Advances in Nanoparticles, 2016, 5, 27-43
Published Online February 2016 in SciRes. http://www.scirp.org/journal/anp
http://dx.doi.org/10.4236/anp.2016.51004

A Review on Metal Nanostructures: Preparation Methods and Their Potential Applications

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Received 4 November 2015; accepted 3 February 2016; published 6 February 2016

Abstract

Metal nanostructures have been of great research interest in recent years due to their physicochemical, plasmonic properties and potential applications. A lot of work has been done on the controlled synthesis of metal nanostructures for various applications. In this review, we try to focus on recent developments in synthesis and applications of metal nanostructures. Firstly, we summarized different preparation methods and then briefly explained their potential applications.

Keywords

Surface Plasmon Resonance (SPR), Metal Nanostructures, Plasmonic Applications

1. Introduction

In recent years, the increasing number of published papers shows great interest of engineers and scientists for the importance of metal nanoparticles (NPs) such as silver (Ag), gold (Au) and copper (Cu) etc., because of their physicochemical properties, nano meter (nm) size and surface plasmon behavior [1] [2]. These metal NPs are used in catalysis, electronics for circuit development and as self-assembled new nanostructure materials, but only few show different sets of applications with light interaction. The interaction of light in a field with metal nanostructures is called as plasmonics [3]-[7]. The plasmonic nanostructure materials are defined by means of their strong interaction with incident light and free electrons, where metal nanostructures act as a source to convert light into localized electric field (electromagnetic excitations coupled with collective oscillations of free elec-

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How to cite this paper: Qazi, U.Y. and Javaid, R. (2016) A Review on Metal Nanostructures: Preparation Methods and Their Potential Applications. *Advances in Nanoparticles*, **5**, 27-43. http://dx.doi.org/10.4236/anp.2016.51004

trons) in metals [8], and called as localized surface plasmon. Incident light can work more effectively with strictly controlled size and shape of metal nanostructures [4] [9]. Nanoplasmonic research is successfully gaining interest due to its various worth seeing applications, such as super lenses, surface enhanced Raman spectroscopy (SERS), single molecule spectroscopy [10]-[14], plasmon-enhanced fluorescence, nanoscale lasing, enhancement of non-linear optical signals, quantum computing, plasmon assisted photo lithography, photocatalysis, light harvesting, biochemical sensing and possibly conversion of solar to chemical energy with plasmonic metal nanostructures [15]-[25]. In last few decades, Au and Ag nanostructures have been prepared chemically on a large scale for plasmonic applications. Ag is 50 times cheaper than Au and became a good candidate in plasmonics due to its unique and convenient physicochemical properties for the next generation plasmonic technologies [26]-[28]. It is to be noted that plasmonics restrict to a coupling between electromagnetic excitation and metal nanostructure for the generation of surface plasmon and do not show plasmonic functions in the absence of incident light. Nanoplasmonic is quite unique as different sizes of nanostructures from 10 to 100 s of nanometers can be used which act as a link between nano and micrometer levels. Thus, plasmonics is a new subfield of nanotechnology which has ambition to understand the control of light with metal nanostructure in innovative approach.

The self-assembly of metal nanoparticles into small clusters, provides suitability to control and tune their optical properties by coupling surface plasmon of nanoparticles and enhance the electric field in between the particles gaps. These gaps in self-assembled nanostructures with high intensity field are known as hot spots. The enhancement of electric field is very important in terms of optical and chemical properties of nanoparticles as plasmonic properties are strongly influenced by electric field. We can control the self-assembly of nanostructures by varying the number of nanoparticles in a cluster or the size and shape of individual nanoparticles for the achievement of our required plasmonic applications. The number of scientific research publications on plasmonic nanostructures is rapidly increasing and expected to be continuing in the near future [29].

2. Preparation of Metal Nanostructures

Various methods are adopted for the preparation of metal nanostructures that should be ideally controllable regarding shape and size, cheap, environment friendly, and high product yield with less waste products. **Figure 1** summarizes various preparation methods of metal nanostructures.

Metal nanostructures show high abundance of plasmon excitation for example scientists are showing much interest in Ag nanostructures due to plasmon resonance in the range of visible spectrum and used in various potential technologies. Here, we will discuss all synthetic processes in reference mostly of Ag, as an example.

2.1. Chemical and Photochemical Synthesis

The optical properties of metal nanostructures are highly dependent on their size and shape. The controlled morphology of nanostructures is an important factor which has strong impacts on their unique plasmonic properties. Until now, various methods are adopted for the preparation of controlled size and shape of nanostructures

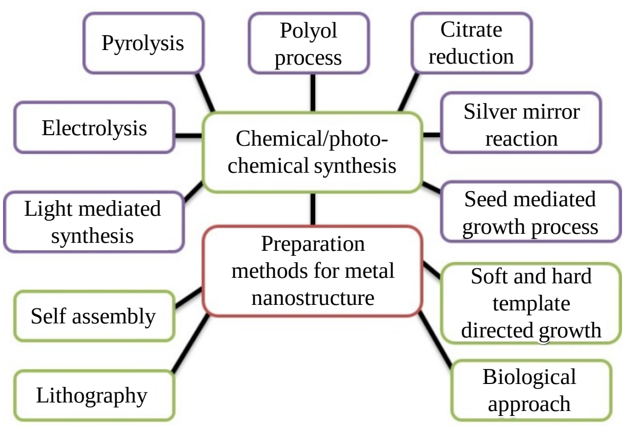

Figure 1. Various methods for synthesis of metal nanostructures.

[30]-[32]. In general, metal/Ag^+ salt precursor is mixed with reducing agent in the presence of stabilizing agent and control the size and shape of metal nanostructures. Past research publications show that silver nitrate, as a precursor, has been abundantly used due to its cheap price and easy availability. Various reducing agents such as sodium citrate, sodium borohydride and alcohols are widely used to reduce metal/Ag^+ ions present in solution into metal/Ag atoms which combine to make aggregates and finally become nanostructures [33]-[35]. On the other hand, stabilizing agents are introduced to control and stabilize the required morphology of metal nanostructures. While, in photochemical synthesis, a variety of light irradiation methods are adopted to synthesize metal nanostructures. Following is the detail of chemical/photochemical synthetic processes.

2.1.1. Light Mediated Synthesis

In this technique, light irradiation is applied to produce nano structures. For example, laser ablation or direct laser irradiation on an aqueous solution of metal salt in the presence of surfactant to fabricate specific shape and size distribution of metal nanoparticles where source of light works as a reducing agent [36] [37]. Laser light is also used to modify metal nanoparticles by simple melting such as Ag nanospheres into Ag nanoplates [38]-[43] it is known as tailoring process with light. Laser light mediated synthesis is continuing its success for generating highly desirable and well controlled metal nanostructures.

2.1.2. Electrolysis and Pyrolysis

There are few scientific reports available, describing use of electrochemical approach for the synthesis of metal nanoparticles. For example spherical shaped Ag nanoparticles were synthesized with an average size of 10 nm by reducing Ag^+ ions in the presence of poly vinyl pyrrolidone (PVP) using electrochemical method, where titanium (Ti) electrode worked as a cathode and platinum (Pt) plate with 2 cm diameter worked as an anode. Ag nanoparticles are also synthesized by adopting another method of spray pyrolysis in which an average grain size of 100 nm of Ag nanopowder was synthesized. Among other chemical synthesis, electrolysis and pyrolysis are considered environmental friendly processes as no harmful or toxic reducing agents are used to produce nanostructures [44] [45].

2.1.3. Citrate Reduction

One of the popular methods to synthesize Ag colloidal solutions is citrate reduction of Ag^+ ions as we do not need comprehensive laboratory training skills [46]-[48]. First time, Lee and Meisel introduced this method in 1982 [49]. Generally, Ag nanoparticles are formed, when a calculated amount of sodium citrate is added in an aqueous solution of silver nitrate at boiling and wait for at least 1 h. This is a very simple method but did not produce confined size of nanoparticles. The synthesized product shows variety of sizes in the range of 20 - 600 nm. However, pH control is an important factor, as few shape controlled methods had been discussed by previous researchers by controlling pH values such as at pH = 5.7 triangular shape and pH = 11.1 spherical as well as rod like particles were formed [50] [51].

2.1.4. Polyol Synthesis

One of the well-known and commonly used methods to synthesize a wide range of size and shape of Ag nanostructures [52]-[56]. In general, Ag salt precursor and capping agents are introduced into polyols for the generation of nucleation and growth towards Ag nanostructure. Propylene glycol, 1,2-propylene glycol or 1,5-pentanediol are commonly used reducing agents which reduce Ag^+ ions present in the solution [57]. Reaction parameters such as temperature and concentrations are very important to overcome the possible control on the final reaction product.

2.1.5. Seed Mediated Synthesis

In recent years, another approach to synthesize metal nanoparticles gaining much attention is seed mediated synthesis in which nanocrystals work as seeds for further growth. This method has advantages on the control over the end product morphology [58]-[61]. For example Xia *et al.*, in 2010, synthesized, Ag nanocubes by using spherical or cubic shaped single crystal seed for specific edge length from 30 - 200 nm.

2.1.6. Silver Mirror Reaction

Justus von Liebig discovered silver mirror reaction in 1835 [62] which became much popular for depositing Ag metal on targets or solid surfaces. In this reaction, Tollen's reagent [$Ag(NH_3)_2OH$] is reduced by sugar or any aldehyde containing compound for generating Ag (Equation (1)):

$$RCHO + 2\left[Ag\left(NH_3\right)_2\right]^+ + 2OH^- \rightarrow RCOOH + 2Ag + 4NH_3 + H_2O \quad (1)$$

Successful reaction shows shiny layer formation on the inner side of the reaction container due to the silver deposition. Silver mirror reaction is mostly employed for the detecting aldehyde group in schools [63].

2.2. Soft and Hard Template Mediated Synthesis

Template based synthesis has been used to prepare specific shape and size of nanostructures which highly depend upon the selected template [64] [65]. There are two types of template; soft and hard templates. Commonly, surfactant molecules work as a soft templates and form at critical micelle concentration (CMC). Porous anodic aluminum oxide (AAO) membrane could be a good example of hard templates. These micelle and reverse micelles structures present in the solutions are responsible for the shape and size of final product. Previously published research work shows that soft template assisted methods have been involved in preparation of metal nanostructures of various shapes and sizes such as Ag nanowires [66]-[68], nanorods [69]-[71], hollow spheres, nanoplates [72]-[76] etc. Based on properties, surfactants can be classified as ionic surfactants {e.g., cetyltrimethylammonium bromide (CTAB) [77]-[82], octadecyltrimethylammonium chloride (OTAC), di sodium (2-ethylhexyl) sulfosuccinate (Na (AOT)) [83]-[85] and sodium dodecylsulfate (SDS) [86]}, nonionic surfactants {e.g., oleic acid (OA) [87] [88], oleylamine (OAM) [89] [90], trioctylphosphine (TOP) [91] and trioctylphosphine oxide (TOPO)} and polymer surfactants {e.g., poly(vinyl pyrrolidone) (PVP) [92]-[99], poly(vinyl alcohol) (PVA) [100] and poly(ethylene oxide) [101]}. Template assisted synthesis has some advantages such as final product is in well dispersed form due to reduction in the formation of aggregates [102]-[104], reaction conditions are mild as well as benign route in respect of reactants and solvents. However, still there is need to improve the parameters to achieve the desired shape and size of nanostructures which is a challenge for researchers.

2.3. Lithographic Approach

Lithography is concerning with the study and application for the fabrication of nanometer structure with at least one dimension from 1 to 100 nm scale. Unlike the liquid based process, lithography, process of deposition and other nano-fabrication techniques provide controllable installation ability for metal nanostructures [105]. Many kinds of lithographic techniques have been presented by researchers such as optical lithography (OL), electron beam lithography (EBL) [106], scanning probe lithography (SPL), multiphoton lithography (MPL) etc. and applied as per requirements for example EBL technique is used to fabricate mask by depositing Ag nanostructure on a substrate [107].

2.4. Self-Assembly of Metal Nanoparticles

Self-assembly of metal nanoparticles can be distinguished on the basis of shapes of individual nanoparticles, methods used for self-assembly and the nature of forces involved for self-assembly of nanoparticles. Nanoparticles are the basic building blocks for assisted self-assembly process [108]-[110]. This process can generate the formation of large sized final product such as nanosheets at microscale in comparison with other methods [111]. One another fast strategical approach for self-assembly of stable metal nanoparticles is DNA linkage [112]-[115]. Self-assembly assisted by DNA has been adopted recently to develop systematically arranged plasmonic nanoparticles into chain, triangular shape, 3D lattices and Janus nanoclusters [116]-[121]. For example, fabrication of free-standing monolayered Au nanoparticle super lattice sheet formation with DNA linkage. Direct interaction of nanoparticles in solutions such as major driving forces, attractive and repulsive forces impact on the whole self-assembly process which can be discussed as hydrophobic, electrostatic, hydrogen bonding and biospecific interaction of self-assembly of metal nanoparticles.

2.5. Hydrothermal Process

Hydrothermal process is simple and has been widely used for the synthesis of different kinds of nanostructures [122]-[124]. Usually, high temperature is required to proceed it. For example, Yan *et al.*, synthesized triangular shaped Ru nanoplates with thickness ~3 nm using $RuCl_3 \cdot H_2O$ and HCHO (40 wt%) in the presence of PVP at

160˚C for 4 h. It was also noticed that only change in the concentration of Ru salt and PVP, irregular shape formed with thickness ~1.5 nm. On the other hand, when Ru salt was replaced with silver nitrate, triangular shaped Ag nanoplates were formed with sharp and curved corners in the presence of PVP at 160˚C for 6 h.

2.6. Biological Approach

Recently, it has been noticed that living microorganisms such as bacteria, fungi and plants have great potential for the synthesis of metal nanoparticles such as cadmium sulfide (CdS) [125], Ti/Ni, titanate [126], zirconia [127], Au [128] [129] and Ag [130] [131]. The use of microorganisms is environmental friendly and benign synthesis route for which provides good control over size distribution of nanostructures. For example, Ag nanostructures were synthesized using bacteria with size less than 200 nm.

3. Potential Applications

3.1. Photocatalysis

Novel functional nanomaterials have gained much attention due to their unique catalytic, optical, electronic, magnetic, and mechanical properties. In present years, whole world is focused to overcome near future problems such as environmental pollution, energy conversion, and the production of organic compound which compelled researchers to develop novel catalysts, that can catalyze desirable chemical transformations, using light. Plasmonic metal nanostructures with controlled morphology, shape, and pore structure have a higher surface area, which leads to the increased catalytic activity. So, there is an ever-increasing interest to design novel catalysts and establish a structure-activity relationship for the understanding of reaction mechanism. This allows researchers to develop a wide range of suitable catalysts for energy, environment remediation, and organic synthesis applications. Until now, a lot of work has been done in searching cost effective, sustainable and green energy sources to meet global energy requirements for current environmental contamination and the growing threat of present energy crisis. An environmental friendly renewable energy resource such as solar energy to remove organic pollutants in the presence of highly effective photocatalysts has been regarded as the ultimate solution for green chemical processes and hydrogen production [132].

On the bases of photocatalysis science, there are two types of photocatalytic reactions *i.e*. homogeneous photocatalysis and heterogeneous photocatalysis. The most prominent features of the photocatalytic system are the required band gap, suitable morphology, high surface area, stability and reusability [133]-[136]. A number of metal oxides are known as photocatalysts such as oxides of vanadium, chromium, titanium, zinc, tin, and cerium. During the reaction process, a metal oxide is activated with UV light, visible light or a combination of both that depend upon its light absorption range and as a result, photo excited electrons are moved from the valence band (VB) to conduction band (CB) and form an electron/hole pair (e−/h+). This photo-generated pair (e−/h+) is able to reduce and/or oxidize a compound adsorbed on the surface of photocatalyst. More recently, it is investigated that defected metal oxide could perform well in visible light by using band gap engineering to create small band gap as shown in **Figure 2**. The favorable combination of electronic structure, light absorption properties, charge transport characteristics and excited lifetimes of metal oxides has made it possible for their application as

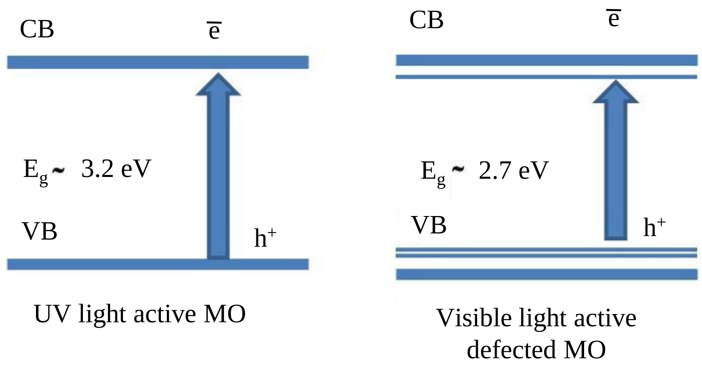

Figure 2. Photo excitation of UV light active and visible light active metal oxides (MO).

photocatalyst [133]-[137]. However, it is particularly difficult to find enough stable semiconductor system with a suitable band gap for visible absorption, required for driving subsequent redox reactions. Metal oxides such as (TiO_2, ZnO, SnO_2 and CeO_2) have been widely investigated for photocatalytic applications [138]; however, the relatively wide band gap limits their photocatalytic applications because UV light only accounts for 4% of the total solar spectrum [139].

Until now, varieties of photocatalyst for water splitting have been reported but most of these are only active under UV light irradiation and only a few have been demonstrated to operate under visible light up to a wavelength of 500 nm [140]. Recently, Chengsi Pan *et al.* developed photocatalyst that is working over a wider range of visible light spectrum approximately 600 nm [141]. On the other hand, surface modification along with combination of suitable co-catalysts for enhancing photocatalytic reactions [142], self-assembly and growth mechanism are equally important factors as well as designing new strategies for the development of novel nanomaterials. Thus, scalable, cost-effective routes to synthesize novel nanostructured, visible-light responsive catalyst with narrow bandgap and a larger wavelength range are desired which accounts for 43% of the spectrum of sunlight.

3.2. Surface Enhanced Raman Scattering (SERS)

SERS is a surface sensitive powerful technique used to detect various molecules because of its high sensitivity and selectivity [143]-[150]. Metal plasmonic nanostructures such as Ag and Au have been known for more effective SERS substrates that can increase the Raman Scattering of the molecules [151]-[154]. Plasmonic nanostructures with sharp corners are familiar for keeping SERS activities that can increase electromagnetic field (E-field) and create "hot spots" [155] [156]. For example, Xia *et al.* described recently the importance of sharpness of metal nanostructures in SERS such as the signal intensity of 4-mercapto-pyridine for triangular shape and hexagonal Pd nanoplate was 4.3 and 3.4 times that of Pd cuboctahedra [157]. By controlling the sharp corners of the nanostructures, SERS signals can be increased; however, the stability of the sharpness is an issue. To overcome this problem, bimetallic nanostructures such as Pd-Ag core-shell were synthesized for SERS applications [158] that showed relatively stable signals.

More recently, research has been carried out to examine the hot spot (area with high E-field enhancement) in single particle or dimers to determine the contribution of hot spot SERS signal enhancement factor [159]-[166]. On the other hand, theoretically, the maximum amplitude of the normalized E-field of Ag dimer with 2 nm gap distance is nearly 25 and 200 times that of Ag nanoplate and spherical particle, respectively. Moreover, it was observed that SERS signals on metal self-assembled nanostructures (Ag) could be regulated by controlling the structural orientation of metals.

3.3. Localized Surface Plasmon Resonance (LSPR) Detection/Sensing

Detection and sensing of molecules is one of the best applications of plasmonic structures and was commercially launched as propagation surface plasmons (PSPs) on metal films. These metal films are chemically used for selected binding of target molecules such as DNA or proteins. After binding, the dielectric charge near the surface of metal films is generated. Such bindings could be monitored between the metal film and exciting source for generation of PSPs. Still, on behalf of this technique, a large number of instruments are available in the market and used for biological science [167]. PSPs technique has been extended to LSPR for detecting and sensing metal nanoparticles. When the dielectric environment change by introducing the detecting molecules, the resonance frequency of LSPR shifts and that is the main idea for sensing molecules [168]-[171]. In more recent advancements, much focus is to increase the LSPR sensitivity of metal nanostructures. For example, when the spectral overlapping between LSPR and the molecular electronic resonance takes place, a strong resonance coupling can be observed [172]. It is also observed that the morphological changes in the nanostructures are very important factor which strongly correspond with LSPR. Recently, the development of colorimetric indicators depending on time and temperature are praise able [173].

3.4. Photo-Thermal Therapy

Metal nanostructures, could be a dominating nominee for photo-thermal therapy due to strong LSPR absorption especially in the Near Infra-red (NIR) region, photo-thermal stability and biocompatibility factors [174]-[180].

For example, palladium (Pd) nanosheets in an aqueous solution were illuminated by NIR laser light and monitored for the NIR photothermal efficiencies at various temperatures. It was observed that the temperature increased from 28˚C to 48.7˚C after 10 min. NIR laser irradiation, when Pd nanosheets were present into the solution. In contrast, the temperature of 1 ml aqueous solution without Pd nanosheets raised only 0.5˚C by NIR laser irradiation. This technique was applied to the liver cancer cells and the results were remarkable as nearly 100% cancer cells were killed just in 5 min. laser irradiation. It was also observed that the photo-thermal stability of Pd nanosheets was good as compared to Au and Ag when irradiated with NIR laser for 30 min. For further increase in photo-thermal stability of Pd nanosheets, a coating with Ag thin layer was preferable. Other than Pd nanosheets, it has been reported that Au nanoprism showed excellent photo-thermal stability under longer NIR laser (wavelength 1064 nm) irradiation time which is a good point as Infra-red (IR) light could have high tissue penetration and lower optical absorption by blood and soft tissues [176]-[181].

3.5. Surface Enhanced Fluorescence (SEF)

Fluorescence technique is used in many applications such as microscopy imaging, optical devices and medical diagnosis. Scientists show great interest to improve the fluorescence sensitivity of fluorophore substrates; however, it becomes more interesting when limited to a single molecule due to the requirement of many potential applications. For this purpose, SEF technique is used. It is based upon the emission of resonant molecules in the locality of emitters [182]-[184]. SEF is highly dependent on near field coupling between excited states fluorophores and surface modes. Especially, surfaces of plasmonic nanostructure with localized surface plasmons are efficient SEF substrates. Likewise in SERS, molecular distance from plasmonic nanostructures affects the SEF experiments because of the interaction of molecules and nanoparticles [185] [186].

3.6. Surface Based Electronic Devices

The main theme on which molecular sensors work is the molecular absorption. Molecular absorption has impact on the emission peak, position and intensity of plasmon absorption. Optical integration and electronic circuiting could be helpful in making high speed, small size computer microprocessors [187] [188]. On the other hand, surface plasmons are important as acknowledged for transmitting information on computer chips. Plasmonic nanostructures are helpful in supporting high frequencies (100 THz) as compared to simple conventional wires (10 GHz). The ability of plasmonic nanostructures to work on high frequency distinguishes them for developing useful electronics such as plasmonstors [189]. In general, lithographic techniques are used in developing electronic devices based on plasmonic circuits [190]-[193]. Lithographic techniques are also helpful to overcome the problems in terms of location, geometry and orientation. Surface plasmons are material sensitive for propagation and supportive for measuring thickness of monolayers on colloid films. Many companies like Biacore have made instruments based on this principle. A team of Korean scientists in 2009 discovered a method for improving efficiency of organic light emitting diodes [194].

3.7. Other Applications

As discussed before, metal plasmonic nanostructures are promising materials for potential applications. Many new applications are rising in wide potential areas [195]-[199]. New advanced applications could be in the field of lasers, super lenses, active plasmonics, photovoltaics [200]-[211], phase changing materials [212] and polymers [213], etc. **Figure 3** describes a flow sheet diagram of various potential applications of metal nanostructures.

4. Conclusion

The concept of developing and organizing metal nanoparticles into plasmonic nanostructures is awesome which supports to establish a new way of knowledge towards metal nanostructure interactions in solution, synthesis of new materials with various plasmonic properties and functional nanomaterials. In recent years, research work on plasmonic metal nanostructures shows great interest of scientists and provides a strong hold over the control of light. Herein, we explained various preparation methods and potential applications of metal nanostructures such as photocatalysis, SERS, photothermal therapy, LSPR based sensing and detection, plasmonic based electronic devices and SEF technique. However, there is still room for improvements and big challenges that need to be

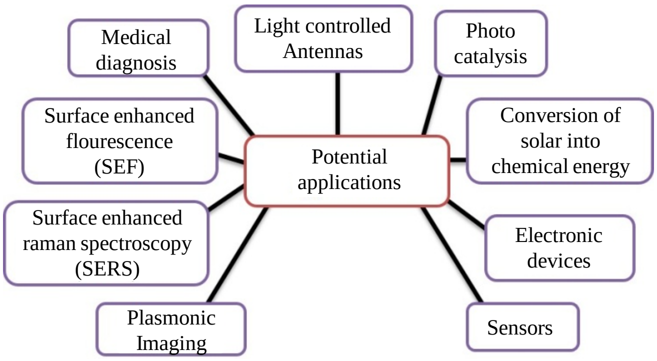

Figure 3. Flow diagram of various potential applications of metal nanostructures.

discussed in consideration of new important applications. The fast growing research developments in the field of plasmonics will open a new era for generating small and ultrafast devices.

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