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Influence of Nanosilver Synthesis Conditions on its Architecture

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Abstract: *Silver nitrate reduction in presence of Ethylene glycol (EG) and Poly Ethylene glycol (PEG) represents one from the simplest techniques for silver nano-powder fabrication. Where EG act as reducing agent and PEG act as stabilizing agent. The impact of presence either the reducing agent or the stabilizing agent at the reaction media on the properties of synthesized silver were recorded. Regarding to the chain length of PEG was discovered to play the key role in the formation of nano-silver. Accordingly, the variation in the stabilizing agent (PEG) molecular weight on the morphological structures of produced silver was recorded. The formation of nano-silver was confirmed using UV–Vis spectroscopy. The UV– visible scanning results for the aqueous reaction medium containing silver ion has been demonstrated characteristics peak at 420 nm that corresponding to the plasmon absorbance of silver nanoparticle. X-ray diffraction (XRD) was utilized to determine the crystalline structure of the prepared silver nano-powders.The crystalline structure of prepared nano-silver was produce in hexagonal, cubic crystal and face centered cubic configurations with different plane of orientation. Scanning electron micrographs of synthesized silver indicated that nano-silver were prepared in morphological structures as nano-particle.*

Keywords: *Silver, Nano-structures, Nano-material, Reduction, Characterization.*

1. Introduction

Nanomaterials have at least one dimension that is 1–100 nm. Nanomaterials are intensely studied currently due to their unique optical, electrical, and catalytic properties. To utilize and optimize the chemical/physical properties of nanosized metal particles, a large spectrum of research has focused on the control of the size and shape, which is crucial in tuning their physical, chemical, and optical properties [1–4].Metal nanoparticles have been the core interest of many researchers in areas of nanotechnology and nanoscience due to their interesting properties and their ease of synthesis [5–7].

Preparation and characterization of novel nanostructures (cubes, disks, plates, prisms, triangles, wires, rods and macroporous) of noble metals, silver and gold, have come a long way of various investigators in many laboratories [8–12] and their uses in photography, catalysis, biological labeling, superconductors, photonics, optoelectronics, super magnets, surface-enhanced Raman scattering detection, environment, sensors, antimicrobial and antibacterial activities, etc. are unlimited [13–17].

Silver nanoparticles can be synthesized using various methods such as chemical reduction [18], electrochemical [19], γ-radiation [20], laser ablation [21], photochemical [22], sonochemical [23] and sputtering [24]. Among these the most popular method for preparation of silver colloid is chemical reduction of silver salt in the presence of any stabilizing agent. Chemical reduction is the most frequently applied method for the preparation of stable silver nanoparticles and its colloidal dispersions in water or organic solvents [25–27]. Most commonly used stabilizing agents are polymers [28] and surfactants [29]. Compared with the major reducing agents reported to date for the preparation of metal nanoparticles such as hydrazine, sodium borohydride, and DMF, ethylene glycol is an environmentally benign material. Its polymer, poly ethylene glycol has been widely applied in the pharmaceutical and

biomedical industries as prodrugs. Ethylene glycol and diols can be used as a reducing agent to prepare metal particles through the so-called polyol process at high temperatures, but control of the reaction is difficult and large metal particles are often formed [30,31].

Silver nanoparticles (Ag NPs) are studied intensively for their potential use in catalysis, biosensors, biomedicine, and environmental filtration [32–36]. Ag NPs have been shown to have inhibitory and bactericidal effects [37]. Enhanced antibacterial activities of some antibiotics, such as penicillin G, amoxicillin, erythromycin, and clindamycin were observed in the presence of Ag-NPs against Staphylococcus aureus and Escherichia coli [38].

In this study, we report the dramatic effect of the reaction parameters and the stabilizing agent (PEG) molecular weight in the nano-silver formation. As the first investigation, we discovered that PEG is able to act as both reducing agent and stabilizer. The reducing reactivity of PEG was very sensitive to its molecular weight.

2. Materials and Methods

2.1 Preparation of silver NPs

The preparation of silver NPs was performed by the addition of an aqueous AgNO3 solution to different stabilizing agent [PEG (400, 1000, 4000, 6000, 8000, 10000 and 20000), TEA, SDS and PVP 40000] in presence of EG as reducing agent. In a typical procedure, 5 g of stabilizing agent was dissolved in 95 g of DI water to prepare a 5 wt% solution.

The aqueous $AgNO_3$ (0.05 M) and EG solution was then added dropwise with a flow rate of 1 mL/min to the stabilizing agent while the temperature was kept constant at 80 °C under magnetic stirring with bubbling of H_2. After the $AgNO_3$ solution had been added completely, the reaction was allowed to proceed under constant stirring at room temperature.

The transparent solution converted to the characteristic pale pink or gray-black color, which indicated the formation of silver nanoparticles. After one day the silver nanoparticles formed as amirror at the wall of beaker.

2.2- Physical Characterization of the Synthesized of silver nanoparticles

The physical properties for both the prepared inorganic siver nanoparticles were investigated using different techniques namely X-ray diffraction, and scanning electron microscope.

2.2.1- X-ray Diffraction (XRD)

X-ray powder diffractometry was carried out using X-ray diffractometer with Cu Kα radiation beam (λ= 0.154060 nm) in order to determine the structure of the prepared silver nanoparticles. The glass sheet samples of the silver nanoparticles were packed in to a flat aluminum sample holder, where the X-ray source was a rotating anode operating at 30 kV and 30 mA with a copper target. Data were collected between 10º and 80º in 2θ.

2.2.2- Morphological Characterization (SEM)

The glass sheet of silver naoparticles were stocked over a holder. Then it was gold-sputtered before examination. The samples were scanned to identify the structure of prepared samples and estimate the particle diameter at different magnifications. The mean diameter of the grains was determined from the SEM pictures by measuring at least 5 crystals for each formulation using the software Image tool.

3. Result and discussion

Silver nitrate was reduced at room temperature using EG to produce silver in nano-scale. The influence of presence the stabilizing agent in the nanosilver production was identified using PEG (400, 1000, 4000, 6000, 8000, 10000 and 20000), TEA, SDS and PVP 40000.

The products obtained from the synthetic condition using the different stabilizing agents where characterized using UV–Vis spectral analysis, XRD, and SEM.

3.1.4.1 UV–Vis spectral analysis

UV–Vis spectral analysis after synthesis was employed. Fig. 1. show the UV–Vis spectra of the silver nanoparticles in the range250–650 nm. A typical silver nanoparticle absorption band in the visible region between 350 and 460 nm was observed at 420 nm. Moreover, there was no clear change in peak position of all different stabilizing agent but stabilizing agent PEG (400, 1000, 4000, 6000, 8000, 10000 and 20000), TEA, SDS and PVP 40000 were arranged in order of absorbance decrease, indicating the amount of produced silver nanoparticles.

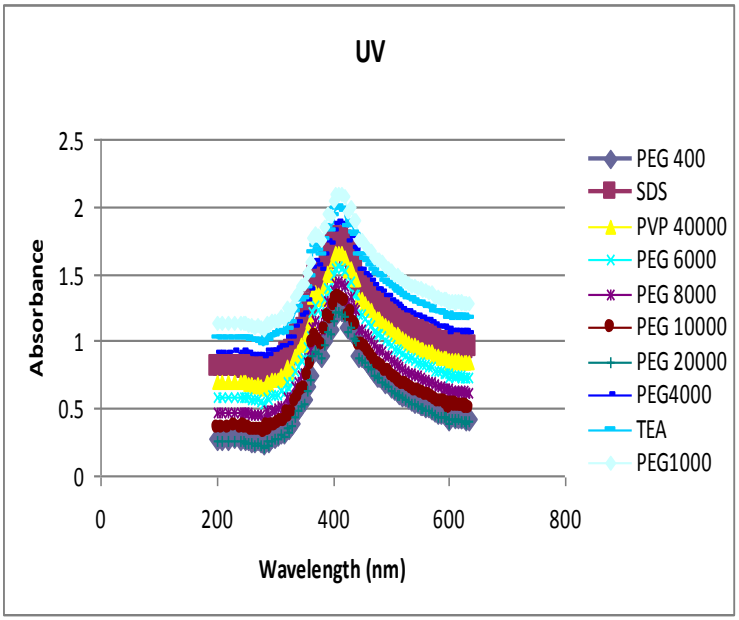
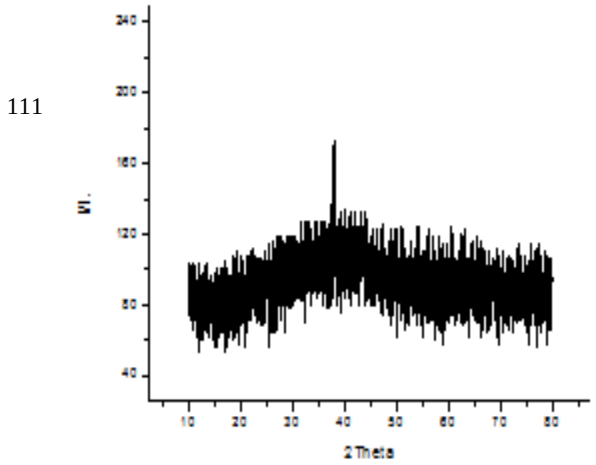


Fig. 1. UV–visible spectrum of the product obtained from stabilizing agent

3.1.4.2 XRD assay

In order to characterize the crystal structure of the silver particles on glass substrates, XRD measurements were performed (Fig. 2,3). The different intense peaks that present in each stabilizing agent were compared with that in the reference of silver phase (JCPDS Card No. 03-065-8428). The X-ray diffraction pattern of the product synthesized by wet chemical reaction method by using PEG 1000 and TEA as stabilizing agent is shown in Fig.2(a and b).

a)



b)

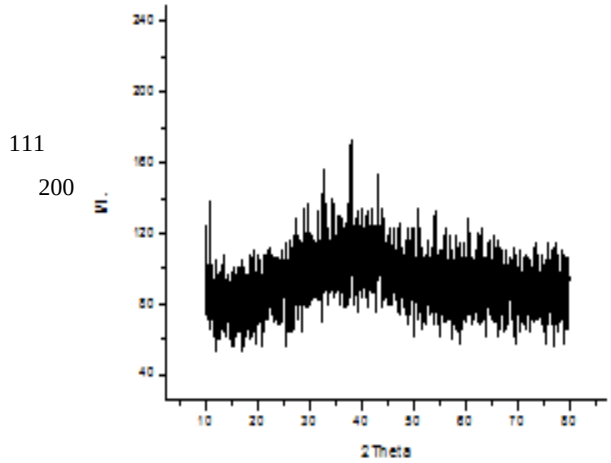


Fig. 2. XRD patterns for the silver nanoparticles produced by different stabilizing agent
a) TEA b) PEG 1000

For Fig.2a only one reflections can be seen which correspond to the (111) of cubic silver. This reflection correspond to pure silver NPs with cubic symmetry.For Fig.2b A number of reflections can be seen which correspond to the (111) and (200) reflections of cubic silver. All the reflections correspond to pure silver NPs with cubic symmetry. The highest peak intensity for cubic materials is represented by (111) orientation plane, which is observed in the prepared sample. The intensity of investigated peaks reflected the high degree of crystallinity of the produced silver nanoparticles. However, the diffraction peaks are broad which indicating that the crystallite size is very small. The XRD shows that silver nanoparticles formed are crystallined.

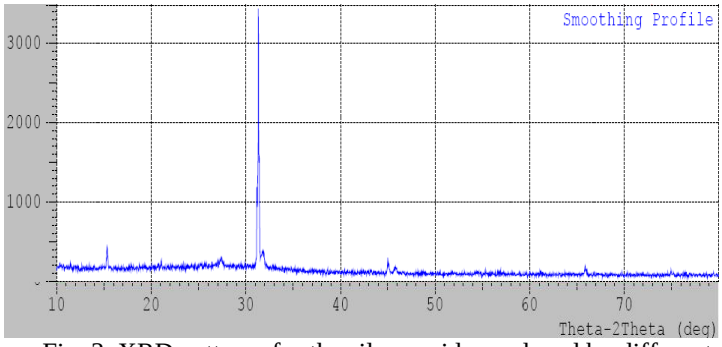


Fig. 3. XRD patterns for the silver oxide produced by different stabilizing agent PEG (400, 4000, 6000, 8000, 10000 and 20000), SDS and PVP 40000

On the other hand Fig.3. showed the X-ray diffraction pattern of the other product synthesized by wet chemical reaction method under heating by using PEG (400, 4000, 6000, 8000, 10000 and 20000), SDS and PVP 40000 as stabilizing agent show diffraction peak presence at 2ɵ = 31.8580° compared with reference card of silver oxide No(# 40-1054) illustrate the formation of silver oxide with different intensity.

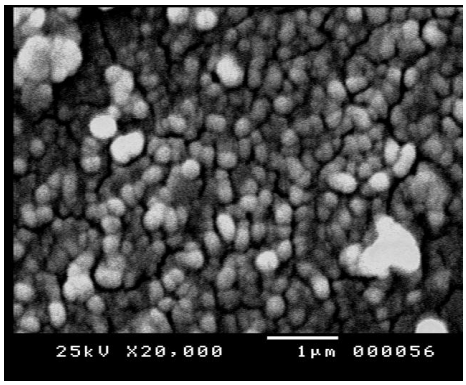


Fig.4. SEM images of nanosilver produced by PEG1000 as stabilizing agent

So, the PEG 1000 and TEA surfactant has the ability to produce pure silver compared with the remaining studied surfactants that produced silver oxide but silver nano particles that formed by PEG1000 higher purity than that formed by TEA. In this regard PEG1000 surfactant agent was selected as the most proper stabilizing agent for the production of pure silver with high production yield as this preparation conditions.

3.1.4.3 Morphological characterization of nano-silver

Fig.4. showed representative SEM image of the produced silver nanoparticles synthesized by PEG1000 as stabilizing agent after reacting with silver nitrate. It was observed that the majority of the silver nanoparticles that produced from the PEG1000 reduction are relatively uniform in diameter and have particles morphology with average particle diameter ranging size at 60–80 nm. However, there is some few large silver aggregated due to the combination between different silver nanoparticles together.

4. Conclusions

In conclusion, the objective of this study was to prepare silver NPs by using different stabilizing agent. Stabilizing agent PEG 1000 was confirmed to be used in synthesis of nanosilver particles. UV–Vis spectroscopy, X-ray diffraction (XRD), and scanning electron microscopy (SEM) were used to characterize formed nanosilver materials. The results clearly confirmed the presence of silver nanoparticles with Stabilizing agent PEG 1000. In final words, this study tried to fill partially the need to develop clean, non-toxic and environmental friendly methods for the synthesis and assembly of silver nanoparticles.

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