

Spherical nanosilver: Bio-inspired green synthesis, characterizations, and catalytic applications

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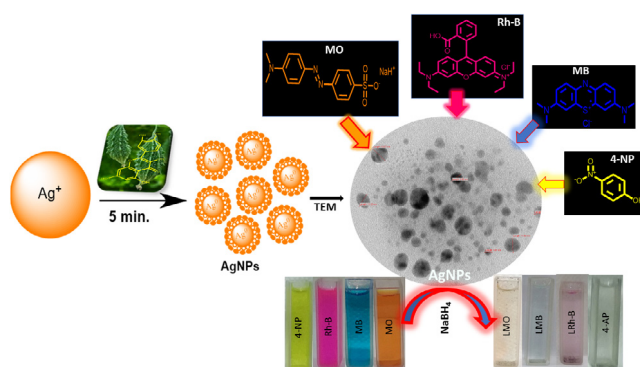
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HIGHLIGHTS

- Rapid (~5 min) green synthesis of crystalline and spherical AgNPs with mechanism.
- Extensively characterized via UV-Vis, PL, FEG-SEM, EDX, TEM, AFM, FTIR, XRD and XPS.
- Enhanced photocatalytic activity (less t, high K_{app}) with least amount of catalyst (2.0 mg) had been reported for the first time.
- FT-IR confirmed reduced and fragmented products after degradation and recovered AgNPs was stable (ensured by PXRD) and reusable.
- The photodegradation pattern followed pseudo first-order reaction kinetics.

GRAPHICAL ABSTRACT



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ABSTRACT

Bio-inspired green synthesis of noble metal nanocatalyst for visible light induced reductive degradation of organic pollutants is a promising strategy for water purification. This study reports a quick synthesis of spherical and stable *M. charantia* fruit extract supported silver nanoparticle (AgNPs) as a green catalyst and first time applied for the degradation of various industrial dyes pollutant. The bioinspired green protocol was followed for synthesis and the formulation mechanism of AgNPs was elucidated. Flavonoid worked as reducing agent and protein worked as a stabilizer during catalyst fabrication. The catalyst was characterized using TEM (spherical nanostructure with size <16 nm), FEG-SEM (spherical), EDX (Ag at 3 Kev), AFM (average roughness 4.9 nm, skewness -0.0164), XPS, XRD, UV-Vis. (SPR at 432 nm) and FTIR techniques to investigate size, morphology, elemental composition, chemical state, crystallinity, plasmonic behavior and involved functional groups. FTIR ascertained the reducing and stabilizing nature of phytocomponents of fruit extract in AgNPs synthesis.

AgNPs exhibited enhanced photocatalytic activity during degradation in presence of NaBH_4 at room temperature under visible-light irradiation. The degradation time and apparent rate constant (from pseudo first order) for the AgNPs@MB, AgNPs@Rh-B, AgNPs@MO and AgNPs@4-NP system were $t = 8$ min ($K = 0.277 \text{ min}^{-1}$), $t = 13$ min ($K = 0.162 \text{ min}^{-1}$), $t = 7.5$ min ($K = 0.269 \text{ min}^{-1}$) and $t = 7$ min ($K = 0.262 \text{ min}^{-1}$), respectively. A discrepancy in degradation capacity among dye can be ascribed to relatively large molecular size and formation of the surface oxide layer. Results revealed that 91% of MB was degraded by 2.0 mg catalyst with rate constant 0.277 min^{-1} . Degraded products were evidenced by FT-IR and degradation pattern followed pseudo-first order kinetics. The blend of green synthesis, intrinsic

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biocompatibility, superior photocatalytic activity, stability and recyclability of AgNPs are potentially applicable for industrial wastewater treatment and environmental remediation.

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1. Introduction

Nanobiotechnology is an emerging field dedicated to formulate and utilize green derived metallic, biocompatible and smart nanostructures applicable to drug delivery, diagnosis, imaging, sensing and catalyst [1]. For the economic growth, industrial prosperity, environmental remediation and public health, nanocatalyst driven water contaminates monitoring is crucial. Development of nanoscale (≤ 100 nm) superstructure have flourished in recent years owing to their localized surface plasmon resonance (LSPR), unique quantum confinement, fast dissolution, high surface to volume ratio, high reactivity and ease of surface functionalization [2–6]. Moreover, its excellent adsorbent tendency due to high surface area with more active exposed co-ordination sites affords catalytic properties for oxidization reaction [7]. As compared to bulk, in nanosize regime metallic nanoparticles exhibits remarkable change in their electrical and optical properties. Light interaction with noble metal nanostructure provides many fascinating optical phenomena. In addition, due to nanosize, a significant change in reduction potential is also observed in comparison to bulk as high Fermi potential of metal nanoparticles leads to more negative (lowering) reduction potential values. This property allows them to work as catalyst for many electron transfer reaction [8]. Advanced fabrication of efficient photocatalysts and its surface alternation behavior is promising for solving many technological, energy and environmental challenges and applicable for catalytic elimination of environmental pollution [9,10]. In the existing forefront of scientific research, due to ease of separation, heterogeneous photocatalyst has demonstrated their incredible capability in countless chemical transformations in industries, energy science and wastewater treatment [11,12]. Especially for water purification, heterogeneous photocatalysts are intensively investigated for their promising feasibility including (i) Low operation cost (ii) works at ambient temperature (iii) complete mineralization of organic pollutant. Development of nano-sized catalyst assists in photo-oxidation process, which is a crucial step during wastewater treatment.

In the current scenario, water pollution is a critical and alarming problem across the globe due to the uncontrolled discharge of dyes effluents from textiles, leather, paint, food, cosmetics, ceramics, paper, pulp, beverages and plastic industries into water bodies [13]. Meanwhile, increasing global populations further enhanced the consumption of pure water. Subsequently, there is an inversely proportional relation between clean water shortage and increasing wastewater. Ultimately, it may happen that water is everywhere but not a drop to drink. Current data indicates that various industries are releasing dyes with an approximate production rate over 7×10^5 tons every year due to commercial activities [14]. Various kinds of carcinogenic, hazardous, mutagenic, severe colored and chemically stable dyes are the serious threat to the ecosystem and environment [15]. For example; 4-nitrophenol (4-NP) exposure has a severe toxic effect on methemoglobin formation, which causes kidney and liver damage. Its acute exposure causes a headache, cyanosis, nausea, and drowsiness, while its reduced product 4-aminophenol (4-AP) is the component of biologically active and important intermediate of many analgesics and antipyretic drugs such as paracetamol and phenacetin. Cationic textile dye methylene blue (MB) exposure causes diarrhea, abdominal pain, nausea, vomiting, skin irritation, headache, fever, dizziness etc. in the human being. Acute exposure to anionic methyl orange

(MO) could cause vomiting, cyanosis, increased heart rate, shock, jaundice, and tissue necrosis in humans. Cationic dye rhodamine B (Rh-B) is also carcinogenic causes hypothalamic cell apoptosis, which leads to hormonal imbalances. It has also been reported to cause skin, eyes and respiratory tract irritations. The concentration of dye compound from 10–200 mg/l in water causes significant water pollution worldwide [16], which is a global worry and threat towards survival of human due to their toxicity and non-biodegradability.

To fulfill the undeniable need of pure water, various treatment methods, such as sedimentation, adsorption, chemical coagulation, filtration, flocculation, ozonation, chlorination, ion-exchange, reverse osmosis (RO), ultrafiltration (UF), microfiltration (MF), electrolysis, are commonly used. Some of which just transfer the phase of a pollutant from liquid to solid and other are operationally intensive, depends on large set-up, which necessitates high costs, high energy requirement, engineering expertise, hence, it lacks practical utility and requires further treatment [17]. To meet these technological need, Advanced oxidation process (AOP) is a promising strategy, characterized by the *in-situ* generation of non-selective and highly reactive strong oxidant $\bullet\text{OH}$ onto catalyst surface. Reactive oxygen species (ROS) are active green oxidants, which promotes mineralization of persistent and non-biodegradable organic pollutants to CO_2 and H_2O . In this innovative age of technology transfer, for industrial application, energy efficient photocatalyst are exceedingly demanded to efficiently utilize visible light that comprises $\sim 43\%$ of total sunlight [18]. In order to reduce the pollutants, nature-driven non-toxic green catalysts are replacing the chemical agents. Hence, it is important to develop visible light responsive green photocatalyst.

Currently, green synthesis of metallic, plasmonic and heterogeneous nanocatalyst has garnered particular attention because it is an inexpensive, sustainable and cost-effective alternative for wastewater treatment [19]. Plasmonic material displays unique interaction between light and conduction electrons, which leads to collective excitation of conduction electron, a surface plasmon. It involves the coupling of photons and electrons forming a hybrid between excited electronic level and light wave, produces enhanced optical near-field at metallic nanostructures interface, known as surface plasmon resonance (SPR). SPR depends upon the excitation, electron–nucleus interaction, material composition as well as shape and size of the nanostructure. Among plasmonic nanostructure, exploring silver nanoparticles (AgNPs) as catalyst is most vital and fascinating, which displays an array of unprecedented advanced intriguing physiochemical and optoelectronic properties based on its outstanding biocompatibility, excellent stability, unique surface plasmon resonance, high surface area, working capability as a redox catalyst and ability to relay the electrons between donor and acceptor. Particularly, due to strong SPR, AgNPs exhibits high absorption coefficient in the visible spectral range [20]. Beside this, AgNPs are known for their potential antimicrobial and antibacterial effects against bacteria, fungi, virus and hence it is suitable for disinfection of water. While experimenting with Ag catalyzed reduction reaction, smaller AgNPs exhibits better activity owing to its higher surface to volume ratio and more negative redox potential, which facilitates the electron transfer from AgNPs surface to reactants. The green approach of synthesis efficiently provides size and morphology-controlled silver nanocatalyst owing to its phytocomponents which act as reducing, capping and stabilizing agents and remarkably affords

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