride (CdTe), zinc oxide (ZnO), and mercuric selenide (HgSe), among others. The simplest explanation is the exceptional blue-shift of absorption, as well as the PL spectra of semiconductor NPs with reduction particle size, especially when the size is adequately small. Semiconducting QDs have been used in wide range of applications such as cellular imaging, trace level detection of analytes, solar energy conversion, photocatalysis, optical devices [43].

3.4. Ceramics NPs

Ceramics can be pronounced as having a definite solid core, built or constructed by the provision of heat or both heat and pressure, composed of one of the following:

i. Metal and nonmetal
ii. At least one metal and a non-metallic elemental solid or a non-metal,
iii. A combination of at least two non-metallic elemental solids, and
iv. A combination of at least two non-metallic elemental solids and a non-metal.

Ceramic NPs are predominantly composed of oxides, carbides, phosphates, and carbonates of metals and metalloids such as calcium, titanium, silicon, etc. Most of ceramic NPs consist of silica or alumina. The porous nature of NPs contributes to them physical shield from degradation and degranulation. Nanophase ceramics can be divided into NPs, nanoscaffolds, and nanoclays [45].

Their special properties such as high heat resistance and chemical inertness enable their applications in diverse areas. They are widely explored in biomedical applications particularly in drug delivery due to controllable size, surface functionalization, porosity, and surface area to volume ratio. The critical factor that controls the properties of ceramic NPs is method of preparation as well as control of the affecting variables [46]. Ceramic NPs possess extraordinary mechanical strength, reasonable body response, exceptional pH resistance, high stability, high load capacity, simplicity of incorporation into hydrophobic and hydrophilic systems, and different routes of administration (oral, inhalation, etc.). However, some disadvantages in biomedicine may include low biodegradability, high density, and potential toxicity.

3.5. Polymeric NPs

Polymeric NPs are solid colloidal particles of size range 10 nm–1 μm. They are generally made of biodegradable and biocompatible polymers. They are used as drug carriers by encapsulating or entrapping the drugs. The drugs can adsorb over the surface physically or chemically. They are excellent carriers because of small size, water-solubility, non-toxicity, high shelf life, and excellent stability [47].

Based on the synthesis method, they can be classified into two types of architectures [48]:

i. Nanospheres: they represent a matrix system with a drug uniformly dispersed in it.
ii. Nanocapsules: they are the NPs where drug is surrounded by the polymeric membrane or in other words drug is embedded with a cavity surrounded by a polymeric membrane.

Natural hydrophilic polymers such as proteins and polysaccharides are used in the synthesis of polymeric NPs. Synthetic hydrophobic polymers are also employed either in prepolymerized form or polymerize during the synthesis process. In general, three kind of methods can be found concerning synthesis of polymeric NPs [49].

i. Synthesis from dispersion of performed polymers;
ii. Polymerization from monomers;
iii. Ionic gelatin or coacervation of hydrophilic polymers.

Polymeric NPs demonstrate excellent feature of surface modification through chemical processes, superb pharmacokinetic control, and can entrap and deliver a wide range of drugs. The most prominent NPs in this regards include the one made of polylactic acid, gelatin, poly(lactic-co-glycolic acid) copolymer, chitosan, etc. Moreover, such polymers can also be coated on the surface of other types of NPs [50].

3.6. Biomolecules derived NPs

Biomolecules such as proteins, nucleic acids, lipids, and polysaccharides have unique characteristics and can be utilized to prepare NPs. Such NPs are comparatively received less attention than inorganic NPs in the past but now they are also at the forefront of many research and development applications. Biomolecules derived NPs are getting famous because of the growing demand of biocompatible and biodegradable NPs. Moreover, biological NPs are easily available and non-immunogenic. Apart from their own unique functions, biomolecules can conjugate with other inorganic NPs to generate special biomolecule-NPs hybrids. Biomolecules such as proteins, nucleic acids, lipids and polysaccharides based NPs have been used in various applications [51–54].

4. Properties of the NPs

New particles possess some unique characteristics compared to the bulk counterparts they are synthesized from. These properties include but not limited to extremely high surface areas, excellent reactivities, exceptional mobilities, superb mechanical, chemical, and electrical properties. On the other hand, it is true that the properties of some nanoparticles are highly dependent on the particle size and the material they are derived from. That is the reason we see some nanoparticles with the exceptional optical, electrical, or magnetic properties while others do not possess these properties. In this section, we are going to discuss the general properties of the NPs as well as the specific properties that are related to particular NPs with the emphasis on tracing the origin of unique characteristics.

4.1. General Properties

These are the properties, which are related to almost all kind of nanomaterials, so do the nanoparticles. Indeed, these properties form the foundation of many applications of NPs.

4.1.1. Size of the NPs

The size of the NPs is very critical in determining their properties as well as target applications. Size of the NPs is greatly affected by the synthesis method and reaction parameters and in turn this feature affects their role in different applications. The iron oxide NPs synthesized by hydrothermal showed high crystalline iron oxides with a mixture of magnetite and maghemite crystalline phases. However, with the increase in NP size, the ratio of magnetite to maghemite phase increased and reached to a pure magnetite phase for the 123±44 nm sized particles. Moreover, with increase in reaction temperature from 100 to 180 ℃ for 12 h, the size of the NPs increased from 14.5±4 to 29.9±9 nm according to transmission electron microscopy analysis. Similarly, at 180 ℃, as the reaction time increased from 1 to 48 h, the size of NPs increased from 20.6±6 to 123±44 nm [55]. Au NPs with size less than 10 nm were prepared and evaluated for their optical properties [56]. The optical properties of the Au NPs are highly dependent particle size and so their applications are. The antibacterial properties of Ag NPs showed dependence of their size and shape against different bacterial strains [57].

The size of the NPs greatly impacts the targeted applications. For instance, an electrode was prepared by nucleating and growing a single Pt NP on a tunneling ultramicroelectrode (TUME) with 1–40 nm or greater dimensions. It increased the mass transfer rate and was useful for measuring electron transfer parameters for fast ET reactions [58]. The variations in the size of iron oxide NPs also affect bandgap and strain [59].

4.1.2. Shape of the NPs

Apart from the size of the NPs, shape, and structure are also very important in many technological applications. The challenge of controlling the shape of the NPs by controlling the synthesis parameters has been somehow and to large extent, addressed. Thus, the NP of the same material with different shapes can be synthesized to deal with specific applications. Indeed, shape-controlled synthesis of NPs is now a well-established research dimension in the nanosciences. The shape-controlled inorganic NPs from solution via nucleation and growth theory and the control of synthesis conditions have been discussed in a review [60]. The typical morphologies of NPs in different dimensional set up are indicated in **Figure 3**.

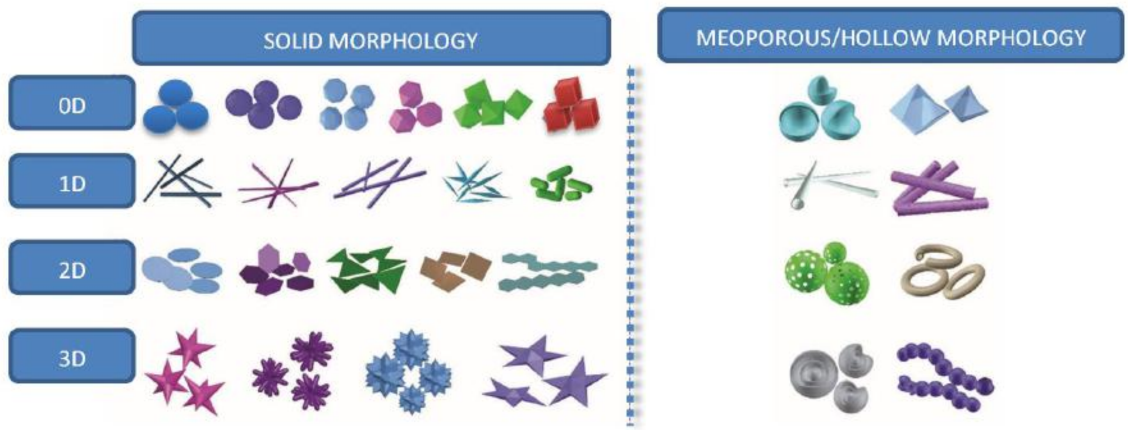

Figure 3. Typical morphologies of solid and mesoporous/hollow inorganic nanoparticles with 0D, 1D, and 2D shapes and other 3D complex structures. Reused with permission from [60]. Copyright 2016 Royal Society of Chemistry.

This is well known that electrocatalytic activity of many electrochemical reactions is governed by the surface area as well as the structure of the catalyst, NPs based catalyst are no an exception to it. Shape-controlled octahedral cobalt disulfide NPs supported on nitrogen and sulfur-doped graphene/carbon nanotube composites were employed for oxygen reduction in acidic electrolyte [61].

The spherical, cubic, ellipse TiO_2 NPs and nanorods were synthesized by manipulating the use of different surfactants during the preparation. The effect of the shape was also reflected in their photocatalytic activity [62]. Se NPs have been synthesized in a variety of shapes such as nanowires, nanoribbons, nanoplates, nanotubes, and nanospheres using different approaches. Spherical Se NPs are mainly studied for biological activity. Recently, cubic-like SeNPs were synthesized via self-assembly process and the effect of the shape on their antitumor activity was investigated [63].

The size and morphology are related with fabrication method. A recent review on ZnO NPs based solar photocatalysts has tabulated the fabrication method and its effect on the resulting particle size and morphology [64].

4.1.3. Surface area of the NPs

Many applications of NPs are due to their high surface areas as it plays significant role catalysis, adsorption, electrochemical reactions, reactivity etc. Super paramagnetic ascorbic acid coated Fe_3O_4 NPs with high specific surface area (179 m^2/g) and diameter less than 10 nm were prepared using a hydrothermal approach and used for adsorption of heavy metals and exhibited reasonably good adsorption capacities [65]. The method of synthesis and experimental conditions have direct effect on the surface area of the resulting NPs and their activity in specific applications. For example, $LaFeO_3$ NPs synthesized by SBA-16 template method showed much high surface area compared to the conventional citric acid method. The high surface area NPs exhibited excellent activity toward photocatalytic degradation of Rhodamine B [66]. Similarly, SnO_2 NPs synthesized by a homogeneous precipitation ethanol-thermal method with $CO(NH_2)_2$ and $SnCl_4 \cdot 5H_2O$ as starting materials showed very high specific surface area (200 m^2/g, size 8–9 nm) compared to single homogeneous precipitation method (55 m^2/g). Moreover, the former showed excellent gas sensing properties [67]. Ecotoxicity of SiO_2 NPs to the green alga *pseudokirchneriella subcapitata* was related to the surface area of the material [68].

4.2. Properties specific to certain types of NPs

Many properties are associated with specific NPs such as optical, magnetic, antimicrobial, so all types of NPs may not exhibit these properties. Here, this section will briefly discuss these specific characteristics of NPs.

4.2.1. Optical properties

The optical properties of the metal NPs are of great interest to the scientific community and history of the use of such NPs dates back to mid-1800s [69]. Au NPs have been widely discussed in the literature with regards to optical properties, it is because they demonstrate unique and tunable optical properties mainly due to surface plasmon resonance phenomenon. This phenomenon enhances the properties like Mie scattering, surface plasmon absorption, surface-enhanced luminescence and surface-enhanced Raman scattering (SERS) from adsorbed molecules. They can be easily synthesized in good quality and yield in different shapes and configurations. In addition, they are biocompatible and thus suitable for clinical applications [8]. Moreover, colloidal Au NPs surface can be easily functionalized with a variety of biomolecules, antibodies, and ligands and thus they can be employed for targeting the cancer-related biomarkers on the cancer cells. This aspect provides molecular-level specificity. Apart from the diagnostics, they are useful in the treatment options. The strong surface plasmon absorption of the Au NPs, followed by rapid photothermal conversion, has also been used for the selective photothermal therapy of cancer, by using a suitable immune targeting strategy. The optical tuning of SPR of Au NPs into near IR region is possible by changing their shape from spherical to nanorods. This aspect is of great value in *in vivo* imaging and therapy [70]. Au NPs have also been used as labels in lateral flow assay technology [71], as well as in biological recognition in biosensors [72]. Since the color and maximum absorption wavelength of AuNPs vary depending on particle size and inter-particle spacing. Such properties have been widely utilized in developing colorimetric sensors for the detection of wide variety of analytes in environmental, food, and biological samples. Such techniques are simple, fast, sensitive, and applicable for on-site monitoring and rapid analytical scenarios [73]. Apart from the AuNPs, other noble metal, some metal oxide, and metal sulfide NPs have also shown optical properties and thus related applications [74–77]

Upconverting NPs absorb two or more photons of low energy and convert them into a single photon of high energy. Typically, absorption takes place in IR region while emission in visible or UV region. Such NPs also have wide applications in bioimaging, biosensing, drug delivery and theruptics mainly due to their excellent biocompatible and low cytotoxicity. A detailed review on the design, nanochemistry, and applications in theranostics can be consulted [78]. Optical properties of NPs are controlled by their size and aggregation.

4.2.2. Magnetic properties

Magnetic NPs are a special group of NPs that can be controlled using the magnetic fields. They are generally composed of a magnetic material and a chemically functional material. The magnetic material may include iron, nickel and cobalt and chemically functional material is selected or synthesized as per the nature of the application. Magnetic NPs display extraordinary new singularities such as high field irreversibility as well as saturation field, superparamagnetism, additional anisotropy contributions, or shifted loops after field cooling. These phenomena are coming from narrow and finite-size effects and surface effects that dominate the magnetic behavior of individual NPs [79].

Magnetic NPs are used for a broad spectrum of industrial applications such as contrast agent in nuclear magnetic resonance, therapeutic agents in cancer treatment, materials for data storage, separation of pollutants from water and other media, etc. However, for every application, they need to exhibit specific properties. For example, in data storage, they should be stable as well as demonstrate switchable magnetic state to signify bits of information that are not affected by temperature variations. Similarly, for biomedical application, magnetic NPs must possess super paramagnetic behavior at room temperature. The stability of the NPs under physiological conditions is important factor for biology and diagnostics [79]. Another important factor for biomedical applications of the magnetic NPs is their biocompatibility and toxicity in the system which indeed depends of the nature of the magnetic component, size of the particle, core and outer functional groups. The iron based magnetic NPs such as magnetite are widely used in biological applications. The other materials based on cobalt and nickel can oxidize and may induce toxicity and thus of little importance for *in vivo* applications. Hollow MNPs have been used in drug delivery because of their safety as pharmaceutical excipient [6]. Moreover, they have also been employed in bacterial detection and infection treatment [80].

Magnetic Fe_3O_4 NPs have been widely employed as a sorbent for the removal of pollutants from water and wastewater due to ease of their separation with the aid of external magnetic field. However, their activity may reduce due to agglomeration. Thus, their surface coating or functionalization with special groups has emerged as a solution for their potential applications in environmental remediation. Thus, they have been modified with inorganic materials, organic small molecules, natural biopolymers, synthetic polymers, and others [81]. Magnetic NPs have been integrated with surface enhanced Raman spectroscopy (SERS) for the analysis of the environmental pollutants. Magnetic NPs used for SERS can be classified into four types (i) core-shell (ii) special shaped (iii) core-settalite (iv) multifunctional (**Figure 4**). The reason of using magnetic NPs for SERS is their ability to provide high enrichment factor, sensitivity enhancement due to capturing of analytes through functionalized surfaces, well-ordered nanostructure, SERS substrates suitable for field applications [82] .

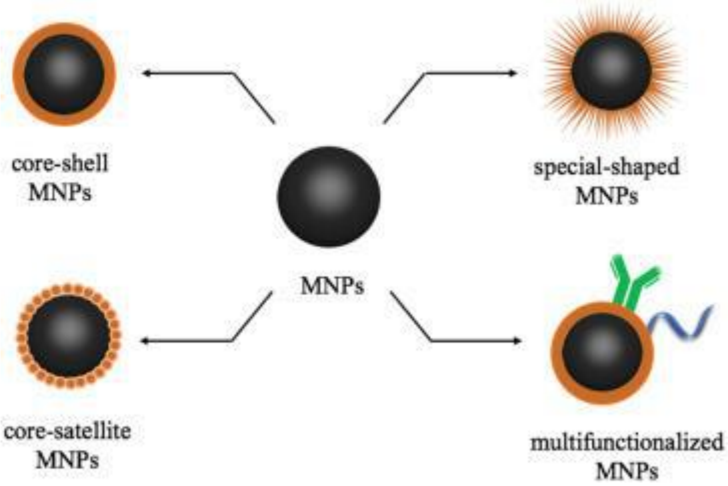

Figure 4. Magnetic nanoparticles as SERS substrates with different shapes. SERS: surface-enhanced Raman scattering. Reused with permission from [82]. Copyright 2018 The Research Center for Eco-Environmental Sciences, Chinese Academy of Sciences. Published by Elsevier B.V.

Magnetic NPs have been used for electrochemical immunoassays due to the features of separating and enriching the analytes from the sample and then bringing them to the electrode either magnetically or immunospecifically [83].

4.2.3. Antimicrobial properties

Several NPs have been employed as antimicrobial agents due to their excellent antimicrobial properties. Ag NPs are more often used compared to other NPs. Metallic silver, other silver compounds are well known for their antimicrobial properties since centuries, and they had been used in burn, wound, and antibacterial infections. Though their use was declined in recent times due to development of advanced antimicrobial drugs, they are again emerging as alternatives to counter the antibiotic resistance developed by the bacteria. Since it attacks a broad range of targets, it is unlikely that microorganisms develop resistance against Ag NPs compared to conventional antibiotics.

Though metallic silver and its other compounds are effective against the microbes, the emergence of nanotechnology has created new opportunities such as high surface area NPs can be produced which are in turn more effective than their bulk counterparts. They have been proven very effective against a number of microbial strains. Thus, they have been used in wound dressings, medicals devices, also impregnated in cloth fabrics. There are some concerns about the silver toxicity but it has also been noted that they are non-toxic at minute concentrations [7]. The nanocomposites of silver with other materials such as silica has also shown excellent antimicrobial properties [84]. High-purity metallic chitosan-copper NPs showed excellent antimicrobial potential against *Staphylococcus aureus, Bacillus subtilis, Pseudomonas aeruginosa, Salmonella choleraesuis, and Candida albicans* strains [85].

ZnO NPs and nanomaterials have good biocompatibility, and this is the reason they have been studied for their cytotoxicity and interaction with cells, tissues, biomolecules. They demonstrate good antimicrobial potential and interact with the cells of bacteria through chemical and physical mechanism. In chemical mode, they produce photo induced reactive oxygen species, H_2O_2 and release Zn^{2+} ions while in physical interaction they induce antimicrobial effects via cell envelope rupturing, cellular internalization or mechanical damage. The effect of different morphologies of ZnO nanomaterials is also reviewed in a review [86].

Apart from the metal and metal oxide NPs, many polymeric materials are used in combination with them for the proper activation and delivery of NPs [87]. It has been reported that a large percentage of hospital acquired infections spreads through contaminated surfaces or catheters mostly made of plastics. A variety of polymers is used in biomedical and health care applications. Incorporation of antimicrobial NPs to such polymer matrices can reduce the ratio of infectious diseases spreading through the plastics [88].

Curcumin is a natural antimicrobial compound found in turmeric and being employed as a home remedy for many diseases. However, it has low solubility in water and poor bioavailability. To cope with these issues, curcumin NPs were synthesized and evaluated for antimicrobial activity which was significantly improved by decrease of particle size [89].

The readers interested in characterization tools, toxicity factors, exposures and control strategies of NPs might benefit from previously published reviews [9][90].

5. Applications of NPs

As was mentioned previously, NPs are applied in many scientific and industry fields. The specific properties of NPs as well as their high surface-to-volume ratio has led to their use in several analytical applications. NPs have been applied for different applications in the time-honored disciplines of analytical chemistry such as spectroscopy, electronic detection, and separations, as well as in various sensor technologies. In this Section, the use of NPs in different areas is reviewed, with special emphasis on analytical chemistry science.

5.1. Separations

NPs are very useful in the separation science as they can facilitate mass transfer and increase separation Efficiency what is due to their high surface-to-volume ratio. NPs are chemical stable over a wide range of pH. As was previously mentioned, there is a large selection of NPs, including , polymers, metal oxides, fullerene nanomaterials and carbon nanotubes. These nanoparticles can be applied Directly as the packing material, but also to modify the packing material in columns in different type of chromatography, electrochromatography and capillary electrophoresis (CE). NPs can also be useful as additives of running buffer solution in microchip electrophoresis and CE. Nowadays, the most popular in separation area are monilith columns in combination with NPs. However, nanosized metal organic frameworks (MOFs) as well as various magnetic NPs play also an important role for separat in different analytes. The lates have been applied for concentration of analyte and separations, where the analyte detection usually occurs by some type of spectroscopic or electrochemical techniques [91]. Information on the application of different kind of NPs in analytical separation and electroanalytical sensing are given in Table 2.

5.1.1. Analytical sample preparation

Despite the tremendous advancement in analytical instrumentation sample preparation is a critical step in chemical analysis. The requirement of sample preparation arises from one or more of the following:

(i) Complex nature of the matrix that is not compatible with analytical instrumentation;
(ii) Low concentrations in the real samples which can be detected by the instrument only after enrichment;
(iii) Analytes cannot be measured directly with available instrumentation and need chemical derivatization before analysis.

The sample preparation is performed either by conventional approaches such as solid phase extraction and liquid phase extraction or by miniaturized techniques such as solid phase microextraction, liquid-phase microextraction, dispersive solid phase microextraction, etc. NPs have been used as extraction medium in both conventional and miniaturized approaches. Below are the few examples of using NPs in analytical sample preparation.

Au NPs were used as a coating in SPME for extraction of organochlorine pesticides in environmental samples before their GC-ECD analysis [92]. Silica fiber modified with self-assembled Au NPs based HS-SPME was used for extraction of PAHs in water samples prior to HPLC-UV analysis. The LODs were in the range of 0.10 – 0.89 ng/mL. ZnO NPs have shown excellent gas adsorption properties and now been explored as adsorbents in sample preparation. ZnO NPs affixed to a composite made from polythiophene and hexagonally ordered silica was used as SPME coating for the determination of volatile organic compounds of *Matricaria chamomilla* [93]. NP-incorporated PDMS fibers were also used for extraction of VOCs of *Eucalyptus Leaf* [94].

Magnetic and functionalized magnetic NPs are widely used as extraction medium in solid phase extraction of different classes of analytes in varying complexity samples. The magnetic extraction phase is easier to separate after the extraction through the aid of an external magnet. Cetyltrimethyl ammonium bromide-coated Fe_3O_4@decanoic acid was used for the dispersive micro solid-phase extraction of acidic and basic drugs from biological fluids and wastewater samples. Reasonably low LODs, wider linear dynamic ranges, and good reproducibility were observed [95]. Polypyrrole/magnetic NPs composite was used for the extraction of antidepressant drugs from biological samples [96].

The magnetic NPs based extraction of DNA from different samples is highly advantageous compared to conventional organic solvent-based methods due to fast extraction process, low-cost, elimination of tedious centrifugation or precipitation, and potential for automation and scaling up. A high yield of genomic nucleic acid was observed from different sample sources using magnetic NPs [97].

5.1.2. Columns containing NPs

As mentioned, monolithic capillary columns embedded with NPs are one of the most popular nowadays. Such solution has been used by Xu et al. [98] who modified porous polymer monolithic capillary column with gold NPs that enables the selective capture of cysteine-containing peptides. This solution was proposed to reduce the complexity of peptide mixtures generated in bottom-up proteomic analysis. The column was manufactured from a poly(glycidyl methacrylate-*co*-ethylene dimethacrylate) monolith through reaction of some of its epoxide moieties with cysteamine to affording a monolith rich in surface thiol groups. *In situ* chloroauric acid reduction within the column was applied to form gold NPs attached to the surface of the pores of the monolith. This solution retains the excellent hydrodynamic properties of the monolithic column while providing a means to selectively retain cysteine-containing peptides from an analyte due to their high affinity for gold. The created column could selectively capture cysteine-containing peptides from their mixtures with other peptides and subsequently release them for micro-HPLC-MS analysis when all other peptides have been analyzed or eluted.

In another work, porous polymer monolithic columns with gold nanoparticles as an intermediate ligand for the separation of proteins in reverse phase-ion exchange mixed mode was used [99]. Here, the pore surface of monolithic poly(glycidyl methacrylate-*co*-ethylene dimethacrylate) capillary columns was functionalized with thiols and coated with gold nanoparticles. The final mixed mode surface chemistry was formed by attaching, in a single step, alkanethiols, mercaptoalkanoic acids, and their mixtures on the free surface of attached gold nanoparticles. Use of these mixtures provided fine tuning of the hydrophobic/hydrophilic balance.

Metal organic frameworks are another popular NPs used to modify capillary columns applied for separation. It is well known that the diffusion resistance related with metal organic framework packed columns direct to poor resolution when separating large molecules. Thus, coating capillary columns with MOFs can potentially resolve this problem. However, one problem exist here. As the traditionally synthesized MOFs are in the micrometer size range, they are difficult to coat capillary columns with them. This has led to capillary columns coated with nanosized MOFs [91]. Such columns were used by Chang et al. [100], who used MOFs to design tandem molecular sieves as a dual platform for selective SPME and high-resolution GC separation of target analytes in complex matrixes. An elegant combination of a ZIF-8-coated fiber for SPME with a ZIF-8-coated capillary for GC allows selective extraction and separation of n-alkanes from such samples as petroleum-based fuel and biological fluids. The proposed tandem ZIF-8 molecular sieves offered many advantages such as good enhancement factors and wide linearity with 3 orders of magnitude for the tested analytes. The large diversity in structure and pore size allows various combinations of MOFs for designing an MOF-based tandem molecular sieve platform to achieve different selectivities in extraction and chromatographic separation and to solve headache problems in complex real sample analysis.

In another work, the slurry-packed MIL-101(Cr) column for HPLC separation of substituted aromatics was reported [101].The MIL-101(Cr) offered high affinity for the ortho-isomer, allowing fast and selective separation of the ortho-isomer from the other isomers within 3 min using dichloromethane as the mobile phase.

Nanoparticles are many offen used as pseudostationary in electrokinetic chromatography. The utility of novel latex NPs as pseudostationary phases for electrokinetic chromatography with UV and mass spectrometric detection was demonstrated by Palmer et al. [102]. The NPs were synthesized using ab initio reversible addition-fragmentation chain transfer (RAFT) in emulsion polymerization, which yields small (63 nm) particles with a narrow size distribution, a hydrophobic core, and an ionic shell. The synthetized NPs provided efficient and selective separations, with retention and separation selectivity dominated by hydrophobic interactions. The NPs were highly retentive, such that they were effective at relatively low concentrations. Addition of the NPs to the background electrolyte at these concentrations had a minor effect on the noise with UV detection, no measurable effect on the separation current, and minor effects on analyte ionization efficiency during electrospray ionization. The NPs did not cause fouling or degradation of the electrospray-mass spectrometer interface even after several weeks of use.

5.2. Detection

5.2.1. Electrochemical sensing

NPs modified electrodes have been employed for electrochemical sensing of a wide variety of analytes in different matrices. These NPs work as electrocatalysts and enhance the sensitivity of detection due to their high surface area and other features that lead to enhanced electron kinetics. Apart from this, they are also used to immobilize and label biomolecules as well as reactants in many cases [103]. Metal, metal oxide, and carbon NPs are widely reported in this regard.

Au NPs and their composites modified sensors have been used for the detection of dopamine (DA). Au NPs are selected for this kind of sensing because of their high surface, low toxicity, excellent biocompatibility, high dispersity and most importantly high electrical conductivity [104]. Au NPs are also used in biosensors as they can impart stable immobilization of biomolecules while retaining their activity. They can provide direct electron transfer between redox proteins and electrode surface eliminating the need of mediators [105].

Pt NPs are also used but sometimes their selectivity and sensitivity are not suitable for real-life applications. In such cases, they are combined with other metals such as iron, cobalt, and nickel. Fe@Pt core-shell NPs based electrochemical sensor was used for hydrogen peroxide, glucose and formaldehyde [106]. WO_3 NPs modified glassy carbon electrode (GCE) showed excellent electrocatalytic activity toward the detection of DA [107].

Carbon NPs showed high porosity, adsorption capability, effective surface area, electrical conductivity as well as catalytic activity due to which they are excellent modifier materials for electrochemical sensing. They have been used in electrochemical sensing of pharmaceutical drugs, heavy metals, neurotransmitters, biological species, as well as non-enzymatic, enzymatic, and immunosensing. A recent review gives a comprehensive overview of this topic [39].

5.2.2. Optical Sensing

Various kinds of NPs have been used for optical sensing which include but not limited to metal and metal oxide, polymeric, carbon nanodots, quantum dots, and other fluorescent and luminescent NPs arising from different materials. In some cases, NPs are used as labels while in other cases NPs are labeled with other optical signal producing materials to recognize the sensing events. Optical sensing is generally based on fluorometric or colorimetric sensing.

Fluorescence is one of the techniques used for optical chemical and biochemical sensing. Fluorescence-based optical sensors require sensing receptors that will produce a measurable change after interacting with the analyte. For this reason, various fluorophores are used such as organic dyes. Recently, inorganic NPs based fluorophores have shown excellent potential for optical sensing due to their unique features and alleviating the drawbacks of conventional dyes. Fluorescent inorganic NPs (FINPs) include but not limited to conventional quantum dots, silicon-based quantum dots, upconverting NPs, metal NPs, carbon nanodots. Some other materials are also used but in combination with fluorescence promoting materials [108]. The mechanism of FINPs-based optical sensing is depicted in **Figure 5**. Details can be read in a review article.

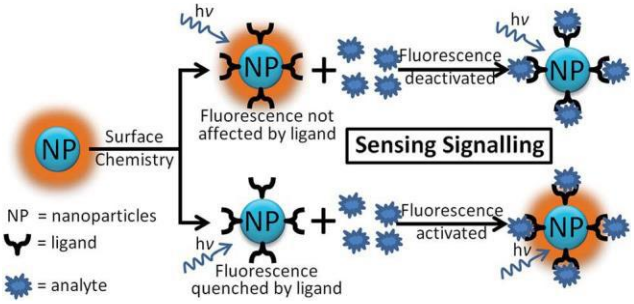

Figure 5. Schematic depiction of possible signalling mechanisms of FINPs that can be adopted for sensing purpose. Reused with permission from [108]. Copyright RSC.

Commonly, the optical properties of small metal NPs are dominated by collective oscillation of electrons at surfaces (known as "surface plasmon resonance", SPR or "localized surface plasmon resonance", LSPR) that are in resonance with the incident electromagnetic radiation. Aggregations of AuNPs lead to distinct color changes. Au NPs have been used in colorimetric sensing of heavy metal ions and other pollutants. The light scattered from Au NPs also lies in visible region, they have been used in imaging applications. Their tailorable physical properties and diverse surface chemistry that can be modified by several biomolecules further extends their application in biodetection and many other areas [109]. Apart from gold, silver, copper, platinum NPs based methods have also been used in colorimetric sensing of different analytes [110,111].

Fluorescent carbon dots (CDs), with sizes of less than 10 nm, have attracted marvelous consideration in miscellaneous research fields. They have low cytotoxicity, outstanding aqueous solubility, good biocompatibility, wider photoluminescence outlines, and high photostability. They have been used in optical detection of different kind of analytes either based on fluorescence enhancement or fluorescence quenching [112]. In some cases, CDs have been used both for fluorometric as well as colorimetric sensing of analytes [113].

Polymeric NPs are widely employed in optical diagnostics for the detection of biomarkers, cancer diagnosis, imaging, and immunoassays. Such sensing relies on NPs and targets analyte binding. The binding event then must be converted into measurable optical sensing. In such cases, enzymes are used as labels because they can catalyze the formation of colored products. In label-free optical sensing, NPs or target molecules should itself be able to emit optically measurable signals [114].

Table 2. Application of NPs in sample preparation, chromatographic columns and electroanalytical sensing.

Application of NPs as sorbent in microextraction processes									
Metal Organic Frameworks									
Matrices	Analytes	MOF (metallic ion and ligand); synthetic Solvent	Microextraction Format	Extraction Time (min)	MOF amount [mg]	LOD	RSD [%]	Methodology	Ref.
Water	Hormones	UiO-66 (Zr^{4+} and H_2bdc); DMF	MOF packed inside porous PP bag	40	10	2.0–10 ng/L	<6.5	µ-SPE-LC-MS-MS	[115]
		MIL-53(Al) (Al^{3+} and H_2bdc); water	MOF powder	30	8	1.5–1000 ng/L	<7.8	µ-dSPE-LC-MS-MS	[116]
	Endocrine disrupting chemicals	UiO-66-NO_2 (Zr^{4+} and O_2N-H_2bdc); DMF	MOF powder	3	20	1.5–90 ng/L	<14	µ-dSPE-LC-DAD	[117]
	PCPs	CIM-81 (Zn^{2+} and Htz + H_2bdc); DMA	MOF powder	1	20	0.5–1.5 µg/L	<13	µ-dSPE-LC-UV	[118]
	Fungicides	Fe_3O_4-CO_2H@MIL-101-NH_2 (Fe^{3+} and NH_2-H_2bdc); DMF	Heterogeneous composite powder	20	20	0.04–0.4 µg/L	<10.2	m-µ-dSPE-LC-UV	[119]
	Phenols	H_2N-MIL-53(Al) (Al^{3+} and H_2N-H_2bdc); DMF	MOF powder	0.17	30	0.4–13.3 µg/L	<6.30	µ-dSPE-LC-PDA	[120]
Vegatabels	Fungicides	Fe_3O_4/ GO-IRMOF-3 (Zn^{2+} and NH_2-H_2bdc); DMF	Heterogeneous composite powder	30	10	0.25–1.0 µg/L	<7.3	m-µ-dSPE-MS-MS	[121]
	SUHs	Fe_3O_4@PDA @ MIL-101(Fe) (Fe^{3+} and	Heterogeneous composite powder	3	60	0.12–0.34	<4.8	m-µ-dSPE-LC-PDA	[122]

		H_2bdc); DMF				µg/L			
Urine	Caffeine	ZIF-67 (Co^{2+} and 2-MIm)l; methanol	Anodized aluminum bar	20	1 cm × 500 µm thickness	50–100	<6.1	SBSME-LC-UV	[123]
	Aromatic amines	JUC-Z2 (Ni^{2+} and 2,2'-bipyridyl); DMF	Functionalized fused silica fiber	40	80 µm thickness	0.010–0.012 ng/L	<7.7	HS-SPME-GC-MS-MS	[124]
	Hormones	MIL-53(Al) (Al^{3+} and H_2bdc); water	MOF powder	30	8	1.5–1000 ng/L	<7.8	µ-dSPE-LC-MS-MS	[116]
	BZPs	Fe_3O_4-NH_2/ bio-MOF-1 (Zn^{2+} and adenine); DMF	Heterogeneous composite powder	40	15	0.71–2.49 ng/L	<8.8	m-µ-dSPE-LC-MS	[125]
Other NPs as sorbent									

Matrice	Analytes	Extractant phase	Extraction time [min]	Extraction recovery/EFs	RSD [%]	LOD	Methodology	Ref
Milk	Diethylstilbestrol	MWCNTs	40	-	-	5.1 µg/L	HF-SPME-HPLC	[126]
	Tylosin	TiO_2; H_2O_2 and a mild acidic	30	540	-	0.21 µg/L	HF-SLPME-UV-VIS	[127]
	Organochlorine pesticides	ZnO NPs incorporated carbon foam	45	-	2.3-10.2	0.19 – 1.64 ng/mL	µ-SPE-GC-MS	[128]
Hair	Anti-inflammatory drugs	Fe_3O_4/SiO_2/TiO_2;	70	405–2450	< 6%	-	HF-SPME-HPLC	[129]
Urine	Gd^{3+} and Gd-based contrast agents	TiO2	-	100-250	1.6-4.4	4.5–5.7 µg/L	CME-ICP-MS	[130]
	Organochlorine pesticides	LDG/G	40	-	2.7-9.5	0.22 – 1.38 ng/mL	SB-µ-SPE-GC-MS	[131]
	NSAIDs	Porphyrin functionalized GO		4.80-9.79	<10	0.5-2.0	µ-SPE-HPLC-UV	[132]

						ng/mL		
Water	Triazine and Herbicides	Octanol+Fe_3O_4 NPs	10	21–185	<11.7	0.02–0.06 µg/L	DLLME-GC-MS	[133]
	Pyrethroids	Octanol+Fe_3O_4 NPs	30	51–108	1.8-2.5	0.05–2 µg/L	HPLC-UV	[134]
	PAHs	Fe_3O_4@SiO_2@PDA-β-CD	25	21-90	<7.9	0.04-0.57 ng/mL	MDSPE-GC-FID	[135]
	Pharmaceutical drgs	MWCNTs	180	250	<9.7	0.02-0.05 µg/L	BaµE-HPLC-DAD	[136]

Application of NPs as sationary or pseudo-stationary phases in different chromatographic techniques

Metal Organic Frameworks

Analytes	MOF (metallic ion and ligand) Synthetic solvent	Stationary phase (type)	Type of column	Size	N [plates/m]	Methodology	Ref.
Chiral neurotransmitters	JLU-Liu23 (Cu^+ and TEDA and 1,3-bis(2-benzimidazol)benzene); DMF	JLU-Liu23	coated	40 cm × 75 µm i.d.	194061	CEC-DAD	[137]
Biochemical compounds	$[Zn_2(bdc)(L\text{-}lac)(DMF)]\cdot DMF$ (Zn^{2+} and L-lactic acid & H_2bdc); DMF	$[Zn_2(bdc)(L\text{-}lac)]$	coated	10 m × 0.25 mm × 1–2 µm	-	GC-FID	[138]
Drugs	γ-CD-MOF (K^+ and γ-CD); water and methanol	γ-CD-MOF	packed	10 cm × 4.6 mm × 2–5 µm	75000	LC-DAD	[139]
Xylene isomers	ZIF-8 (Zn^{2+} and 2-MIm); DMFand methanol	ZIF-8@SiO_2	packed	5 cm × 4.6 mm × 2.2 µm	216202	LC-UV	[140]
C_8 compounds	MIL-101(Fe)-NH_2 (Fe^{3+} and $H_2N\text{-}H_2bdc$); DMF	MIL-101(Fe)-NH_2@SiO_2	packed	25 cm × 3 mm × 3–5 µm	37570	HPLC-UV	[141]

Benzene derivatives	UiO-66-NH_2 (Zr^{4+} and H_2N-H_2bdc); DMF and formic acid	UiO-66-NH_2	coated	25 cm × 50 µm i.d.	-	CEC-DAD	[142]
Other NPs							
Analytes	NP material	Support/ stationary phase	Type of column	LOD	RDS [%]	Methodology	Ref.
Pharmaceutical racemates	SW-CNTs	Poly(glycidyl methacrylate-co-ethylene dimethacrylate) monoliths	packed	-	1.5-15	HPLC-UV	[143]
Mono- and divalent metal ions	MW-CNTs	1-dodecyl-3-methylimidazolium chloride	coated	18.5-124 ng/mL	2-8	CE-DAD	[144]
Volatile and non-volatile compounds, isomers and nonpolar compounds, alcohol and esters	Silica nanoparticles	[BuMIm][BF6]	coated	-	-	GC-MS	[145]
Alkylbenzenes, barbiturates, steroid hormones and alkaloids	MWCNTs	Amino-terminated alkyl chains containing polar embedded groups	packed	-	< 2%	HPLC-UV	[146]
Esters and chloroaromatics	MWCNTs	Amino-terminated alkyl chains containing polar embedded groups	packed	-	5-19	GC--MS	[147]
Applications of NPs in electrochemical sensing							
Modified electrode	Analytes	Technique	LOD (µM)	Real matrix		Ref.	
MIP-rGO/GCE	Adrenaline	DPV	0.003	Urine and formulation		[148]	
Au-Pd/rGO/GCE	Epinephrine	DPVs	0.0012	human serum		[149]	
FB-SPEs	Acetaminophen and guanine	DPV	0.01 and 0.005	Drugs and urine		[150]	
LSG/Cu-NPs	Glucose	Amperometry	0.35	Serum		[151]	
COOH-MWCNTs/CPE	L-tyrosine	Amperometry	0.014	Blood serum and milk		[152]	

MoS_2-Au/Pt@GCE	H_2O_2	Amperometry	0.39	Human serum and blood	[153]
AuNPs/PGE	microRNA-21	DPV	1×10^{-9}	Serum	[154]
PEDOT-LSG	Dopamine	DPV	0.33		[155]
CNHs/GO/GCE	4-nitrochlorobenzene	DPV	0.01	water	[156]
GQD/GCE	doxorubicin hydrochloride	DPV	0.016	Human plasma	[157]
Fe_3O_4@NiO/CPE	Quercetin and tryptophan	DPV	0.00218, 0.01423	Milk and Honey	[158]
CQDs/NH_2-fMWCNT/AgNPs/GCE	Anti-HIV drug Rilpivirine	DPV	3×10^{-5}	Biological fluids, urine, and synthetic human serum	[159]
PPyNWs/PtNPs	Dopamine	DPV	0.6	Human serum	[160]
RGO–ZnO/GCE	Dopamine	DPV	1.08	Plasma and urine	[161]
Au@CuNPs/GCE	Metronidazole		10	Human serum and pharmaceutical samples	[162]
NiO NPs-CPE	Sulfasalazine	SWV	0.002	Tablet and serum	[163]
MIPs@CuO@GCE	Dopamine		0.008	Serum	[164]

AgNPs, silver nanoparticles; AuNPs, gold nanoparticles; [BuMIm][BF6], 1-butyl-3-methylimidazolium hexafluorophosphate; BZPs, benzodiazepines; CD, γ-cyclodextrin; CME, capillary microextraction; CNHs, carbon nanohorns; CPE, carbon paste electrode; CQDs, carbon quantum dosts; DAD, diode array detector; DMA, N,N-dimethylacetamide; DMF, N,N-dimethylformamide; FB, fullerene black; GC, Gas chromatography; GCE, glassy carbon electrode; GO, graphene oxide; GQDs, graphene quantum dots; H_2bdc, terephthalic acid (benzene-1,4-dicarboxylic acid); O_2N-H_2bdc, 2-nitroterephthalic acid (2-nitrobenzene-1,4-dicarboxylic acid); HS, Headspace; Htz, 1,2,4-triazole; 2-MIm, 2-methylimidazole; ICP, inductively coupled plasma; MAA-EDMA, LSG, laser-scribed graphene; poly(methacrylic acid-ethylene glycol dimethacrylate); MIPs, molecularly imprinted polymers; MS, mass spectrometry; MS_2, Mollebydnum disulfide; MW-CNTs, Multi-walled carbon nanotubes; NH_2-H_2bdc, 2-aminoterephthalic acid; NiO, nickel oxide; LC, Liquid chromatography; PCPs, personal care products; PDA, photodiode array detector; PEDOT, poly(3,4-ethylenedioxythiophene; PGE, pencil graphite electrode; PP, polypropylene; PPy NWs, polypyrrole nanowires; PtNPs, platinum nanoparticles; RGO, reduced graphene oxide; RSD, inter-day relative standard deviation; SLPME, solid/liquid-phase microextractio SPE, Solid phase estraction; SPME, Solid phase microextraction; SUHs, sulfonylurea herbicides; TEDA, triethylenediamine; UV, ultraviolet

5.3. Other applications

5.3.1. Composite fillers

Some NPs such as CNTs impart special characteristics to the composites when used as filler. They not only give the mechanical strength but also improves electrical conductivity, thermal strength, and electromagnetic shielding. Hence, they have been used in many commercial products such golf clubs and tennis rackets. CNT composites mature the formability of the module, which is imperative for small components such as the lens unit of a mobile phone [165].

Low silica NPs loaded polypropylene composites exhibited enhanced tensile strength [166]. Silica NPs were used as surfactants and fillers for latexes made by miniemulsion polymerization [167]. Ag NPs deposited boron nitride nanosheets were also used as fillers for the polymeric composites which exhibited enhanced thermal conductivity [168].

5.3.2. Agriculture

Agriculture is an extremely important sector to fulfil the growing food demands. However, it is also facing several threats due to increasing population, climate change, urbanization, and food contaminations due to pesticides and fertilizers. The world population is expected to increase to 9 billion by 2050. Thus, the role of the modern technologies should be fully exploited to revolutionize the agriculture sector. Thus, several types of NPs have been utilized for this purpose. Few examples are presented in coming paragraphs.

Most of the pesticides and fertilizers are wasted and do not reached to the affected sites due to leaching, uncontrolled spray, hydrolysis and photo and microbial degradation. NPs and nanocapsules can efficiently deliver the pesticides to the affected target sites without damaging the normal parts of the plants. Moreover, NPs can be employed to develop the sensors that can determine the required amount of pesticides for a crop. The use of fertilizers and pesticides can be reduced based on their concentrations in the final products.

This is an unfortunate reality that excessive and non-discriminative use of persistent pesticides in last six decades in contamination of soil as well as ground water resources leading to health issue in the human and wildlife. The first and foremost step in purification and treatment of such lands and water is to measure the current concentrations of persistent organic pesticides [169]. Highly efficient pesticide residue determination can be performed using NPs based sensing method. The content of nutrients and moisture of soil can be measured for effective use of fertilizers. Nanoencapuslated fertilizers will release slowly resulting in the reduced consumption.

ZnO NPs treated peanut seeds showed improved seed germination, plant growth, enhanced stem and root growth. ZnO NPs colloidal solution can be utilized as nanofertlizers for only providing the nutrients to soil but reviving its chemistry without use of chemical fertilizers. Moreover, such fertilizers are required in significantly less amount compared to conventional fertilizers. Some NPs and nanopowders can serve the purpose of pesticides [170].

Different kind of NPs have been employed in agriculture sector for performing different actions. **Figure 6** shows the applications of carbon-based nanomaterials in agriculture.

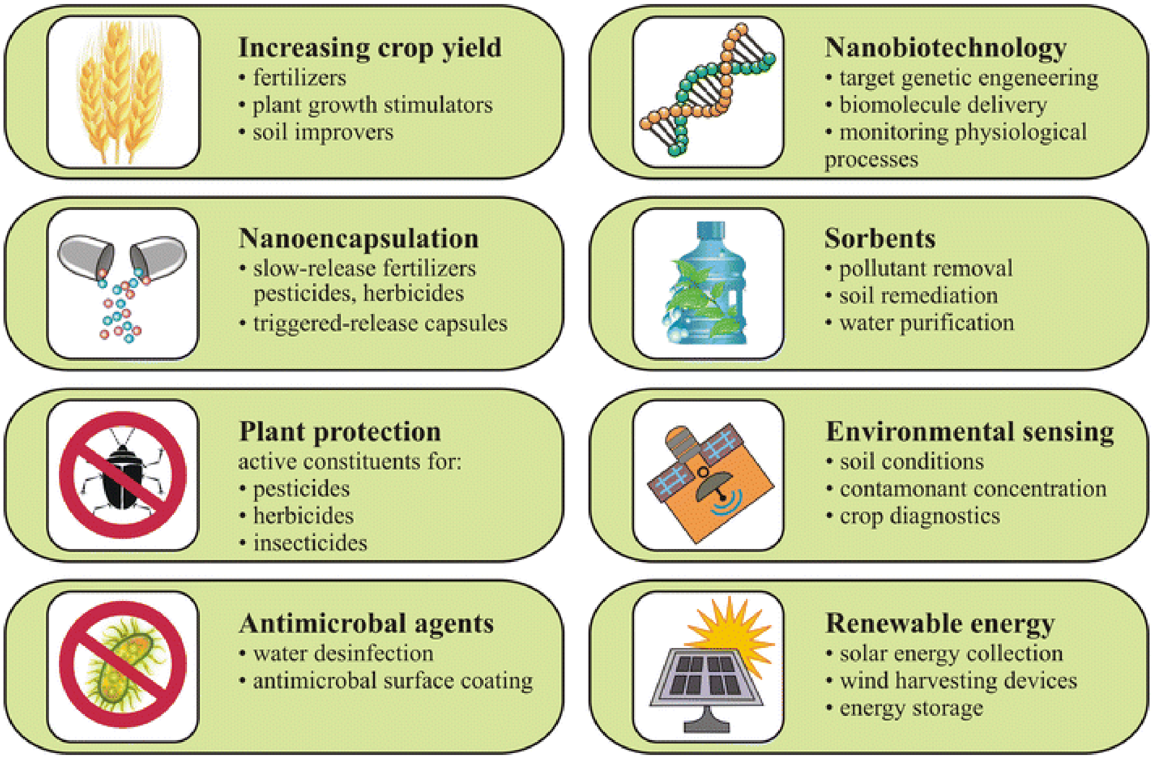

Figure 6. Potential applications of carbon-based nanomaterials in environmetal and agricultural sectors. Reprinted from [171] . Copyrights (2016) Olga Zaytseva & Günter Neumann.

5.3.3. Gas sensors

Metal oxide NPs are widely used in gas sensing applications. 3D hierarchically porous ZnO supported Au NPs were used as gas sensors utilizing the high accessibility of porous ZnO and catalytic activity of AuNPs [172]. The flame made SnO_2 NPs presented high and quick response to both reducing (propanal) and oxidizing (NO_2) gases [173]. Semiconducting $CuFe_2O_4$ NPs were used for the sensing of reducing gases and showed fast response and recovery [174]. AuNPs modified metal oxides have been largely used in conductometric gas sensing [175]. A recent review describes gas sensing applications of semiconducting metal oxides [176]. Though not exploited commercially, some carbon based nanostructures such as CNTs, graphene, nanofibers have shown excellent potential for gas sensing compared to existing materials [177].

5.3.4. Biomedical

The most prominent applications of NPs in biomedical sciences include disease diagnosis and treatment. NPs have been employed as contrast agents in many kinds of imaging. They have been used in targeted drug delivery for safe and effective treatment of many diseases. Theranostic NPs can be used for diagnosis as well as treatment [178].

Blood brain barrier is a distinctive restricting that inhibits the entrance of many substances including the therapeutics into the central nervous system. There are numerous diseases that affect CNS, thus the delivery of drugs to target site is very crucial in the treatment. New efforts have been focused on the developing the design of NP based drug delivery systems [179].

Unique properties arising from quantum size effects and the large surface area of magnetic NPs affectedly transform some of the magnetic features and display superparamagnetic phenomena and quantum tunneling of magnetization, since each NP can be assumed as a single magnetic domain. They have also been employed in biomedical applications like cellular therapy, tissue repairing, magnetic resonance imaging, drug delivery, etc. [180]. Though conventional magnetic NPs such as iron oxide NPs have been successfully employed in biomedical applications both in vitro and in vivo but still they have limitations such as low magnetic moment, low sensitivity in MRI, and low drug loading capacity, thus various new forms of magnetic NPs have been developed over the time, detail of which can be studied in a review dedicated to this subject [181].

Due to large surface area pore volume, and functionalization with other materials, mesoporous silica NPs showed effective drug loading in drug delivery applications [182]. Mesoporous silica NPs which release their drug cargo in response to ultrasound are discussed in a feature article [183]. Au NPs, due to their unique optical, chemical, and surface properties, have also been widely employed in diagnostics as well as therapy [184]. Upconverting NPs overcome many disadvantages of conventional fluorophores such as photobleaching, high background noise from autofluorescence, and photodamage to biomaterials. Upconverting NPs have been used both in biodetection and imaging [185].

Layered double hydroxides (LDHs), also known as hydrotalcites or anionic clays, denote an attractive class of inorganic materials. Typically, LDHs are two-dimensional nanostructured materials comprising of positively charged layers of metal hydroxides with charge-balancing anions and some water molecules situated in between the layers [186]. They have high adsorption capacity, good anion exchange capability, excellent biocompatibility, and pH dependent solubility. These properties are exploited in designing LDH NPs based drug delivery applications [187].

Biodegradable polymeric NPs have also demonstrated exciting applications in biomedical discipline. For example, poly(lactic-co-glycolic acid) (PLGA) is one of the most magnificently developed biodegradable polymers. PLGA NPs has fascinated substantial consideration due to their striking properties due to their excellent biodegradability and biocompatibility, FDA and European Medicine Agency approval in drug delivery systems for parenteral administration, well described methods for different kind of drugs, effective drug protection, potential of sustained release, surface modification for better interaction with biological systems, and potential for targeting specific organs or cells [188]. Stimulus-responsive polymeric NPs are the smart NPs that can change their structure, shape, and property after exposure to external factors such as pH, temperature, magnetic field, and light. They have been explored both in drug delivery and *in vitro*/*in vivo* imaging [189]. Molecularly imprinted NPs can show enhanced affinity and selectivity towards the target biomolecules and their role in biomedical applications is discussed in a recent review [190].

5.3.5. Energy

NPs have emerged as significant contributors in the future energy technologies. They are playing a key role in development of renewable energy systems, reformers in the production of hydrogen from different carriers, electrocatalysts in fuel cells, and many other applications. This is due to increasing demand of activity per unit area and decreasing the use of costly standards such as platinum in various processes [191]. Plasmonic NPs have increased the performance and feasibility of photovoltaic devices [192]. NPs along with some other materials or alone have been employed as anodes in lithium ion batteries to enhance their reversible capacity, cyclic performance, and rate capability. Co_3O_4 NPs obtained with size of 10−30 nm were homogeneously anchored on graphene sheets as spacers to keep sheets separate and fully utilize the potential of electrochemically active NPs and graphene sheets for energy storage in lithium ion batteries [193]. Hollow structured Co_3O_4 NPs prepared via template free synthesis showed excellent performance as anodes in lithium ion batteries [194]. Biomass derived carbon NPs have also been used as anodes in sodium and lithium ion batteries to have enhanced performance [195].

Water splitting into hydrogen and oxygen is an excellent way of energy generation and storage. This process can be accomplished using one of the photochemical, electrochemical, and photoelectrochemical methods depending on the nature of the catalyst. In electrochemical water splitting efficient electrocatalyst are required for hydrogen or/and oxygen evolution reactions. Nickel phosphide (Ni_2P) NPs proved excellent catalyst for both hydrogen and oxygen evolution. The high activity was due to formation of the core–shell ($Ni_2P/NiOx$) structure under catalytic conditions [196]. A single bifunctional material that can catalyze both OER and HER can reduce the cost of the process by simplifying it. However, the challenge lies in the fact that it should efficiently catalyze both the OER and HER and should attain a low overall overpotential and provide an improved current density. Ni_3FeN NPs synthesized from Ni-Fe LDH nanosheets was utilized as a bifunctional material and it showed excellent performance due to metallic character, unique electronic structure, high water adsorption capacity, and increased activity due to small sized particles [197]. Co-doped $NiSe_2$ NPs film electrodeposited on a conductive Ti plate showed excellent performance both for HER and OER in strongly basic media [198]. Earth-Abundant Iron Diboride (FeB_2) NPs also proved excellent bifunctional electrocatalyst for overall water splitting [199].

5.3.6. Environment

NPs are widely used in environmental remediation for photodegradation, detection, selective removal, and adsorption of environmental pollutants. Depending on the nature of the core materials, NPs can have unique optical, electrical, and magnetic properties that can be utilized in environmental applications. Moreover, these NPs can be functionalized with the moieties that can selectively capture target pollutants. NPs have been used for extraction and pretreatment of the analytes before their detection by analytical instruments. NPs have been used SERS substrates for pollutant detection. Some NPs such as Au and QDs have been used to enhance the sensitivity of the detection through signal transition [200]. Heterogeneous photocatalysis is one of the inexpensive choices for degradation of organic pollutants from waste effluents. The reusability of the catalysts over several cycles reduces the cost of the process. However, the desired photocatalysts should demonstrate efficiency under the visible light for real-life applications. The metal NPs modified photocatalysts have been used because they extend the light absorption capacity to broad range solar spectrum instead of confining it to a certain wavelength. However, this is dependent on particle shape, size, and interactions between the particles. Localized surface plasmon resonance (LSPR) effect in metal NPs like Au, Ag, and Pt enhances photocatalytic activity under visible light. Ag NP loaded Ag_2SO_3 photocatalysts were used for degradation of Rhodamine B and phenol under visible light [201]. Semiconductor oxide NPs such TiO_2 and ZnO have been widely used in photocatalytic degradation of pollutants. Magnetic NPs have been used in extraction, removal, and degradation of pollutants [200].

6. Conclusion

In this review, we have discussed the role of NPs in modern science and technology. Beginning from the basics of NPs, we describe their types, synthesis methods, general and specific properties, and advanced applications in different areas (Figure 7). We have critically reviewed the synthesis methods for NPs with their advantages, limitations, and the way they influence the properties of resulting NPs. Properties of the NPs such as size, shape, and surface area have huge impact on the target applications. Thus, modern synthesis methods are designed to prepare size-, shape-, and surface area-controlled NPs. Depending on the nature of the parent material, NPs can have special optical, electrical, magnetic, and microbial properties, which encourage their application in corresponding scientific area. At the end, we briefly enlist few areas where NPs have been widely used and made a big difference. The role of different kind of NPs in analytical sample preparation, electrochemical sensing, optical sensing, composite fillers, agriculture, gas sensors, biomedical, energy and environment is briefly summarized with few relevant examples.

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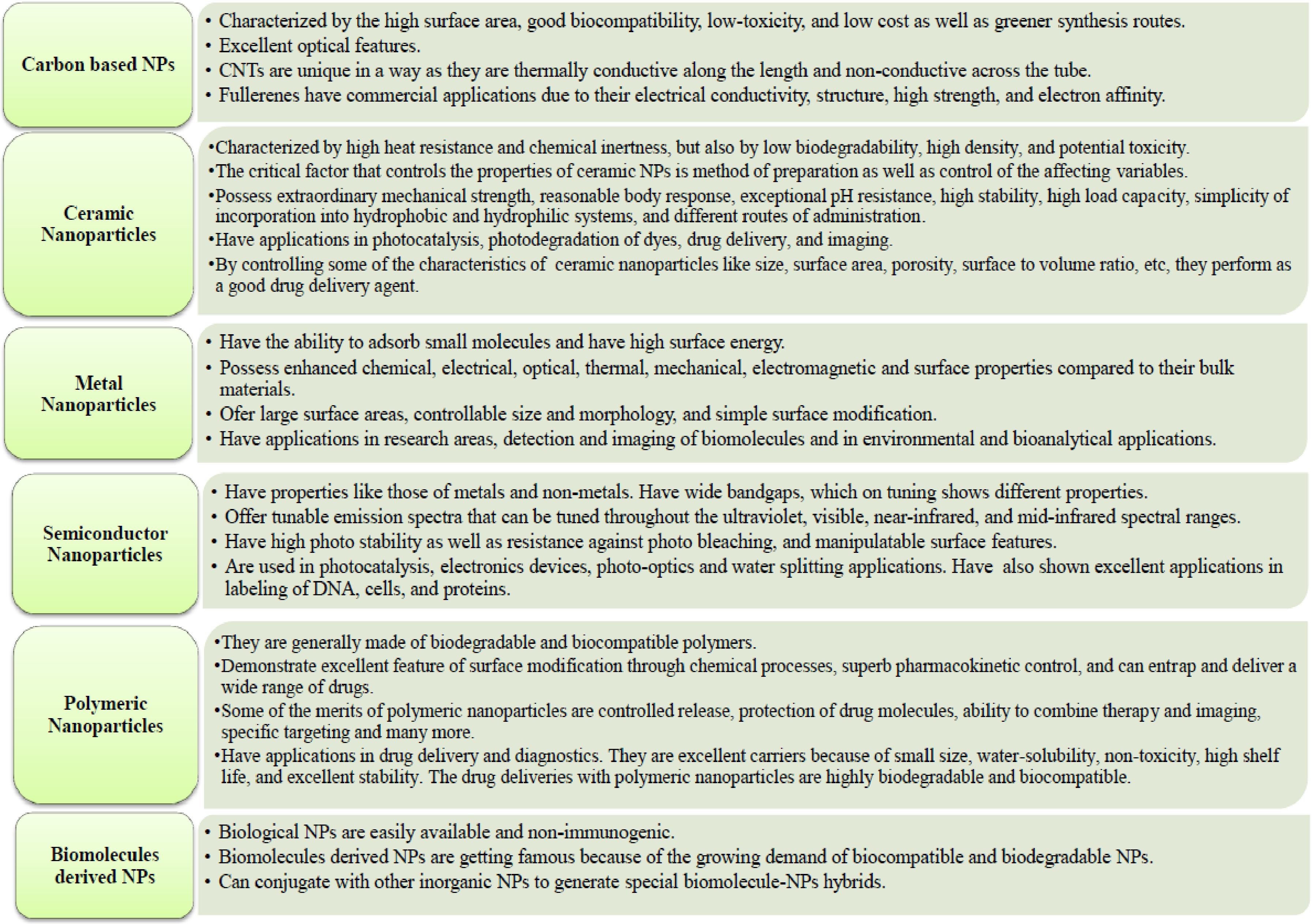
Figure 7. Comparison of the properties and application of the different types of NPs



Carbon based NPs	• Characterized by the high surface area, good biocompatibility, low-toxicity, and low cost as well as greener synthesis routes. • Excellent optical features. • CNTs are unique in a way as they are thermally conductive along the length and non-conductive across the tube. • Fullerenes have commercial applications due to their electrical conductivity, structure, high strength, and electron affinity.
Ceramic Nanoparticles	•Characterized by high heat resistance and chemical inertness, but also by low biodegradability, high density, and potential toxicity. •The critical factor that controls the properties of ceramic NPs is method of preparation as well as control of the affecting variables. •Possess extraordinary mechanical strength, reasonable body response, exceptional pH resistance, high stability, high load capacity, simplicity of incorporation into hydrophobic and hydrophilic systems, and different routes of administration. •Have applications in photocatalysis, photodegradation of dyes, drug delivery, and imaging. •By controlling some of the characteristics of ceramic nanoparticles like size, surface area, porosity, surface to volume ratio, etc, they perform as a good drug delivery agent.
Metal Nanoparticles	• Have the ability to adsorb small molecules and have high surface energy. • Possess enhanced chemical, electrical, optical, thermal, mechanical, electromagnetic and surface properties compared to their bulk materials. • Ofer large surface areas, controllable size and morphology, and simple surface modification. • Have applications in research areas, detection and imaging of biomolecules and in environmental and bioanalytical applications.
Semiconductor Nanoparticles	• Have properties like those of metals and non-metals. Have wide bandgaps, which on tuning shows different properties. • Offer tunable emission spectra that can be tuned throughout the ultraviolet, visible, near-infrared, and mid-infrared spectral ranges. • Have high photo stability as well as resistance against photo bleaching, and manipulatable surface features. • Are used in photocatalysis, electronics devices, photo-optics and water splitting applications. Have also shown excellent applications in labeling of DNA, cells, and proteins.
Polymeric Nanoparticles	•They are generally made of biodegradable and biocompatible polymers. •Demonstrate excellent feature of surface modification through chemical processes, superb pharmacokinetic control, and can entrap and deliver a wide range of drugs. •Some of the merits of polymeric nanoparticles are controlled release, protection of drug molecules, ability to combine therapy and imaging, specific targeting and many more. •Have applications in drug delivery and diagnostics. They are excellent carriers because of small size, water-solubility, non-toxicity, high shelf life, and excellent stability. The drug deliveries with polymeric nanoparticles are highly biodegradable and biocompatible.
Biomolecules derived NPs	• Biological NPs are easily available and non-immunogenic. • Biomolecules derived NPs are getting famous because of the growing demand of biocompatible and biodegradable NPs. • Can conjugate with other inorganic NPs to generate special biomolecule-NPs hybrids.

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