NANOSORBENTS IN PURIFICATION OF WASTEWATER AND REMEDIATION OF CONTAMINATED SOIL: A REVIEW

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Abstract

Nanoparticles have become increasingly relevant in science and industry. This is understandable due to its varied applications from sports to medicine to cosmetics and even the military. This review focuses on the removal of heavy metal ions from contaminated soil and wastewater using nanosorbents. Well-established physical and chemical methods were examined but the focus was on green synthesis. Contaminated soil remediation and wastewater purification using nanosorbents follow the bottom up synthetic approach. Green synthesis is more advantageous than chemical approaches due to its cheapness and being environmentally friendly. Green synthesis also avoids hazardous and toxic methods of nanoparticle synthesis. It also provides a use for otherwise harmful and invasive plant materials. The importance of nanoparticles to biofuels, cosmetics and drugs and medicine was also evaluated. Challenges to more adoption of nanoparticles were also highlighted.

Acronyms

AgNPs	silver nanoparticles
ALD	atomic layer deposition
BET	Brunauer–Emmett–Teller
CNT	carbon nanotube
DLS	dynamic light scattering
DNA	deoxyribonucleic acid
EBFC	enzymatic biofuel cell
EDS	energy dispersive X-ray spectroscopy
EESD	electrochemical energy storage device
FTIR	Fourier transforms infrared
MWCNT	multi-walled carbon nanotube
NLC	nanostructured lipid carrier
NP	nanoparticle
PbSe	lead selenide
PDDA	poly(diallyldiamethylammonium)
QD	quantum dot

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ROS	reactive oxygen specie
SEM	scanning electron microscopy
SWCNT	single-walled carbon nanotube
TEM	transmission electron microscopy
TGA	thermogravimetric analysis
UCNP	upconverting nanoparticle
XPS	X-ray photoelectron spectroscopy
XRD	X-ray diffraction

1. Introduction

The history of modern nanotechnology cannot be complete without the mention of Richard Feynman. In fact, he was regarded as the father of modern nanotechnology. But the word “nanotechnology” was coined by the Japanese scientist, Norio Taniguchi almost fifteen years after Feynman’s works [1]. Nanoscience or nanotechnology is defined as the branch of science or engineering that deals with the creation of objects or materials less than 100 nm in at least one dimension [2]. Nanotechnology consists mainly of processing of separation, consolidation and deformation of materials by one atom or molecule. Nanobiotechnology and nanomedicine on the other hand deal with the intra- and intercellular molecular processes geared chiefly towards the control and manipulation of cell processes [3]. The small dimensions associated with nanoparticles allow them some unique properties like more surface functionality. Their physical properties also greatly differ from that of the bulk material in areas like electronic, optical and magnetic features [4]. Wastewater and contaminated soil contain heavy metals. These pose serious environmental risks both to flora and fauna. Several materials can be used as adsorbents for heavy metal removal from both wastewater and contaminated soil. These include but not limited to activated carbon, zeolites and metal oxides [5].

2. Synthesis of nanoparticles

Synthesis of nanoparticles is usually via chemical reduction processes. The reducing agents used include sodium borohydride ($NaBH_4$), hydrazine (N_2H_4) and methanal (CH_2O). These reducing agents are expensive and harmful to the environment, hence the need for more cost-effective and environmentally friendly reducing agents. This paves the way for green synthesis of nanoparticles. The advantages of the green synthesis over chemical include low cost, environmental benignity, reduction in physiological toxicity and enhancement of biological compatibility [6]. **Figure 1** below shows a diagram of the various nanoparticle synthetic pathways [7].

Synthesis of nanoparticles can be achieved either by “top–down” or “bottom–up” approach. Top–down approaches to the synthesis of nanoparticles have proven to be effective in 2D nanomaterials production. The common methods employed include but not limited to chemical exfoliation (lithium intercalation and acid etching) and sonication-assisted liquid phase exfoliation [8]. Synthesis of monodisperse 20 nm upconverting nanoparticles (UCNPs) was achieved via classical thermal co-precipitation. Top–down synthesis is ideal for plastics in the micro and nanometric ranges because it conforms to homogeneity in size and shape which is lacking when bottom–up approach is used to produce these nanomaterials [9].

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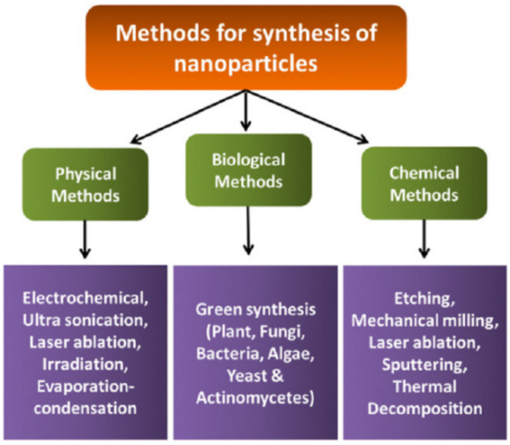

Figure 1. Synthesis of nanoparticles by Khandel, et al. (2018) [7].

The bottom–up approach on the other hand entails using atoms as building blocks to fabricate nanomaterials. Atomic layer deposition (ALD) is a well-known bottom–up approach because it involves the addition of atoms in a layer-by-layer pattern [10]. Green synthesis is a bottom–up approach. In green synthesis, the chemical reducing agent in chemical reduction is replaced by a natural product extract as depicted in **Figure 2** below.

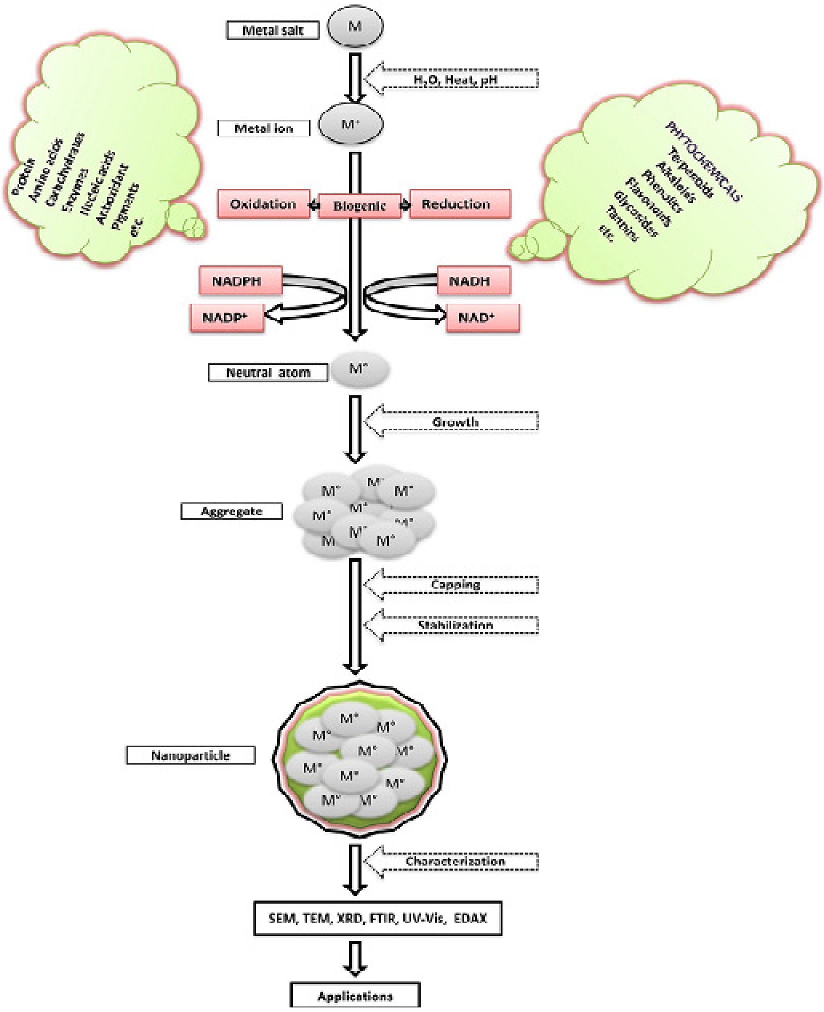

Figure 2. Possible mechanism of biologically mediated synthesis of NPs by Hussain, et al. (2016) [11].

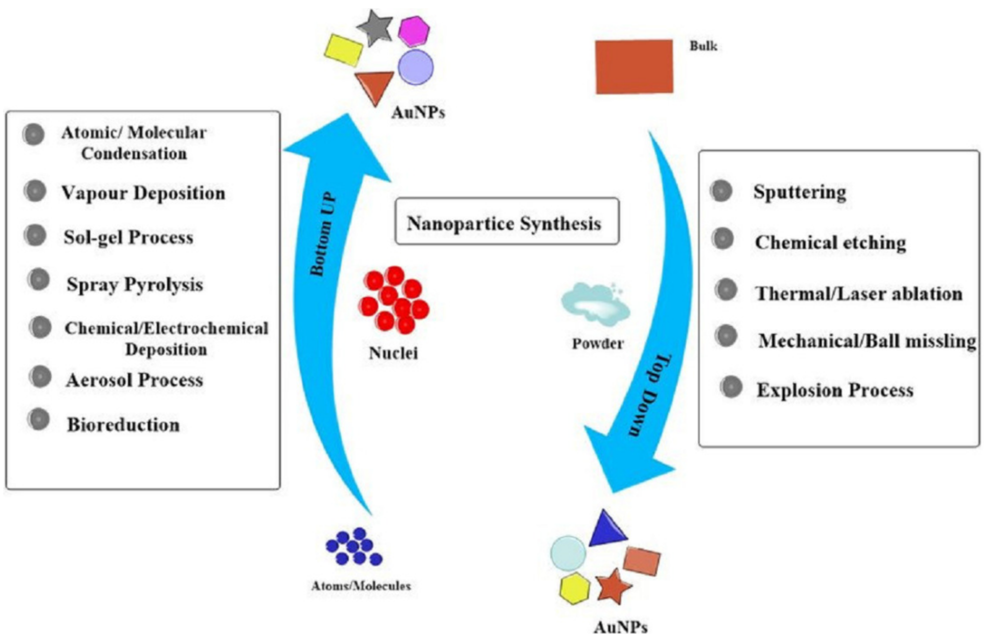

Figure 3. Top–down and bottom–up NPs synthesis by Ovais, et al. (2017) [12].

Green synthesis however is an alternative to the physical and chemical methods depicted in **Figure 3** above. This method provides an environmentally friendly way to synthesize nanoparticles. Green synthesis can be achieved using yeasts, moulds, algae and plants or their products [13]. The molecules of these plants facilitate nanoparticle synthesis by reduction. The synthetic process of metallic nanoparticles is initiated by adding plant extracts to aqueous solutions of metal ions. The extracts could be from leaves, roots, corms, stems or fruits. The sugar, flavonoid, polymers, enzymes and other materials present in the plant extracts act as reducing agents to produce the desired nanoparticles [13]. **Figure 4** below shows the biological synthetic pathway to nanoparticle production.

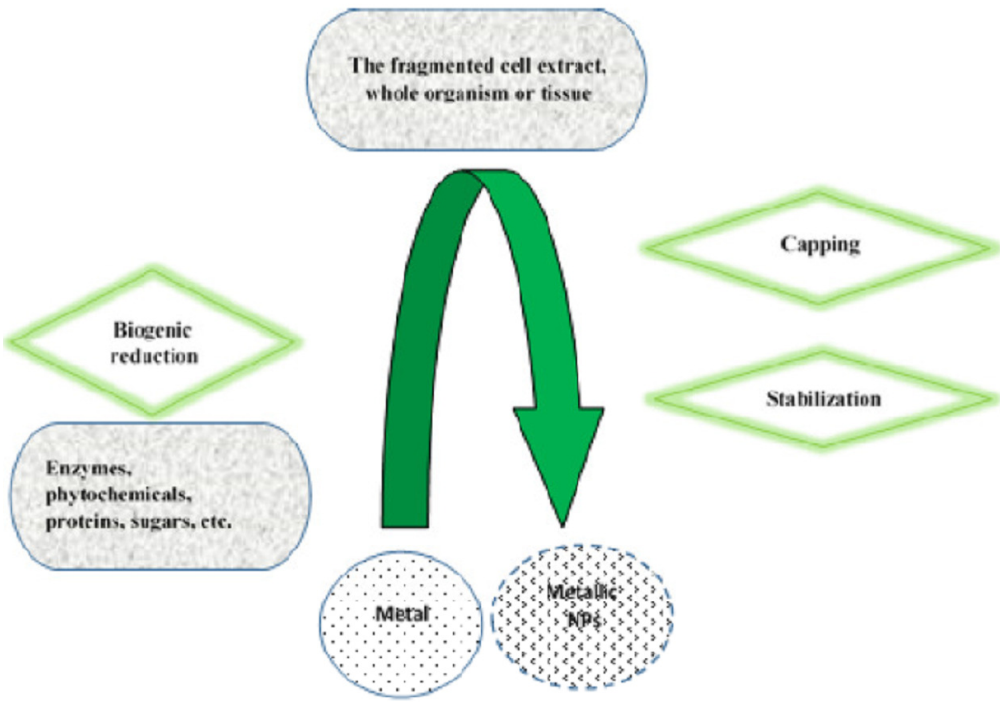

Figure 4. Biological synthesis of NP by Nadaroglu, et al. (2017) [13].

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3. Classification and characterization of nanoparticles

Nanoparticles (NPs) are broadly classified into three: (i) natural, (ii) incidental and (iii) engineered. Natural NPs are nanoparticles that come about through normal physical, chemical and biological processes. They exist naturally and examples include naturally formed metal-based, organic and inorganic nanoparticles such as AgNP, virus NPs, and exosomes. Incidental NPs as the name implies are generated unintentionally as by-products in processes such as combustion and corrosion. Some examples are metal- and carbon-based nanoparticles. Engineered NPs are manufactured specifically for certain purposes like industrial and medical applications. Examples include zinc oxide (ZnO) and titanium dioxide (TiO_2) [14]. Nanoparticles can also be classified based on physical parameters like electrical charge, or via chemical characteristics like the composition of nanoparticle core or shell or origin or based on size [15]. In addition, NPs can also be classified into organic, inorganic and carbon-based nanoparticles. Organic NPs include dendrimers, micelles, liposomes and ferritin as depicted in **Figure 5**. They are known as polymer NPs. They are generally non-toxic and biodegradable whereas inorganic NPs are NPs that are not made of carbon such as metal and metal oxide-based NPs [16].

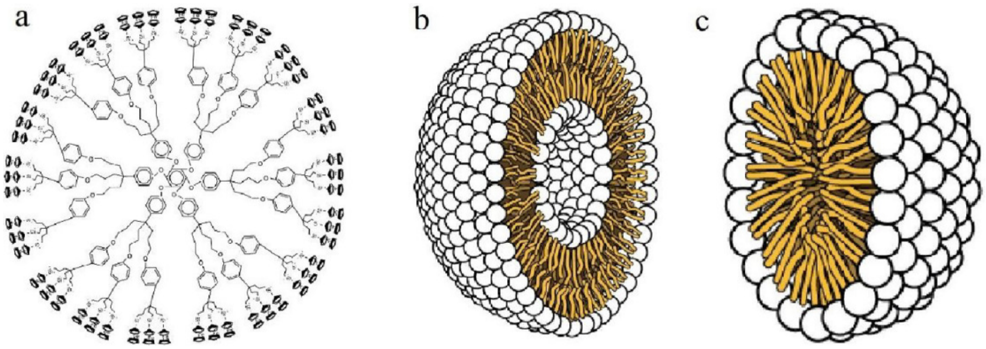

Figure 5. Organic NPs: **(a)** dendrimers, **(b)** liposomes and **(c)** micelles by Ealia & Saravanakumar (2017) [16].

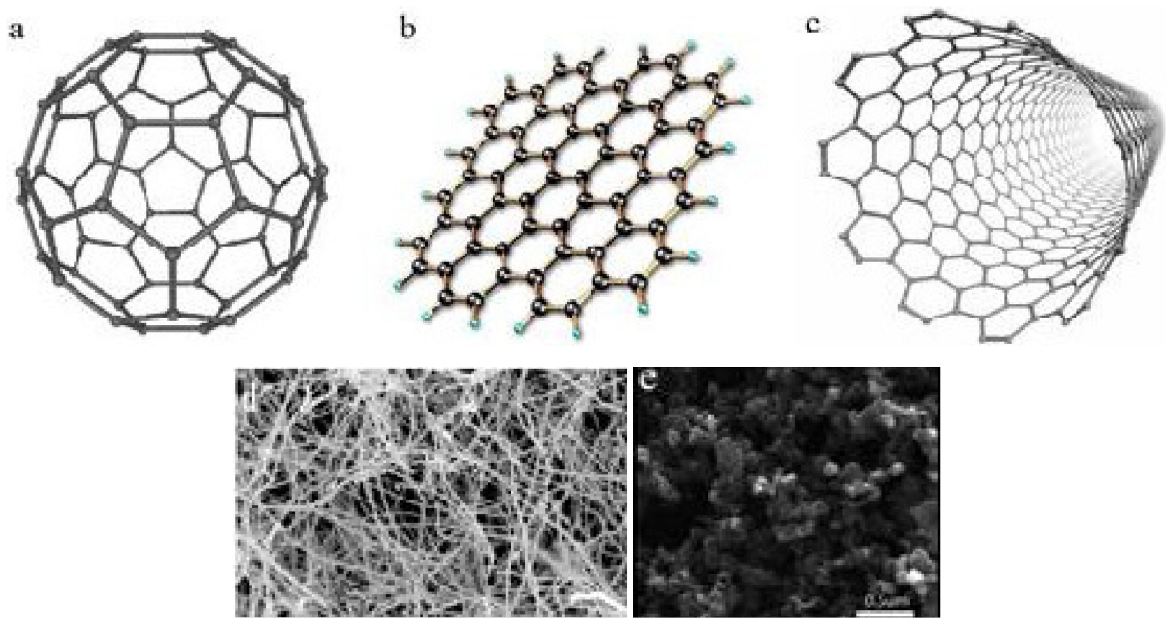

Figure 6. Carbon-based NPs: **(a)** fullerenes, **(b)** graphene, **(c)** CNTs, **(d)** carbon nanofibers and **(e)** carbon black by Ealia & Saravanakumar (2017) [16].

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Carbon-based NPs are made principally from carbon. Examples include carbon nanotubes (CNTs), graphene, carbon nanofibers, carbon black, and fullerenes as shown in **Figure 6** above. CNTs are of two types: single-walled carbon nanotubes (SWCNTs) and multi-walled carbon nanotubes (MWCNTs). Single-walled carbon SWCNTs have also been observed to exhibit high antimicrobial activity than MWCNTs. This increase in activity is because SWCNTs can penetrate better into cells which is a direct function of their smaller nanotube diameter [17].

A higher percentage of the contribution to the properties of NPs is from the surface atoms and energy. This is because NPs possess a much higher surface-to-mass ratio than their bulk materials. Different characterization methods are dependent on the physical properties of the nanoparticles, hence providing an incomplete view of the NP characteristics. The characterization of NPs also runs the risk of affecting the measured quantities [4]. Some of these characterization methods or techniques include transmission electron microscopy (TEM), X-ray diffraction (XRD), dynamic light scattering (DLS), Fourier transform infrared (FTIR) spectroscopy, scanning electron microscopy (SEM), X-ray photoelectron spectroscopy (XPS), energy dispersive X-ray spectroscopy (EDS), thermogravimetric analysis (TGA), and Brunauer–Emmett–Teller (BET) analysis.

4. Applications of nanoparticles

4.1. Biofuels

The morphology, size and structure of metal catalysts and the use of electrolytes were also noted to improve the efficiency of this reduction by enhancing the adsorption of CO_2. This interface-enhanced CO_2 reduction reaction (RR) is also observed in Ag–CeO_x, demonstrating its wide usage and application. Au–CeO_x interface shows much higher activity and Faradaic efficiency that either Au or CeO_x alone for CO_2RR as depicted in **Figure 7** below [18].

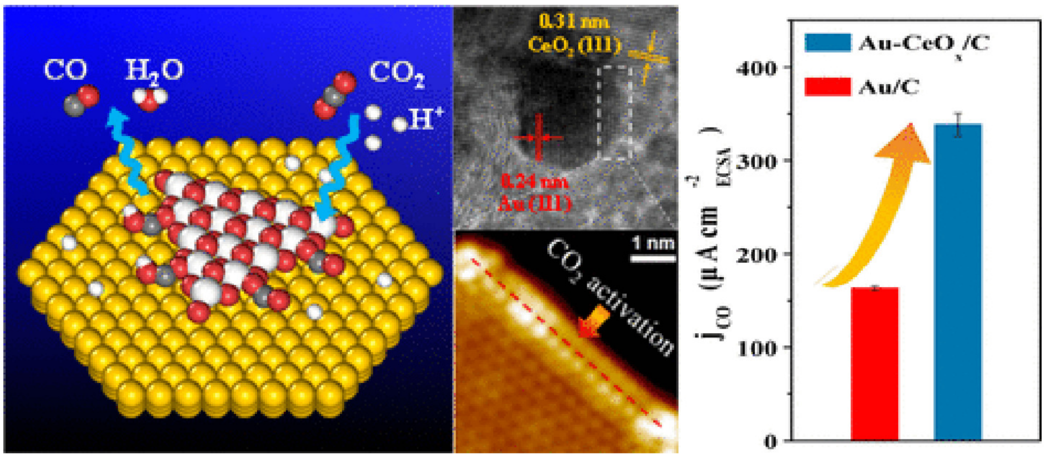

Figure 7. Nanoparticles in biofuel cells by Gao, et al. (2017) [18].

Enzymatic biofuel cells (EBFCs) have recently been used to harness the hidden electrical energy found in the chemical bonds of replicate fuel cells. These biological fuels include lactate, fructose, starch and glucose and are being catalyzed with the assistance of redox enzymes [19]. Nanomaterials and nanocomposites are being currently utilized in the research for effective electrode materials in solar cells, supercapacitors and biofuel cells.

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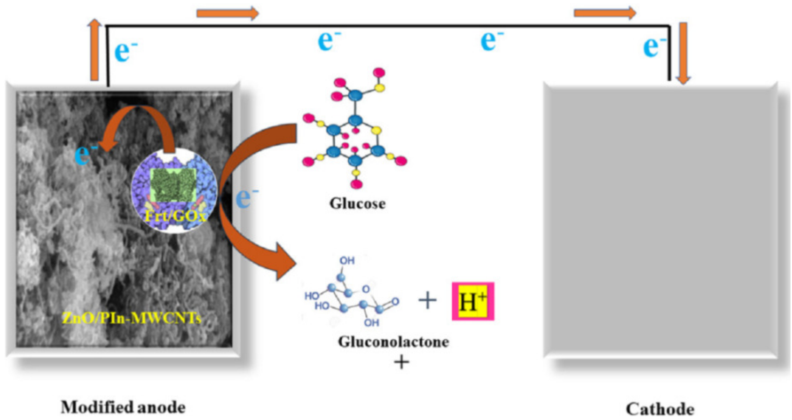

Figure 8. Assembly of fabricated bioanode by Inamuddin, et al. (2020) [19].

Nanotechnology has found great use in improving the property of electrochemical energy storage device (EESD) as shown in **Figure 8** above.

4.2. Medicine and drug delivery

Nanomedicine deals with NP and microparticle-based drug delivery systems for systemic applications. These have various advantages over the non-formulated and free drug delivery systems. NPs can target therapeutics in areas of the body that other conventional drugs cannot reach. These diagnostic and therapeutic nanoparticles broadly fall into two groups, namely inorganic (silica, gold, iron oxide) and organic (micelles, liposomes, polymeric) [20]. **Figure 9** below shows the clinically relevant nanoparticle types.

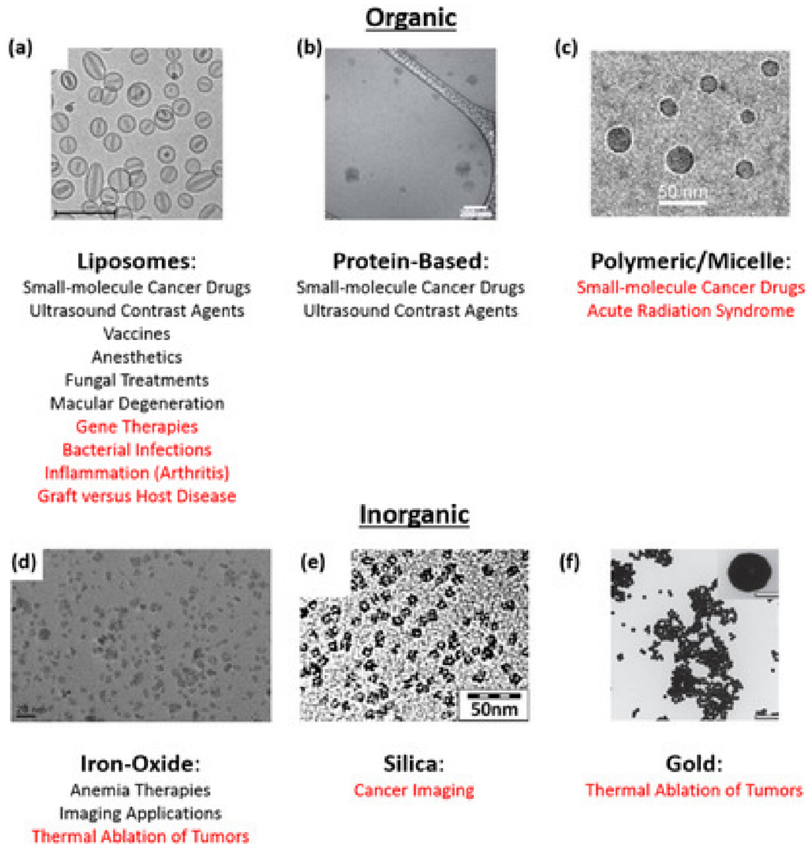

Figure 9. Clinically relevant NPs types by Anselmo & Mitragotri (2016) [20]: already approved applications (black) and applications in clinical trials (red).

NPs have for many years been used as chemotherapeutics in cancer treatment as carriers. They also find use in delivery of many anticancer therapeutic agents like DNA inhibitor oligonucleotides and antisense oligonucleotides among many other molecular targeting agents. NPs are also used as strong antigen or adjuvant carriers in vaccine development [21].

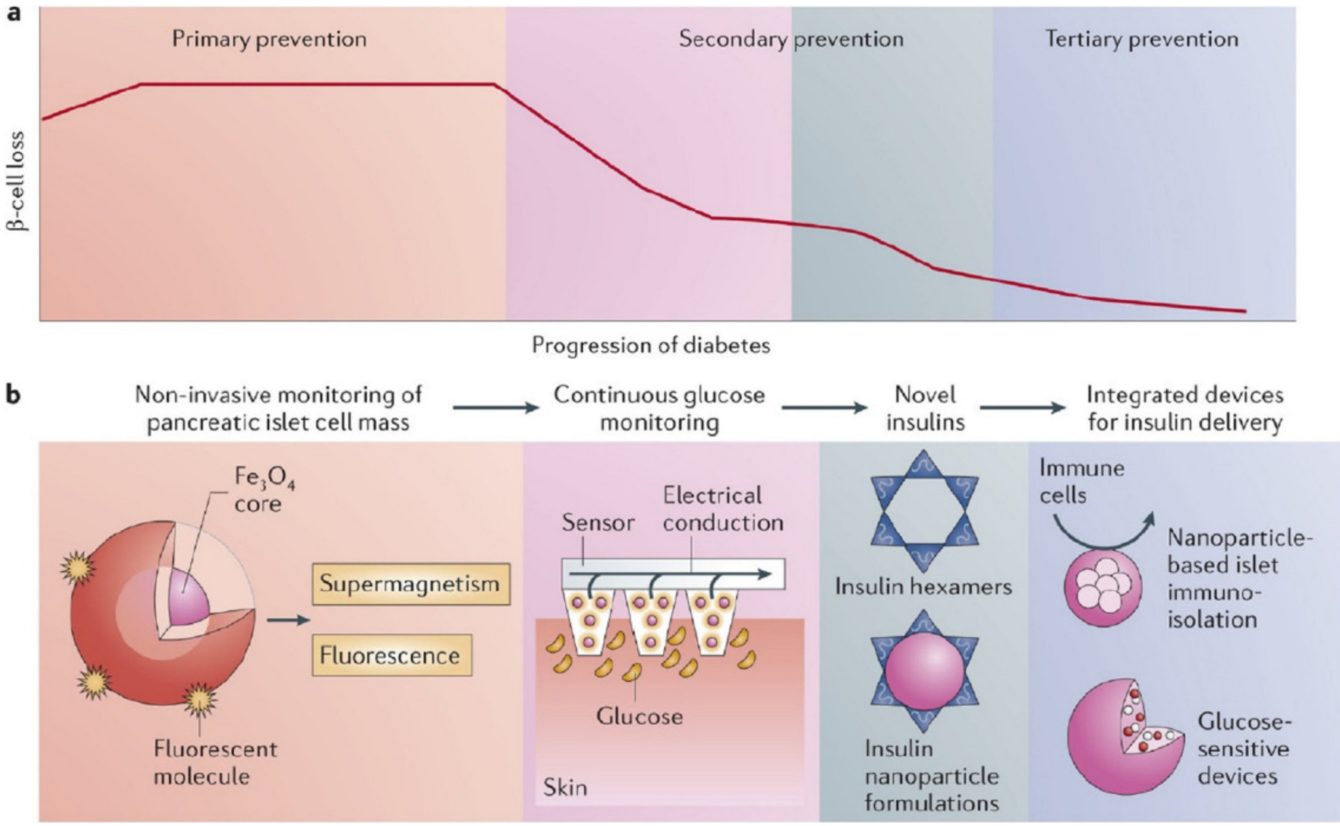

Figure 10. Nanotechnology-based approaches to address challenges in the diagnosis and treatment of diabetes by Veiseh, et al. (2015) [22].

In **Figure 10** above, there is shown the progression of diabetes from primary to tertiary and goes further to show the nano-based monitoring and treatment for each stage. NPs enhance the pharmacodynamics of insulin to better suit the physiological needs of the body. **Figure 10** also depicts how NPs can be used to maintain healthy normoglycaemic levels in those suffering from diabetes.

4.3. Nanoparticles in plants

Seed germination, growth and general development of plants can be given a boost via engineered nanotubes. The effect of NPs on plants is a function of their chemical composition, size, surface area, reactivity and dosage. Seed germination in soybean has been reported to be improved by the application of nano-SiO_2 and nano-titanium dioxide (nano-TiO_2). This is achieved by increasing the rate of nitrate reductase. Silica, palladium, gold and copper NPs also have a pronounced effect on lettuce seeds [23]. NPs are known to be transported in plants from their roots, stem and leaf to other parts of the plant via their vascular system. The degree of movement whether upward through the xylem or downward through the phloem is dependent on many factors including NP delivery method employed, plant species and total time of exposure. NPs transport in plants can increase the metal content of plants [24].

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4.4. Detection applications

Chemirestive metal-oxide NPs find use in conductometric sensor devices. This is the same for catalytic noble metal nanoclusters. Thin film arrays of varying rutile / anatase ratios are produced via gas-phase deposition of rutile titanium dioxide (TiO_2) clusters into an amorphous matrix. To change or customize the gas sensing properties of chemiresistors, catalytically active nanoclusters are added to the sensor surface [**25**]. Gold NPs are used in label-free detection methods. Loss of steric or electrostatic stabilization triggers aggregation of functionalized AuNPs. A formation of unstable AuNPs as a result of neutralization of surface charges promotes this aggregation which results in a color change. In the presence of a positively charged poly(diallyldiamethylammonium chloride) (PDDA), AuNPs aggregate as a result of electric neutralization. Due to high negative charge density of heparin, PDDA is bound strongly via electrostatic interaction to form a stable complex [**26**]. **Figure 11** below shows the effect of introduction of positively charged PDDA on AuNPs aggregation caused by electric neutralization. The concentration of heparin determines the degrees of color change.

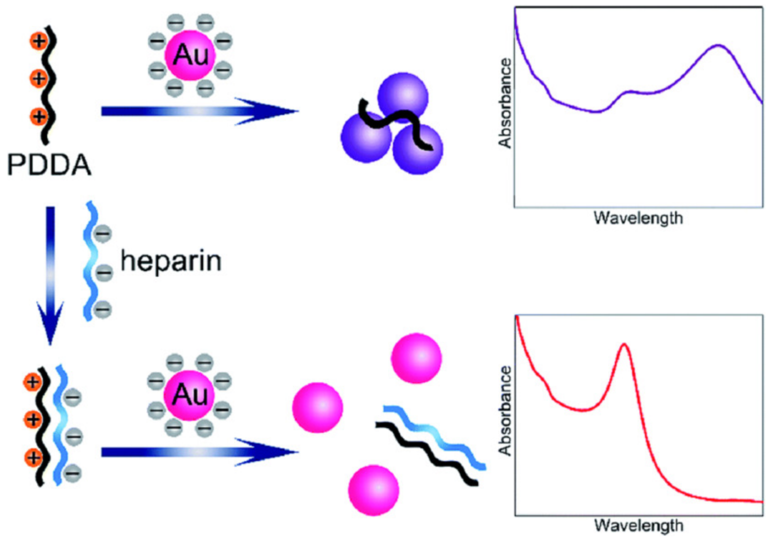

Figure 11. Schematic illustration of the principle for colorimetric detection of heparin by Ma, et al. (2019) [**26**].

4.5. Nanoparticles in cosmetics

Lipid NPs are finding increasing use in cosmetics manufacture. Nanostructured lipid carriers (NLCs) are mostly mixtures of solids and fluids. These lipid NPs are manufactured from lipids, emulsifiers and water / solvents via various methods. Some well-known cosmetics of nanoparticle origin include Cutanova cream nano repair Q10, summer masque crème nano-hydratant and nanolipid restore [**27**].

Silver NPs are used as preservatives in cosmetics, toothpastes and shampoos. This use of silver NPs stem from their anti-bacterial properties. AgNPs inhibit or destroy bacteria by damaging the cell wall of the bacteria. The potency of the NP is a function of its size and mode of preparation. AgNPs are also known to possess healing properties by strengthening the regenerative function of dermis cells. This causes the wounds to heal faster and prevents scar formation [**28**].

4.6. Building and construction

NPs are used in construction and building. These can be seen in CNTs used mechanical durability and crack prevention, silica NPs used to reinforce concrete and copper NPs used to increase corrosion resistance among many others. **Table 1** below lists some major NPs usage in the building and construction industry [**29**].

Table 1. Nanomaterials used in construction materials by Mohajerani, et al. (2019) [**29**].

Area	Nanoparticle	Applications
concrete	silicon	reinforcement in mechanical strength; rapid hydration
concrete	titania	increased degree of hydration; self-cleaning
concrete	CNT	mechanical durability; crack prevention
asphalt concrete; timber	aluminum oxide	increased serviceability
bricks mortar	clay	increased surface roughness
concrete	iron oxide	increased compressive strength; abrasion resistant
steel	copper	weldability; corrosion resistance; formability
asphalt concrete	zycosoil	increased fatigue life; higher compaction

To reduce energy consumption, thermo-responsive windows are manufactured which regulate the incoming solar radiation and / or blackbody radiation output in windows. These windows maximize the use of daylight without reverting to external power sources because they transmit light through glass panels and these panels automatically adapt to a wide temperature range. NPs–polymer composites are greatly employed in the manufacture of electro-responsive windows [**30**]. Metallic NPs, quantum dots (QDs) and some polymer and inorganic NPs are of much importance in the automobile, aeronautics and building industries.

4.7. Military applications

Mobile pigment NPs are used in the manufacture of better camouflage uniforms for the military. NPs are injected into the material of the uniforms for a more efficient camouflage [**31**]. In [**32**], it was reported that adding nanoparticles to cotton and polyester caused changes in reflection in the infrared region of the spectrum. They also deduced that increasing the mineral NPs concentration in printed fabrics limited the color fastness. Nanomaterials are used as absorbers in photodetectors. These detectors are fabricated via their active region being covered by synthesized lead selenium QDs (PbSe QDs). The PbSe particles are dispersed in 2-propanol

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and spun coated with copper contacts. These are later deposited on fibreglass. These are used in infrared cameras for night vision [33]. CNTs are currently being developed as military body armor. CNT is 117-times stronger than steel. CNTs will serve as body armor to soldiers on the battlefield. To enhance durability, alloys are being incorporated into the CNTs [34].

5. Toxicity of nanoparticles

NP toxicity is generally a function of their physical and chemical characteristic. These characteristics include their size, shape, surface area, catalytic activity and active groups on the surface [15]. As NPs are increasingly manufactured, their adverse effect on human and animal health becomes a cause for concern [14]. The ingestion, absorption and inhalation of NPs induce the formation of reactive oxygen species (ROSs) and free radicals. These in turn give rise to different health issues ranging from inflammation, oxidative stress and finally, damage some biological materials like DNA and proteins. NPs can also cause continued toxicity with prolonged accumulation in the body [35]. It has been observed that titanium dioxide (TiO_2) NPs can cause premature hatching of zebrafish embryos. TiO_2 inhibits hatching and facilitates embryo deaths. NP-induced toxicity is also observed in the disruption of gills, skin and endocrine system [36].

6. Nano-sorbents in wastewater and soil remediation evaluations

Removal of toxic heavy metal ions from waste and contaminated water has been a concern to scientists for years. The use of synthetic compounds is of great concern to environmentalists because of the possibility of environmental hazards that can result. The use of traditional sorbents for heavy metal ion removal from waste water and contaminated soil has its disadvantages, mainly the inconvenience of separation in multi-metal ions solutions, poor performance and efficiency [37].

In recent decades, there has been an increasing array of nanostructured materials with the potential of being more effective adsorbents for wastewater treatment and water purification. The generally used adsorbent for contaminated soil remediation is activated carbon black which is costly. There is an ongoing search for better adsorbents and the features to be considered according to [38] include but not limited to:

- high adsorption and removal capabilities;
- stability for both chemical and thermal purposes;
- adequate selectivity;
- regeneration and recyclability (ability to recover used adsorbents);
- adequate tunability of porosity;
- ability to be functionalized;
- structural integrity and high mechanical strength;
- shape recovery;
- ability to self-clean and self-heal;
- amenability for production in bulk via green synthetic route;
- integration in large-scale treatment plants;
  and
- cheap for bulk production.

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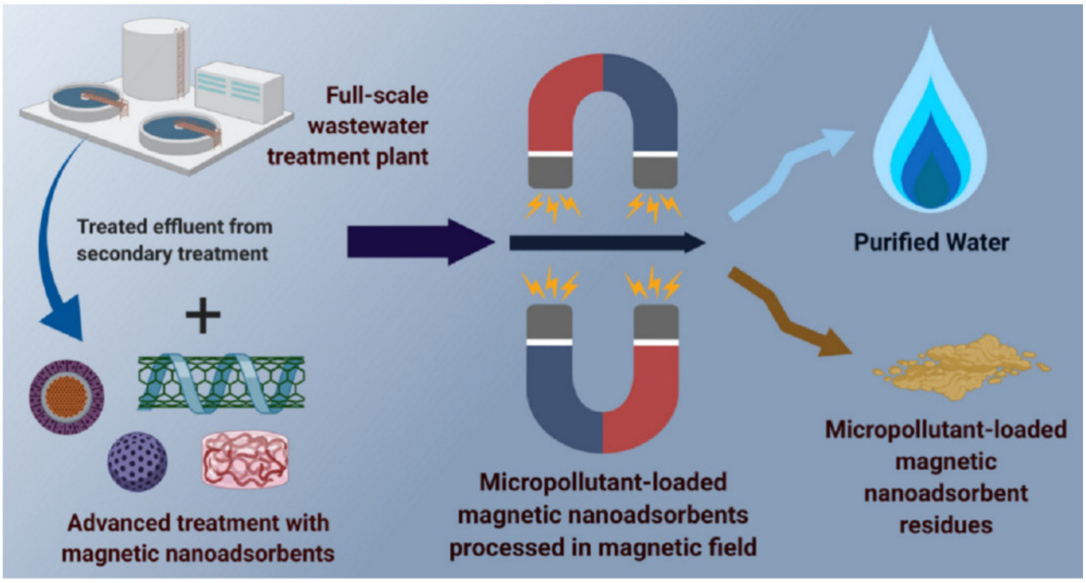

Figure 12. Magnetic separation: water purification / wastewater treatment plant by Mudhoo & Sillanpaa (2021) [**38**].

Figure 12 above depicts the use of magnetic nanoadsorbents in water purification and wastewater treatment via selective adsorption of the target micropollutants. Copper, like other heavy metals, accumulates in the human body hence causing adverse health effects such as high blood pressure, kidney, liver and respiratory problems. The nanoadsorbents used in removing copper ions from aqueous solutions can be classified into seven types (nanofibers, magnetic NPs, nanoscale / metallic oxides, aerogels, carbon-based, polymer-based and clay-based NPs) as depicted in **Figure 13** below [**39**]. Adsorption is usually preferable in heavy metal ion removal due to design simplicity, low cost and high resistance to heavy metals. Contact time, pH and adsorbent dose all play important roles in the amount of heavy metals that can be adsorbed using nanosorbents [**40**].

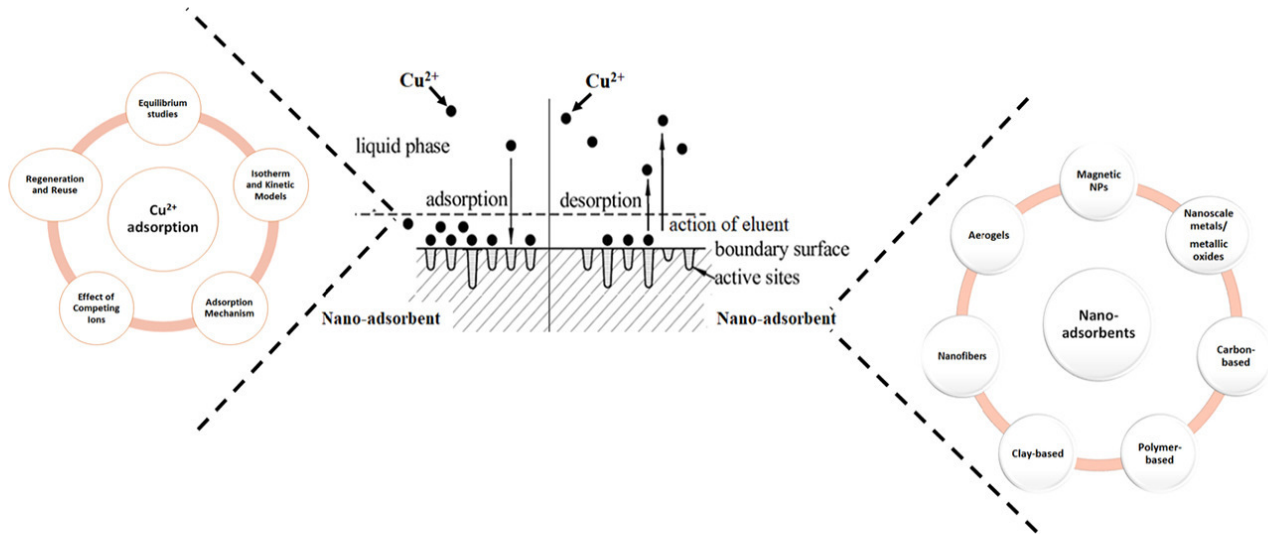

Figure 13. Classification of nano-adsorbents for Cu^{2+} removal by Emenike, et al. (2022) [**39**].

Nano-sorbents need to possess some additional properties for them to be acceptable in removing heavy metals from wastewater and contaminated soil. These are mainly concerned

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with toxicity and economic viability. The nanosorbent chosen has to be cheap, readily available, non-toxic and is not a food source for man or animals [**5**]. In order to remove contaminants from wastewater, Fe–Al binary oxide nanosorbents is synthesized and used. Ferric nitrate nonahydrate $Fe(NO_3)_3{\cdot}9H_2O$ and aluminum nitrate nonahydrate $Al(NO_3)_3{\cdot}9H_2O$ are the precursor materials. The solvent used for dissolution was ethylene glycol and a ratio of 1:3, metal nitrates: ethylene glycol was employed.

The following parameters were also investigated: effects of pH and ionic strength, kinetic studies, effect of adsorbent dosage, effects of agitation speed, contact time, phosphate ion concentration and thermodynamic studies [**41**].

Other adsorbents which have been synthesized with iron oxide incorporating different metal ions include Al(III), Cr(III), Cu(II), Mn(IV), Ti(IV) and Zr(IV). These bimetallic / trimetallic oxide adsorbents result in increased surface area and surface charge which in turn increase the metal fixation and hence lead to a better metal ion removal.

Some other ideal nanosorbents for use in wastewater purification are magnetic nanoparticles. These are good for arsenic species owing to their affinity to arsenate and arsenide. In [**42**], it was started with 100 μg/L As(V) with 0.5 g/L magnetite NPs in a sealed vessel. The contaminated groundwater contained 39 μg/L total arsenic. The adsorption experiment showed that in 1 h, 83 μg/L of arsenate was adsorbed from pipe-borne water. It was also observed that the amount of arsenic in contaminated groundwater solution was 10 μg/L in about 10 min and further reduced to 5 μg/L in about 1 h. These results were in line with the Langmuir isotherm model. It also showed that magnetic NPs successfully removed arsenic ions from wastewater and contaminated soil.

7. Challenges

Challenges usually encountered with NPs stem from toxicity of reducing agents in non-green synthesis methods. Ease of access to analysis equipment like SEM, XRD, TEM also poses challenges especially for developing countries. In nanomedicine, research on long term effects of NPs usage should be encouraged. Use of NPs in other areas of medicine aside cancer treatment should also be given more attention.

8. Conclusion

Nanoparticles synthesis, characterization, classification, applications and toxicity have been investigated. Ability of nanosorbents to remove metallic ions from wastewater and contaminated soil was reviewed. Synthesis of binary oxide nanosorbents was explored.

Several physicochemical properties of nanosorbents make them highly attractive as candidates for wastewater purification and contaminated soil remediation. Challenges associated with nanosorbents stem mostly from the scarcity of the plant material. The plant material to be used need not be a food source for man or animals. Toxicity to human cells also poses problems of which nanosorbents or which synthetic pathway to employ. Nanoparticles entering the human body via ingestion, absorption or inhalation has been reported to be the cause of the formation of reactive oxygen species and free radicals which are harmful.

Further research should be focused on economic viability and further reducing toxicity of nanosorbents.

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