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Fabrication of silver nanoparticles/polyaniline composite thin films using Layer-by-Layer self-assembly technique for ammonia sensing

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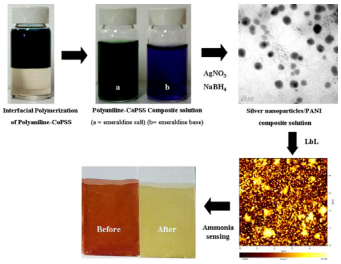
HIGHLIGHTS

- Polyaniline blend CoPSS was synthesized using interfacial polymerization method.
- Polyaniline blend CoPSS was used as the stabilizing agent for silver nanoparticles.
- Multilayer films of Ag nanoparticles/polyaniline-CoPSS were constructed by LbL method.
- The obtained Ag nanoparticles would be of significance for ammonia sensing.
- The color of multilayer thin films was changed from orange-red to yellow.

GRAPHICAL ABSTRACT



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ABSTRACT

In this study, the Layer-by-Layer (LbL) deposition technique was used to prepare composite thin films of silver nanoparticles and polyaniline. The multilayer composite thin films were constructed by alternate deposition of anionic and cationic polyelectrolytes. Anionic polyelectrolyte was prepared by chemical reduction of silver ion using water soluble polyaniline as the stabilizing agent to form anionic silver nanoparticles/polyaniline composite solution. The water soluble polyaniline was prepared by interfacial polymerization of aniline monomer in presence of poly(styrene sulfonic acid co maleic acid), (CoPSS). Silver nanoparticles stabilized with anionic water soluble polyaniline were then deposited in thin film with cationic poly(diallyldimethylammonium chloride), (PDADMAC) and characterized for their optical properties. The optical properties of thin films were measured by UV–visible spectroscopy. The surface of the films was measured by atomic force microscopy (AFM). As these films are to be used as sensor for the ammonia detection, the changes in optical properties of the films were evaluated for various ammonia concentration. A strong color shift from orange-red to yellow was visible when exposed to ammonia. These silver nanoparticles/polyaniline composite thin films displayed fast color change and could be used for ammonia sensing in industrial section.

1. Introduction

In recent years, nanomaterials have received much attention from the scientific community because of their unique properties due to the wide variety of functionalities, size, shape and chemistry. Silver nanoparticles are known as one of the most important

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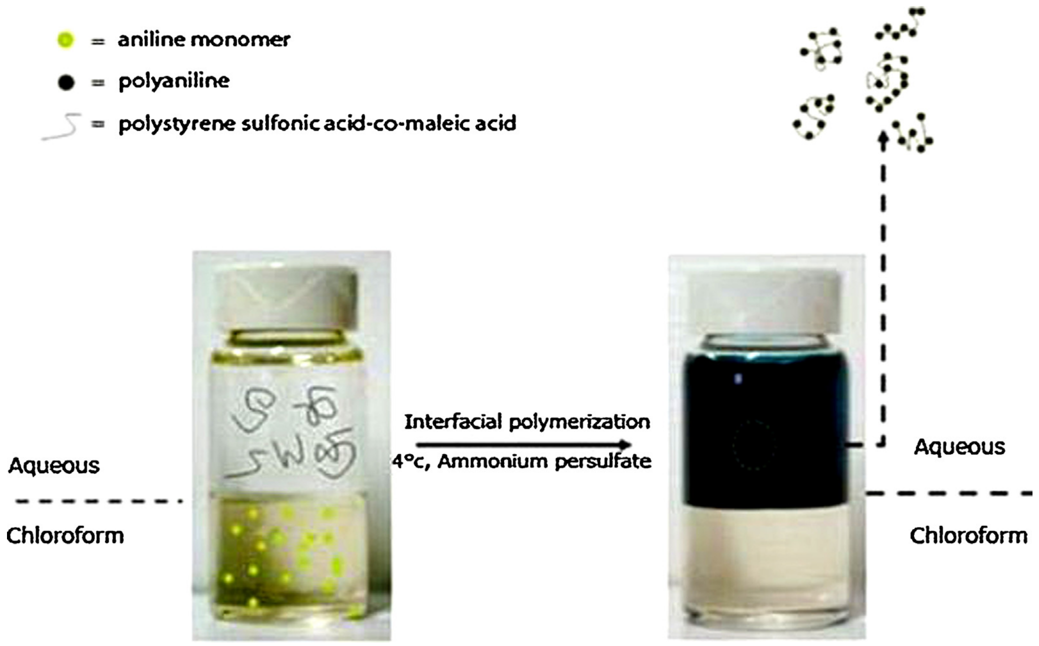


Fig. 1. Interfacial polymerization of aniline in water/chloroform system in the presence of CoPSS.

nanomaterials due to their unique chemical and physical properties. The application of silver nanoparticles is spread on many fields such as sensor [1], catalyst [2], antimicrobial material [3] and electronic devices [4]. Numerous methods have been developed for synthesis silver nanoparticles such as using laser ablation [5], ultrasound [6], gamma radiation [7], ultraviolet radiation [1,8] and chemical reduction [9]. Of each methods, stabilizing agent was purposed in order to control the growth of the nanoparticles. The role of stabilizing agent is to prevent the nanoparticle–nanoparticle interaction by steric hindrance or electrostatic repulsion thus the coalescence and further growth of the nanoparticles [10]. Many stabilizing agents have been used and are chosen such as poly(methylmethaacrylate) [1,11], poly(styrene sulfonic acid) [12], poly(vinylalcohol) [13], gelatin [14], polyamine [15] and poly(vinylpyrolidone) [16].

Polyaniline is unique among the family of conducting polymer, since polyaniline's conducting properties were discovered in the early 1980s [17], it has been extensively studied for many application due to its ease to synthesize, good environmental stability, high conductivity, low monomer cost and reversible doping/dedoping process [18]. Normally, polyaniline is insoluble in water, but several methods have been developed to improve its solubility such as using sulfonic-acid self-doped polyaniline by reacting polyaniline with fuming sulfuric acid ($H_2SO_4{\cdot}SO_3$) in order to promote the sulfonation of polyaniline [19]. Other methods used sulfonated derivatives of aniline monomer such as m-aminobenzene sulfonic

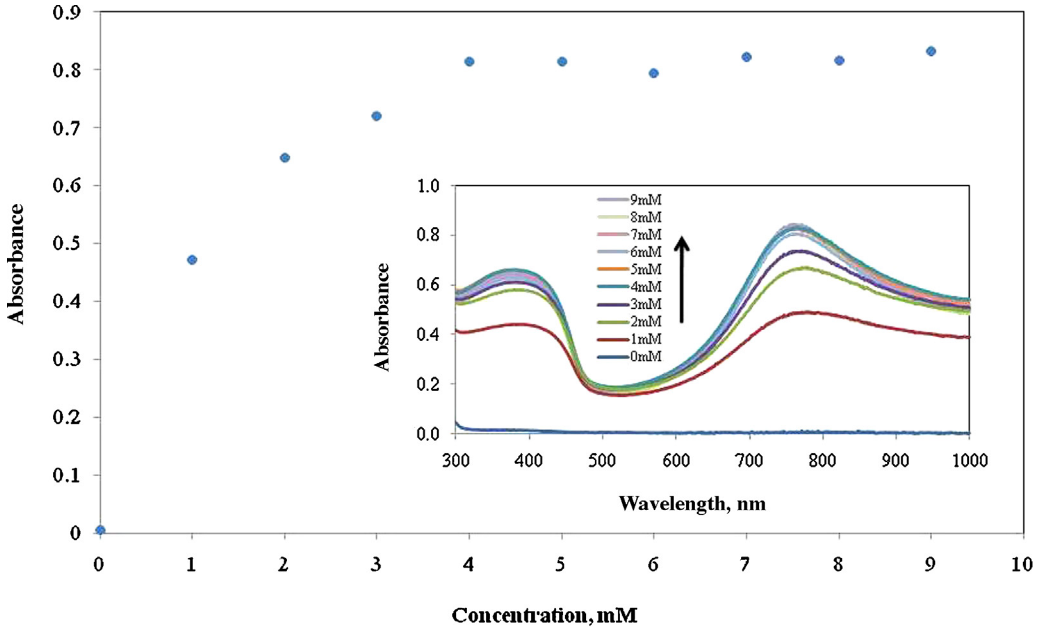


Fig. 2. Plot of the change in absorbance of the aqueous phase after polymerization.

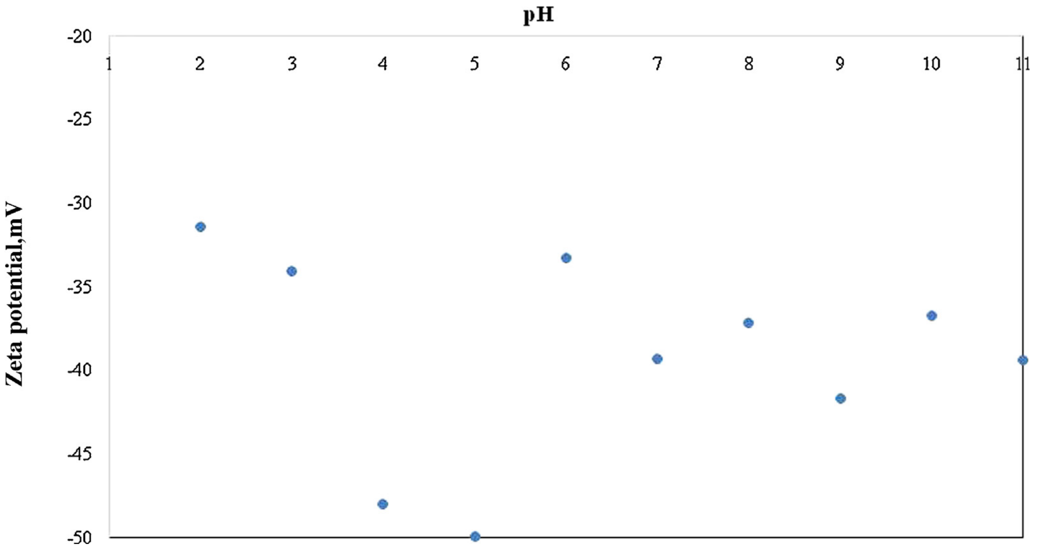


Fig. 3. Zeta potential of water-soluble polyaniline by varying the pH of from 2 to 11.

acid [20] and 2-methylaniline-5-sulfonic acid [21] via chemical oxidation process. In this study, water soluble polyaniline was prepared by the interfacial polymerization in the presence of anionic polymeric acid, poly(4-styrenesulfonic acid-co-maleic acid), sodium salt (CoPSS), due to its provides better solubility to polyaniline and helps in the uniform of stabilizing properties for silver nanoparticles production.

Because of extremely high development of the industry, ammonia is produced in very large quantity by chemical industry such as in refrigeration and fertilizers system. Owing to its toxicity, ammonia is very dangerous for human health even at very low concentration. In the past decade, ammonia sensors have been developed based on different sensing mechanisms such as electrical, mass, or optical-based methods. Among all the sensors developed, optical sensor based on the change in color have attracted extensive attention due to low power consumption, and compatibility with explosive environments. For the fabrication of sensor in this article, silver nanoparticles/polyaniline composite have been prepared and assembled into thin films using the Layer-by-Layer (LbL) technique. This technique, first reported by Decher [22,23] was initially based on the sequential complexation of polyanionics and polycationics species at a substrate interface. This technique developed in the early 90s came as a supplement of previously used methods for the preparation of thin films such as spin coating of polymer solution as well as Langmuir–Blodgett thin film. The LbL technique in the last decade has emerged as an alternative method for the preparation of multilayer thin films mainly due to the versatility of the assembly technique [24], the ease of preparation and the low cost of chemicals used. Based on a simple dipping step, this LbL technique allows the fabrication of sensor thin films and provided outstanding control over the surface properties. The growth of the silver nanoparticles/polyaniline composite thin films was monitored by AFM and UV–vis spectroscopy. Finally, the sensing properties of silver nanoparticles/polyaniline composite

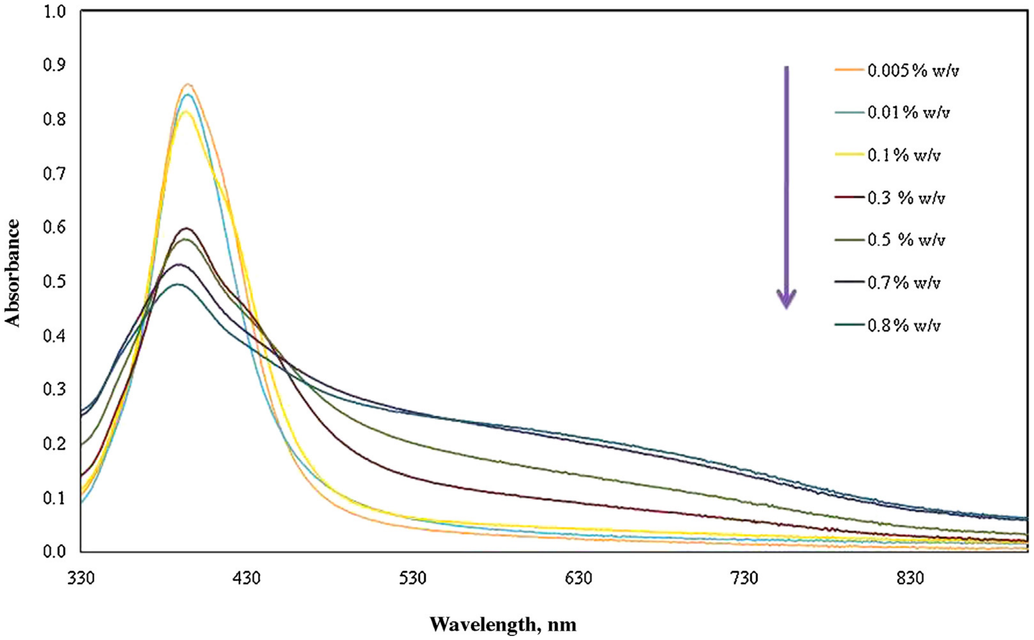


Fig. 4. UV–visible absorption spectra of silver nanoparticles by varying concentration of polyaniline-CoPSS stabilizing agent (0.005, 0.01, 0.1, 0.3, 0.5 0.7 and 0.8% (w/v)).

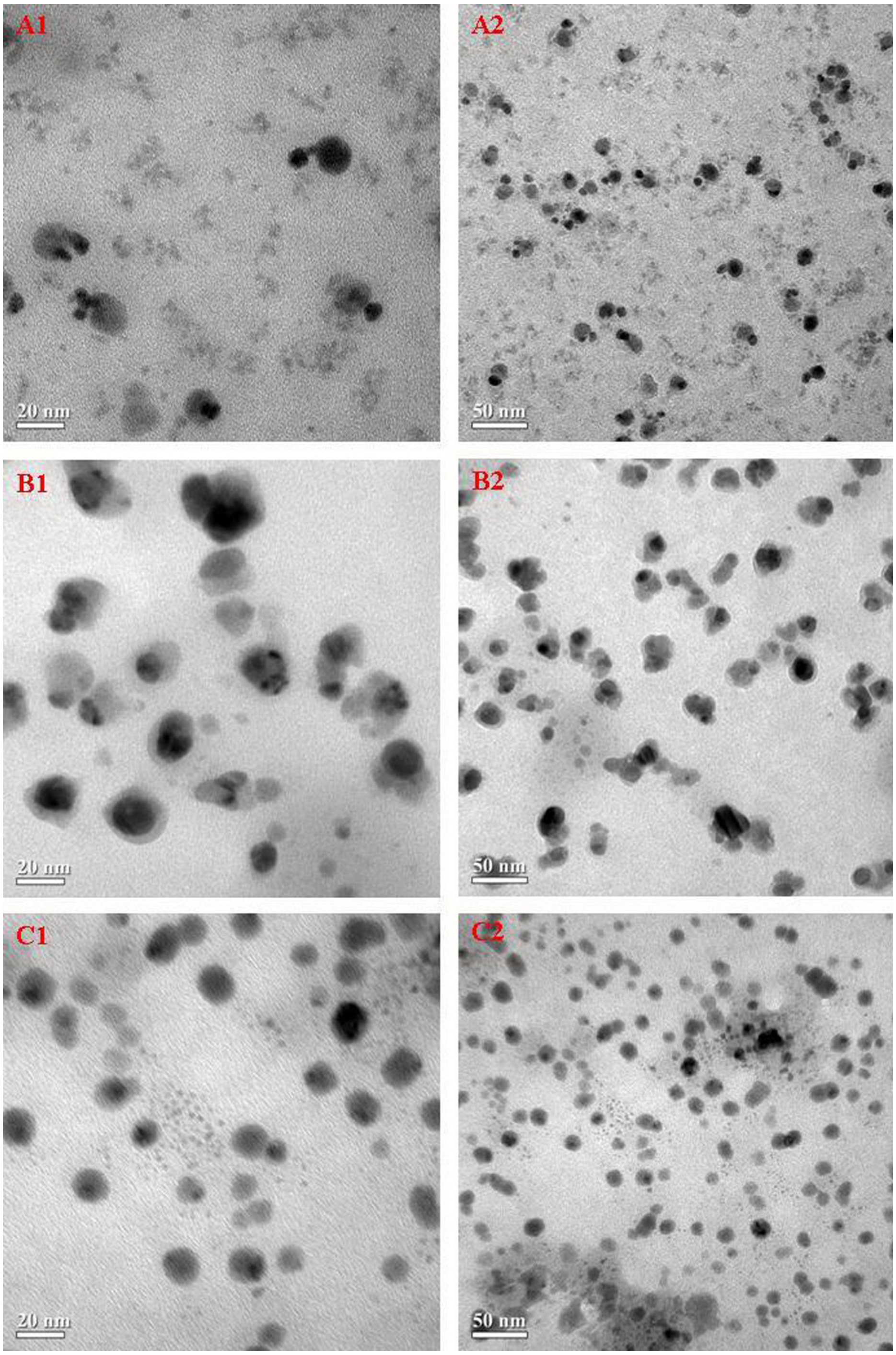


Fig. 5. TEM images of silver nanoparticles at various concentration of polyaniline-CoPSS stabilizing agent (A1 = 0.005% wt, B1 = 0.1% wt and C1 = 0.8% (w/v)).

thin films were tested against increasing ammonia concentration by monitoring the change in LSPR position and amplitude with UV-Vis spectrophotometer.

2. Experimental

2.1. Chemicals

All major chemicals used in this work were of analytical grade and used without further purification or treatment, including aniline monomer (ANI, Aldrich), poly(4-styrenesulfonic acid-co-maleic acid) sodium salt (CoPSS, Aldrich) (molecular weight = 20,000 Aldrich), poly(sodium 4-styrene sulfonate, PSS, molecular weight = 70,000), ammonium persulfate (APS, $(NH_4)_2S_2O_8$, Fluka), silver nitrate ($AgNO_3$, Aldrich), sodium borohydride ($NaBH_4$, Aldrich), chloroform ($CHCl_3$, Aldrich), and hydrochloric acid (HCl, Aldrich). All aqueous solutions were prepared with double-distilled water.

2.2. Interfacial polymerization of water soluble polyaniline blend CoPSS

Water soluble polyaniline in the presence of CoPSS was prepared by interfacial polymerization technique. The experimental

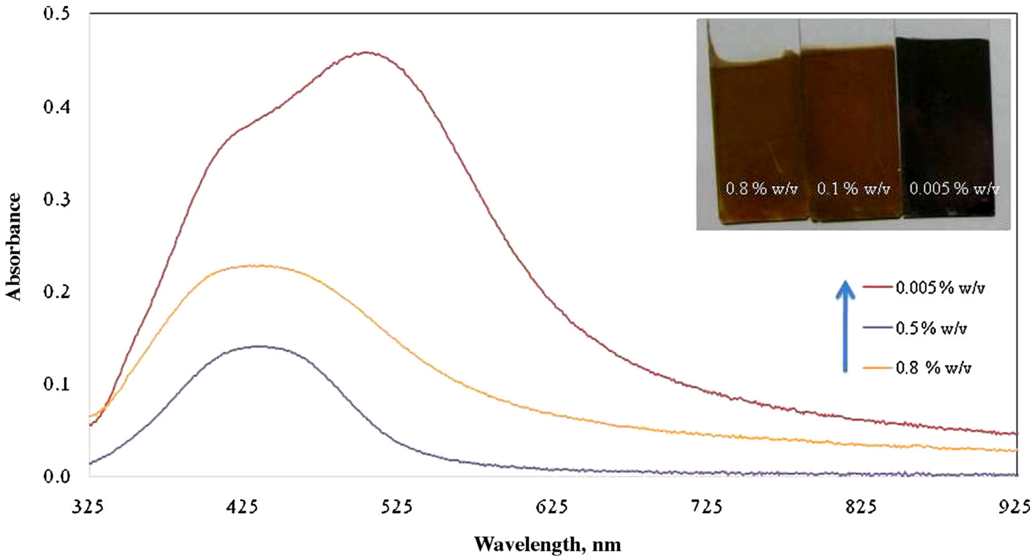


Fig. 6. The absorbance value of silver nanoparticles as the different polyaniline-CoPSS stabilizing concentration.

was performed into two phases of aqueous and organic solution. In aqueous phase, the CoPSS was reacted with oxidizing agent, ammonium persulfate (APS) and HCl dopant concentration. The aqueous solution was carefully poured into aniline monomer dissolved in chloroform solution. The reaction was kept at 4 °C without agitation for 24 h. The green water soluble polyaniline was formed at the interface within a few minutes (Fig. 1). Afterward the aqueous solution was separated from the organic solution. To remove excess acid, byproducts and remained APS from polymerization, the as-prepared water soluble polyaniline was purified either filtration (paper filter, Whatman No. 45) and dialysis (dialysis tube, 3500–6000 MW cutoff, Fisher Scientific).

2.3. Synthesis of silver nanoparticles/polyaniline composite

Silver nanoparticles was prepared using water soluble polyaniline as the stabilizing agent. In a typical experiment, 1 mM silver nitrate solution was mixed with the various concentration of water soluble polyaniline (0.005, 0.01, 0.1, 0.3, 0.5, 0.70 and 0.8% (w/v)). Then 10 mM sodium borohydride was drop into the mixture solution which have the rate of 30 drop/minutes. Sodium borohydride was used in excess volume in order to ensure full reduction of Ag ions. The obtained mixture was allowed to react for 30 minutes under constant stirring at room temperature.

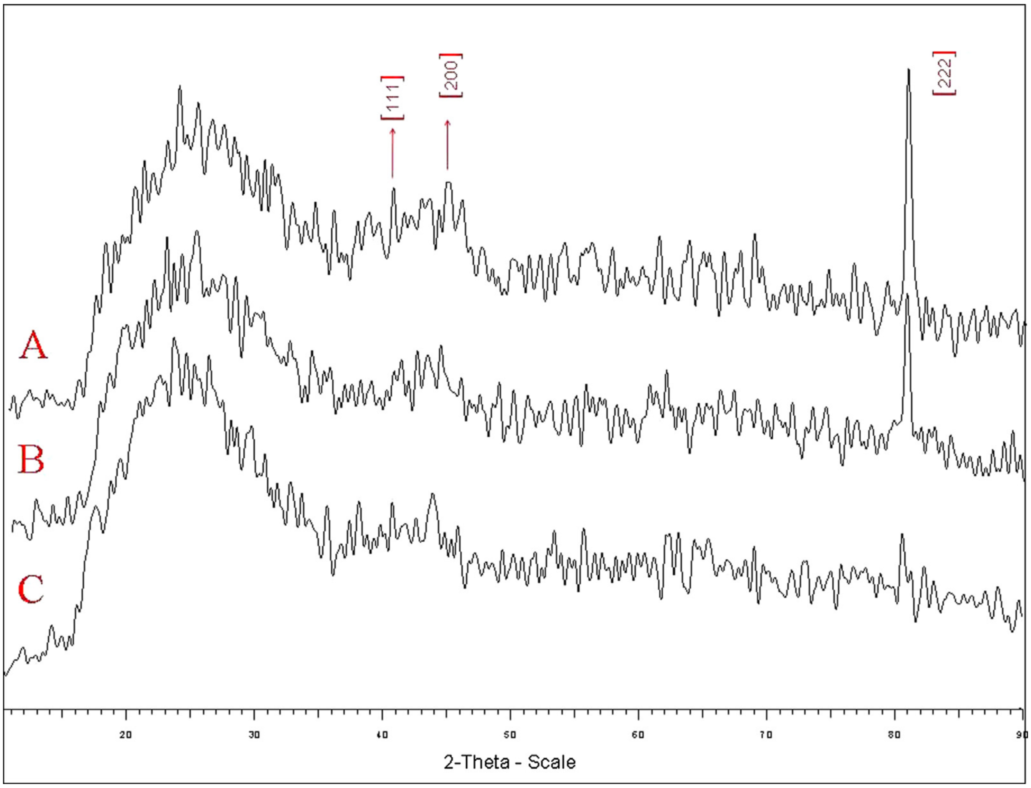


Fig. 7. XRD spectra of silver nanoparticles/polyaniline-CoPSS/PDADMAC multilayer films (A = 0.005% (w/v), B = 0.1% (w/v). and C = 0.8% (w/v)).

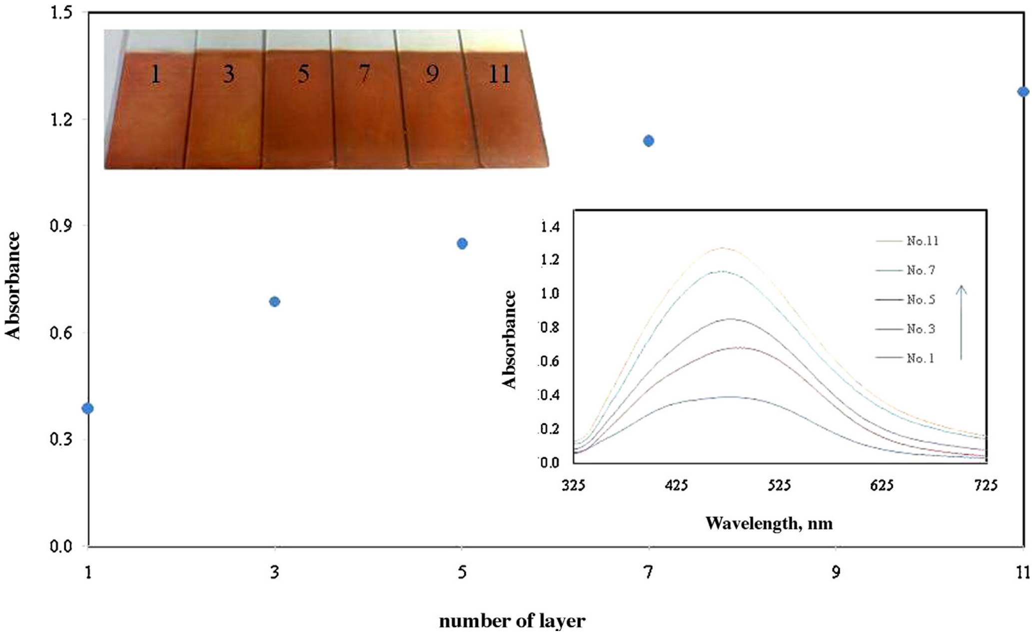


Fig. 8. The absorbance value as the function of number of (silver nanoparticles/polyaniline-CoPSS/PDADMAC multilayer films.

2.4. Layer-by-Layer film assembly

Multilayer thin films were deposited using a home-built automated dipping machine. The general self-assembled produce for preparation multilayer thin films was as follows. First, glass slide substrates were functionalized with strong polyelectrolyte of 10 mM of PDADMAC and PSS in order to generate the positively charges hydrophilic properties of the substrate. Second, the substrates were immersed into the silver nanoparticles/polyaniline composite solution and then rinsed with water to remove the excess of material. Third, the thin films were immersed into 10 mM PDADMAC and then rinsed with water again. The alternate LbL films of each positive and negative charges polyelectrolyte were achieved by repeating this process until the desired number of layer was reached.

2.5. Characterization

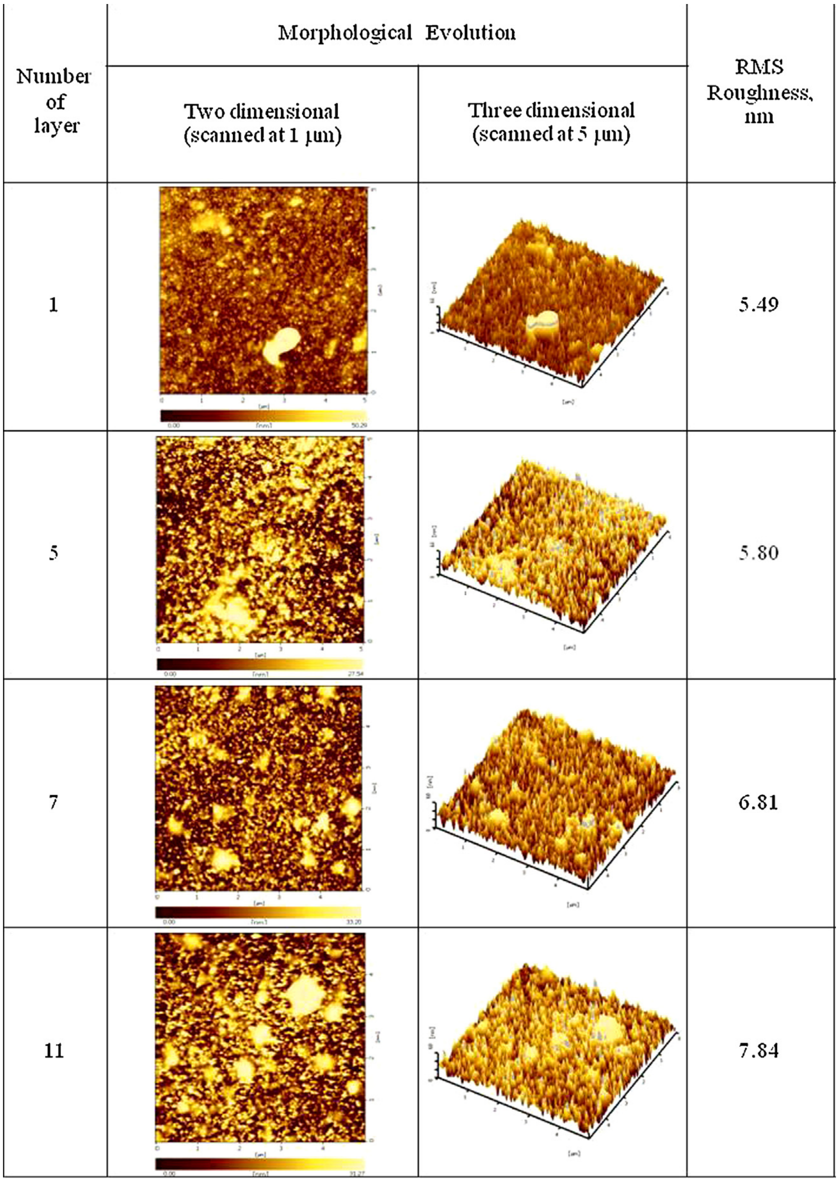
Ultraviolet–visible (UV–vis) spectra were recorded with a Genesys 10S UV–vis spectroscopy. X-ray diffraction (XRD) was done by using Cu Kα radiation on a D8-Discover, Bruker. Transmission electron microscope, (TEM) was done on JEOL, JEM 2001. The surface roughness of the composite thin films was measured by atomic force microscopy, (AFM) (caliper, Veeco) used in tapping mode. Zeta potential was characterized by using the Zeta-seizer nano series, USA.

3. Results and discussion

During the interfacial polymerization, protonated-polyaniline blended with poly(4-styrenesulfonic acid-co-maleic acid), sodium salt can be synthesized by is strongly dependent of the experimental conditions on the polymerization. CoPSS was used to impair the water soluble properties to polyaniline in the interfacial synthesis using the standard condition [25], with increasing CoPSS concentration from 1 to 9 mM. The absorbance of the resulting solution is plotted in Fig. 2. The resulting polyaniline-CoPSS showed good dispersion in water when the concentration of CoPSS was more than 1 mM. It is expected that a minimum concentration of CoPSS is needed to disperse the polymerized polyaniline since CoPSS acted as a template and if CoPSS concentration is too low, the polyaniline precipitate at the interface. The absorbance of the solution was found to increase with increasing the CoPSS concentration from 1 to 4 mM but stable when CoPSS concentration was increased from 5 to 9 mM. As suggested in previously published work, strong electrostatic interaction between sulfonic acid group of CoPSS and amine nitrogen groups of polyaniline backbone are responsible for the templating effect of CoPSS over polyaniline. This is possible because CoPSS which is a polyelectrolyte is completely dissociated into water and adopt a stretched structure. The anilinium cation can adsorbed completely through the electrostatic interaction onto a large amount of sulfonic groups of CoPSS. The stable in absorbance was observed that high CoPSS content tend to encapsulate the polymerized polyaniline too fast and preventing its polymerization.

Our main motivation in this work was to produce silver nanoparticles using water soluble polyaniline as the stabilizing agent. The stabilization is needed to prevent the growth of the nanoparticles. This process occurs during the formation of the nanoparticles when a capping agent adsorbs at the nanoparticles surface. In the present case, water soluble polyaniline in the presence of CoPSS was used as the stabilizing agent for silver nanoparticle production. More specifically, they all broad some carboxylic functional groups along their backbone. Carboxylic functional groups are known to interact and adsorb onto metallic surfaces and are expected to adsorb onto the silver nanoparticles. To further confirm the surface charges of water soluble polyaniline-CoPSS, zeta potential was used to analyze. Shown in Fig. 3 is plotted the effect of pH in polyaniline-CoPSS solution as the function of zeta potential. The polymerized polyaniline-CoPSS were found to have the zeta potential values ranging from −30 to −50 mV when the pH increased from 2 to 11. The negative zeta potential values are due to the carboxylic functional groups of CoPSS template which confirm the polyaniline-CoPSS polymerization.

The effect of the concentration ratio of polyaniline-CoPSS to silver nitrate solution has been studied by varying the reaction conditions. The concentration of silver nitrate was kept constant at 1 mM, different batches were prepared with a polyaniline-CoPSS concentration increasing from 0.05 to 0.38% (w/v) (0.05, 0.15, 0.30 and 0.38% (w/v)). The subsequent formation of silver nanoparticles was monitored by UV–vis spectroscopy as shown in Fig. 4. It



Number of layer	Morphological Evolution		RMS Roughness, nm
	Two dimensional (scanned at 1 µm)	Three dimensional (scanned at 5 µm)	
1			5.49
5			5.80
7			6.81
11			7.84

Fig. 9. AFM images of silver nanoparticles/polyaniline-CoPSS/PDADMAC multilayer films as the function of number of layer.

can be clearly seen that when increase the concentration of water soluble polyaniline, it led to be a broadening and a shift of the absorbance. The shift of plasmon band when increase concentration of water soluble polyaniline stabilizing agent, silver cation can capped with larger amount of anion from polyaniline-CoPSS. This effect may be explained by the changing of the value of the dielectric constant of the media surrounding medium of the nanoparticles. Normally, the position of the adsorption peaks, which are attributed to the surface plasmon resonance, are affected by the nature of metal, size and shape of the nanoparticles and dielectric of the medium. In order to further confirm that the silver nanoparticles could be capped with water soluble polyaniline-CoPSS, transmission electron microscopy (TEM) was executed to investigate the morphology of the silver nanoparticles. The TEM pictures in Fig. 5 show that silver particles are in nano-size and well dispersed. During the growth, particles presented a characteristic shape evolution. The particles were similar in shape for all polyaniline-CoPSS concentration. The particle size is more disperse with increasing polyaniline-CoPSS content and can be seen on panels A1 = 0.005% (w/v), B1 = 0.1% (w/v). and C1 = 0.8% (w/v). This effect is probably due to a more efficient capping of the silver nanoparticles in dilute polyelectrolyte medium.

The prepared silver nanoparticles stabilized with polyaniline-CoPSS were then used for the fabrication of silver nanoparticles/polyaniline composite thin films using LbL self-assembly method. The presence of the charge is fundamental for build up of the film using the advantage of the electrostatic interaction between oppositely charged species of negatively charged silver nanoparticles and positively charged PDADMAC. The films were shown a striking color due to the nanoparticles adsorption (Fig. 6). The lowest polyaniline-CoPSS capping concentration (0.005% (w/v)) was produced a film that appeared shiny due to the close packing of the silver nanoparticles which made the nanoparticles contact one another. This is probably the result of low electrostatic charge and low electrostatic repulsion between particles. This lower electrostatic repulsion between particles has provided higher packing of the particles. On the other hand, when the polyaniline-CoPSS capping concentration is increased (0.1% (w/v) and 0.8% (w/v)) the films appear orange-red color which matches the optical properties of individual silver nanoparticles in

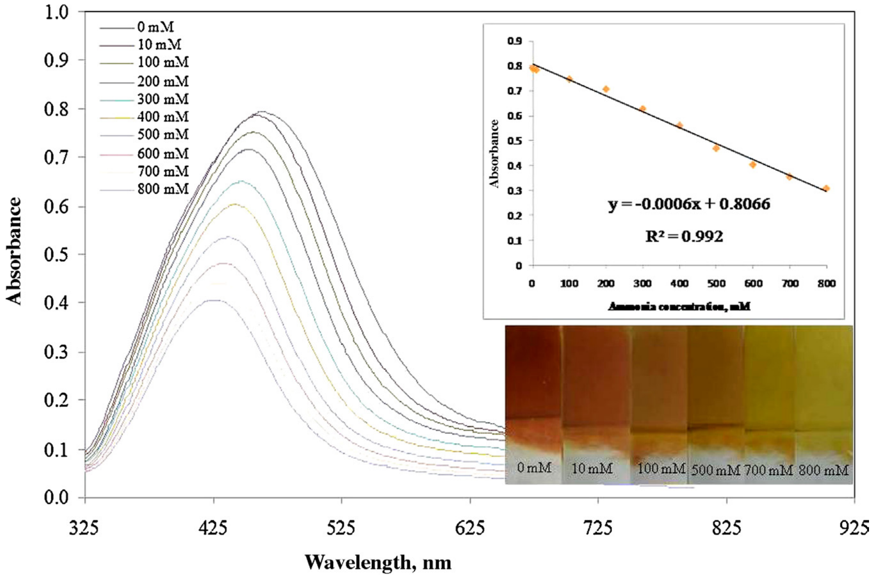


Fig. 10. UV–visible spectrum of silver nanoparticles/polyaniline-CoPSS/PDADMAC multilayer films exposed to various concentration of ammonia (0, 10, 100, 200, 300, 400, 500, 600, 700 and 800 mM).

solution. The nanoparticles posses a vivid orange color as a result of the surface plasmon resonance, (SPR) of the conduction electron, their assembly also led to the formation of an orange colored film.

In order to confirm the growth of silver nanoparticles/ polyaniline composite onto glass substrate, X-ray Diffractometer (XRD) was used to analyze the crystalline structure of the thin films. The X-ray diffraction patterns of silver nanoparticles/polyaniline composite thin films are illustrated in Fig. 7. The prominent peaks at 2θ values of silver nanoparticles/polyaniline composite thin films are shown in 40.1°, 44.8° and 81.2° representing the 1 1 1, 2 0 0, and 2 2 2 Bragg's reflection of the face centered cubic, FCC structure of silver. The XRD diffractrogram of silver nanoparticles which using polyaniline as the stabilizing agent was found agreement with the literature values [26–29] of each Ag nanoparticle production methods.

To investigate the effect of controlling LbL growth, the growth of the silver nanoparticles/polyaniline-CoPSS composite thin films were monitored by AFM and UV–vis spectroscopy as a function of the number of layer. The increase in absorbance values of silver nanoparticles-CoPSS/PDADMAC multilayer films as the function of number of deposition was shown in Fig. 8. It can be seen that after the deposition of a few layer (1–9) the increase in absorbance is constant for each deposition step because the amount of polyelectrolyte being deposited for each dipping cycle is constant. The step increment is the function of the system parameter and is constant if the system parameters are kept constant. For reference, are shown in Fig. 9, 2D and 3D of tapping mode AFM images of silver nanoparticles/polyaniline-CoPSS composite thin films assembled at different number of layer. All AFM images clearly show that silver nanoparticles/polyaniline-CoPSS composite have an interconnection network structure with separated individual silver nanoparticles. The LbL films were shown the separate individual nanoparticles due to the electrostatic repulsion between nanoparticles that act against repulsive force interactions that yield close packed aggregate as well as electrostatic cross-linking between positively and negatively charge electrolyte during the LbL process that lead to randomly oriented, kinetically driven silver nanoparticles arrangements in the film. The RMS roughness values of each AFM images at 1, 3, 5, 7 and 11 layers were calculated to be 5.49, 5.80, 6.81 and 7.84 nm, respectively. It can be seen that the coating

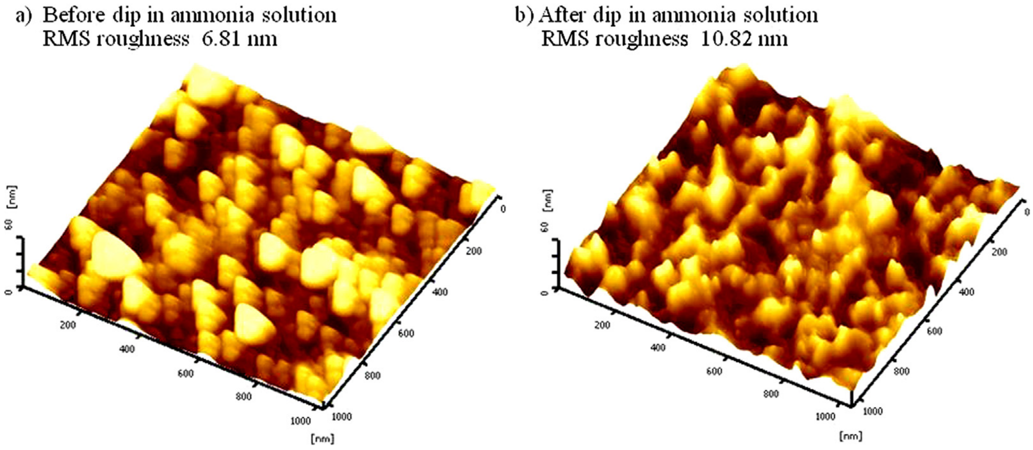


Fig. 11. AFM images of silver nanoparticles/polyaniline-CoPSS/PDADMAC multilayer films before and after dip in ammonia solution.

Fabrication of silver nanoparticles/polyaniline composite thin films using Layer-by-Layer self-assembly technique for ammonia sensing is increasingly dense with individual silver nanoparticles which do not appear to aggregate confirming the efficiency of the polymer coating to onto silver nanoparticles surface.

For the design of optical ammonia sensors, the spectral absorbance of the silver nanoparticles/polyaniline-CoPSS composite thin films was found to display the strong color changes from orange-red to yellow upon adjunction of ammonia concentration. Fig. 10 shows the electronic absorption spectra of silver nanoparticles/polyaniline-CoPSS composite thin films in different ammonia concentration. It can be seen that the initial absorbance peak intensity at 462 nm decreases and it replaced by another peak appearing on blue shift while the ammonia concentration is increased. This color shift suggested the potential development of a sensor or optical device which could be trigger by the changing in dielectric constant of the surrounding medium [30]. This effect is probably due to a decrease in particle distance upon exposure to higher ammonia concentration probably caused by the aggregation of nanoparticles as it has been reported with gold nanoparticles. The AFM images shown in Fig. 11 present the surface topography of the silver nanoparticles/polyaniline-CoPSS composite thin film before and after dip in ammonia solution. It can be seen that the composite thin film after expose into ammonia solution provides the rougher films. The higher roughness value is due to bigger silver nanoparticles particle size.

The range of calibration curve for the determination of ammonia concentration was 0–800 mM (see in cartouche Fig. 10) A limit of detection (LOD, 3σ, $n = 10$) was 0.2038 mM, and a limit of quantitation (LOQ, 10σ, $n = 10$) was 0.6795 mM.

4. Conclusions

Interfacial polymerization of polyaniline in the presence of anionic polymeric electrolyte, CoPSS can open the new method to synthesis the water soluble conducting polymer based stabilizing agent for silver nanoparticles production. The synthetic conditions are very simple, flexible and can be performed with a broad selection of silver nanoparticles production. TEM, UV–visible, XRD provided evidences for the molecular level interaction between silver nanoparticles and polyaniline-CoPSS metric. The resulting composite film was found to be stable when exposed to ammonia solution, which suggest that this composite film could be used for a broad range of conditions.

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