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Research Article

Synthesis of Gold Nanoparticles Stabilized in Dextran Solution by Gamma Co-60 Ray Irradiation and Preparation of Gold Nanoparticles/Dextran Powder

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Gold nanoparticles (AuNPs) in spherical shape with diameter of 6–35 nm stabilized by dextran were synthesized by γ-irradiation method. The AuNPs were characterized by UV-Vis spectroscopy and transmission electron microscopy. The influence of pH, Au^{3+} concentration, and dextran concentration on the size of AuNPs was investigated. Results indicated that the smallest AuNPs size (6 nm) and the largest AuNPs size (35 nm) were obtained for pH of 1 mM Au^{3+}/1% dextran solution of 5.5 and 7.5, respectively. The smaller Au^{3+} concentration favored smaller size and conversely the smaller dextran concentration favored bigger size of AuNPs. AuNPs powders were prepared by spay drying, coagulation, and centrifugation and their sizes were also evaluated. The purity of prepared AuNPs powders was also examined by energy dispersive X-ray (EDX) analysis. Thus, the as-prepared AuNPs stabilized by biocompatible dextran in solution and/or in powder form can be potentially applied in biomedicine and pharmaceutics.

1. Introduction

Synthetic polymers typically polyvinyl alcohol [1], polyvinyl pyrrolidone [2, 3], polyethylene glycol [4], sodium polyacrylate [5, 6], and surfactants [7, 8] have been commonly used as stabilizers in the bottom-up approach of synthesis of gold nanoparticles (AuNPs). In addition, biopolymers such as chitosan [9–11], alginate [12, 13], hyaluronan [14, 15], gum arabic [16, 17], protein [18, 19], gelatin [20], dextran [21], and glucomannan [22] have been also used as stabilizers of colloidal AuNPs solution. On the other hand, materials based on functionalized AuNPs are regarded as one of the most important nanocomposites especially due to their biological and catalytic activities [23, 24]. For example, assembly of chromanol group on AuNPs could efficiently enhance the activity of the vitamin E-derived antioxidant [25]; aptamer-conjugated AuNPs showed the advantages in detection of cancer cells by colorimetric assay [26], AuNPs supported on MgO for CO oxidation [27].

Various methods for the synthesis of AuNPs through bottom-up approach, that is, reduction of Au^{3+} ions in solution, have been reported [11] and the most common method is chemical reduction of gold salt precursor using chemical reducing agents. Compared with other methods, gamma Co-60 ray irradiation has been considered as an effective method with several advantages as described by Hien et al. [15]. In addition, gamma Co-60 ray irradiation has been also considered as a green method to noble metal nanoparticle synthesis [28]. Dextran polysaccharide composed of repeated monomeric glucose units with a predominance of 1,6-α-D-glucopyranosyl linkages and annual world production is of about 500 metric tons [29]. Dextran is readily soluble in water

and electrolytes with excellent stability and it has wide range of use in food, medical related areas, and biological functions, especially for blood flow improvement by reduction of blood viscosity and inhibition of erythrocyte aggregation [30, 31]. Therefore, dextran stabilized AuNPs are promising to use for intravenous administration as X-ray contrast agent [16, 32], cancer therapy [33, 34] including photothermal cancer therapy [35, 36], and for other purposes of application as well [23].

In the present study, AuNPs were synthesized by gamma Co-60 ray irradiation method using dextran as stabilizer and hydroxyl radical ($^{\bullet}OH$) scavenger. The influence of pH, Au^{3+} and dextran concentrations in aqueous solution on the size of AuNPs was studied. AuNPs powders from AuNPs/dextran solution were prepared by spray drying, coagulation, and centrifugation methods. The purity and size of the AuNPs powders were also examined.

2. Experiments

2.1. Materials and Chemicals. Hydrogen tetrachloroaurate trihydrate ($HAuCl_4{\cdot}3H_2O$) was obtained from Merck, Germany. Dextran (MW 60,000–90,000) was purchased from Himedia, India. Other chemicals were of analytical grade and used as received. Distilled water was used in all experiments. Glassware was treated with regia solution (1V HNO_3 : 3V HCl), washed with distilled water, and dried.

2.2. Preparation of Au^{3+}/Dextran Solution and γ-Irradiation. Two stock solutions particularly 10 mM Au^{3+} and 4% dextran were prepared by dissolving $HAuCl_4{\cdot}3H_2O$ and dextran into water. Au^{3+}/dextran solutions were prepared by pouring Au^{3+} solution into dextran solution with desired different concentration while stirring for about 10 min. pH of Au^{3+}/dextran solution was adjusted to 5.5, 6.5, and 7.5 by 0.5% NH_4OH solution. Then, the prepared Au^{3+}/dextran solutions of 100 ml were put into glass bottles with plastic cap. Irradiation of Au^{3+}/dextran solutions was carried out on the gamma Co-60 irradiator SVST Co-60/B (Hungary) with dose rate of 1.1 kGy/h at VINAGAMMA Center, Ho Chi Minh City, at ambient condition with required doses (~6 kGy for reduction of 1 mM Au^{3+}) as reported in our previous paper [15].

2.3. Characterization of AuNPs/Dextran. The absorption spectra of AuNPs/dextran solutions obtained after γ-irradiation were taken on an UV-Vis spectrophotometer model UV-2401PC (Shimadzu, Japan). The size and size distribution of the AuNPs were characterized from TEM images on transmission electron microscope (TEM) model JEM1010 (JEOL, Japan) and statistically calculated from about 300 particles. The AuNPs/dextran solution from 1 mM Au^{3+}/1% dextran sample was used to prepare AuNPs powder by spray drying on spray dryer model ADL311 (Yamato, Japan) and by centrifugation on ultra centrifuged machine model Ultra 5.0 (Hanil Science Industrial, Korea) at 30,000 rpm for 20 min. After centrifugation, the AuNPs precipitate was isolated and dried in forced air oven (DNF 410, Yamato, Japan) at 60°C.

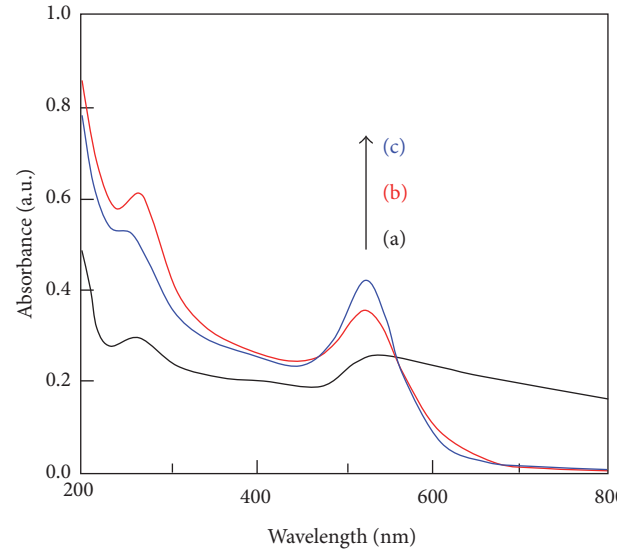

Figure 1: UV-Vis spectra of 1 mM AuNPs/1% dextran solutions at different pH: 5.5 (a), 6.5 (b), and 7.5 (c).

The AuNPs powder was also prepared by coagulation with solvent mixture of ethanol/acetone with equal volume ratio. For coagulation of AuNPs/dextran, 8 volumes of solvent mixture were used to mix with 1 volume of AuNPs/dextran solution. The resulting AuNPs/dextran coagulate was filtered and washed several times with solvent and dried in forced air oven at 60°C. The purity of AuNPs/dextran powders was assessed by energy dispersive X-ray spectroscopy (EDX) on a JEOL 6610 LA. The UV-Vis spectra and TEM images of AuNPs powders redissolved in water were also taken.

3. Results and Discussions

After irradiation, the color of Au^{3+}/dextran solution turned from yellow to ruby-red which is surface plasmon resonance characterized for AuNPs [11, 15]. The mechanism of reduction of Au^{3+} to Au° by γ-irradiation method through the reducing species which resulted from water radiolysis was described by Henglein [1].

3.1. Effect of pH. Results of UV-Vis spectra in Figure 1 indicated that the maximum absorption wavelengths (λ_{max}) of AuNPs/dextran solutions were of 543.5, 526.0, and 520.5 nm for pH 5.5, 6.5, and 7.5, respectively.

Results of TEM images and size distribution histograms in Figure 2 indicated that the size of AuNPs decreased with the increase of pH value and particularly the particle sizes were of 35.5, 24.1, and 6.2 nm for pH 5.5, 6.5, and 7.5, respectively. It is clear that slightly basic medium (pH 7.5) was a suitable condition to synthesize AuNPs stabilized by dextran with smaller size (6.2 nm) and narrower particles size distribution (Figures 2(C) and 2(c)) compared to lower pH (6.5) (Figures 2(B) and 2(b)) or acidic medium pH 5.5 (Figures 2(A) and 2(a)) by γ-irradiation method. The obtained results suggested that adjustment of pH of Au^{3+}/dextran

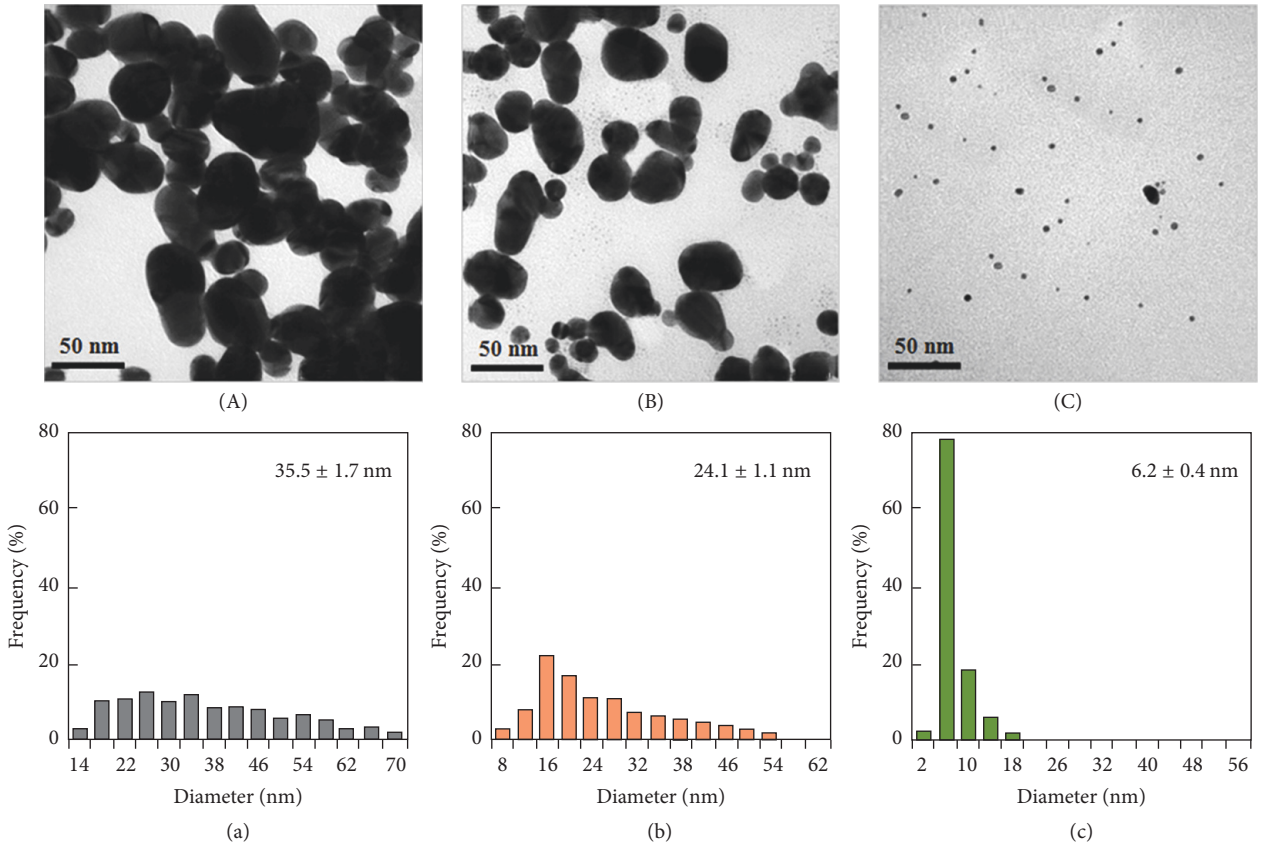

Figure 2: TEM images and size distribution histograms of AuNPs/dextran at different pH: 5.5 (A, a), 6.5 (B, b), and 7.5 (C, c).

solution before irradiation significantly affected the size of AuNPs. The same results of the pH effect were also reported by Phu et al. on radiolytic synthesis of AgNPs stabilized by chitosan [37]. Particularly, the sizes of AgNPs were of 15.0 and 7.3 nm for pH 3 and 6, respectively.

3.2. Effect of Au^{3+} Concentration. In this experiment, AuNPs were synthesized from Au^{3+}/detxran solutions containing 1% (w/v) dextran with various Au^{3+} concentrations particularly 0.5, 1.0, and 2.0 mM. The pH of Au^{3+}/dextran solutions was adjusted to 7.5. The UV-Vis spectra in Figure 3 showed the λ_{max} at 519.5, 520.5, and 524.0 nm for Au^{3+} concentration of 0.5, 1.0, and 2.0 mM, respectively.

The TEM images and the size distribution histograms of the three AuNPs/dextran solutions with various Au^{3+} concentrations were shown in Figure 4. The sizes of AuNPs were of 5.8, 6.3, and 11.9 nm for Au^{3+} concentrations of 0.5, 1.0, and 2.0 mM, respectively. Thus, as Au^{3+} concentration increased the sizes of the AuNPs were bigger and λ_{max} shifted to longer wavelengths. The increase of AuNPs size with the increasing of Au^{3+} concentration was also reported in previous articles for AuNPs/PVP [3], AuNPs/alginate [13], and AuNPs/hyaluronan [15]. The reason for this phenomenon may be due to the development of clusters and the agglomeration among AuNPs when the ratio of stabilizer and Au^{3+} concentration is not high enough [15].

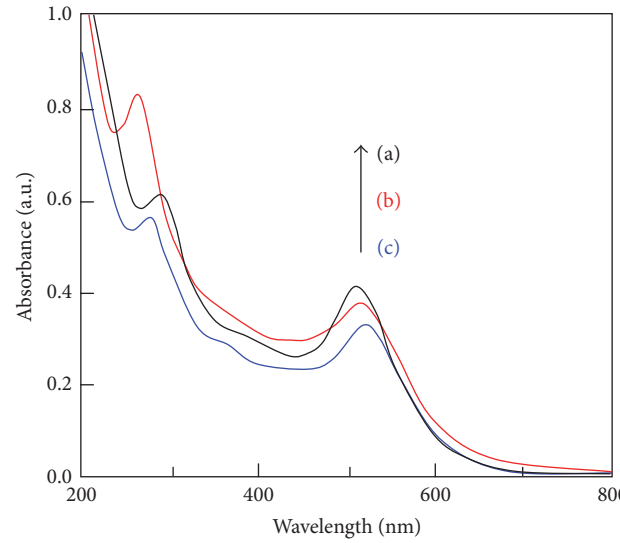

Figure 3: UV-Vis spectra of AuNPs/dextran solutions with various Au^{3+} concentrations: 0.5 (a), 1.0 (b), and 2.0 mM (c).

3.3. Effect of Dextran Concentration. In this experiment, the Au^{3+} concentration was fixed at 1 mM while the dextran concentrations were of 0.5, 1.0, and 2.0% (w/v) and the pH of Au^{3+}/dextran solutions was also adjusted to 7.5. Results of UV-Vis spectra in Figure 5 showed λ_{max} at 522.5, 520.5, and

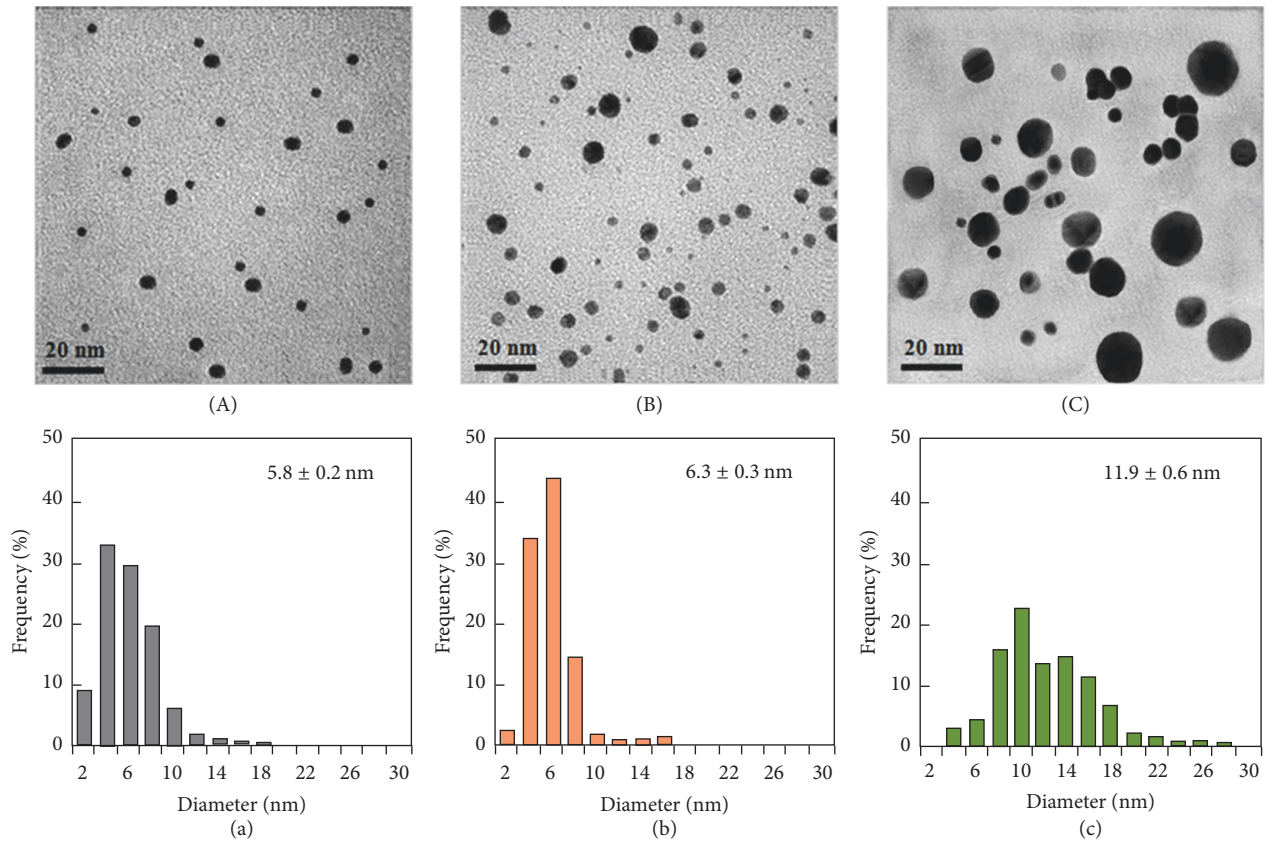

FIGURE 4: TEM images and size distribution histograms of AuNPs/dextran at various Au^{3+} concentrations: 0.5 (A, a), 1.0 (B, b), and 2.0 mM (C, c).

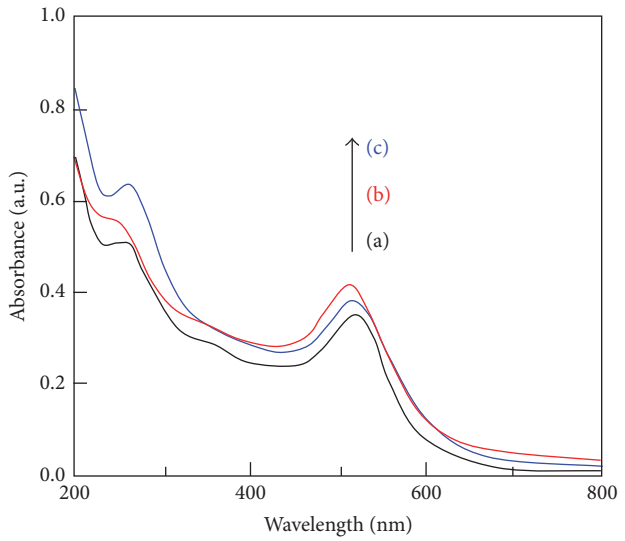

FIGURE 5: UV-Vis spectra of AuNPs/dextran solutions from different dextran concentrations: 0.5 (a), 1.0 (b), and 2.0% (c).

519.0 nm for the dextran concentrations of 0.5, 1.0, and 2.0%, respectively.

The sizes of AuNPs decreased with the increase of dextran concentration particularly the sizes of 8.0, 6.2, and 5.2 nm for dextran concentrations of 0.5, 1.0, and 2.0%, respectively (Figure 6). Thus, the decrease in the size of AuNPs was not much significant as the increase of dextran concentration from 0.5 to 2.0%.

Dextran has good ability to stabilize AuNPs due to the chain of dextran which consists of –OH and –COR groups which stabilize AuNPs thought steric and electrostatic effects [38]. Simultaneously, dextran acted as free radical scavenger via hydrogen abstraction by $^{\bullet}H$ and $^{\bullet}OH$ released during radiolysis of water [11, 13, 15]. Therefore, $^{\bullet}OH$ scavenger such as alcohols is not necessary to add into Au^{3+}/dextran solution as in the case of Au^{3+}/PVP to prepare colloidal AuNPs solution by γ-irradiation method [3]. As described by Hien et al. [15], mass production of colloidal AuNPs solution can be suitably carried out by gamma Co-60 ray irradiation method. The production rate (Q) of a gamma Co-60 irradiator can be calculated by (1) [39] as follows:

$$Q\,(\mathrm{kg/h}) = \frac{(3{,}600 \times P \times f)}{D}, \quad (1)$$

where D is the absorbed dose (kGy), f is the radiation energy utilization efficiency, and P is source power (kW) (100 kCi is equivalent to 1.48 kW).

Assuming that one gamma Co-60 irradiator with activity of 300 kCi (eqv. 4.44 kW) is used for irradiation of Au^{3+}/dextran solution with Au^{3+} concentration of 1 mM, at

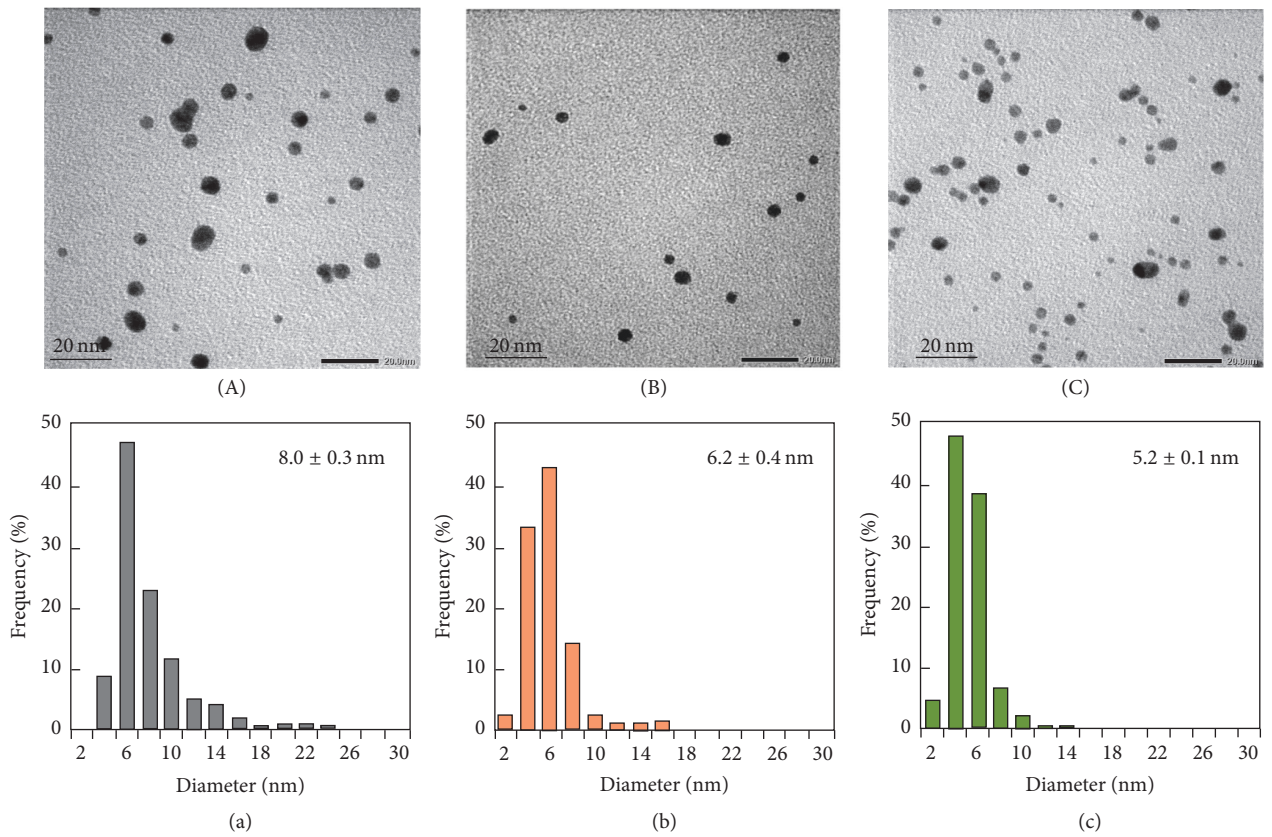

Figure 6: TEM images and size distribution histogram of AuNPs/dextran at different dextran concentrations: 0.5 (A, a), 1.0 (B, b), and 2.0% (C, c).

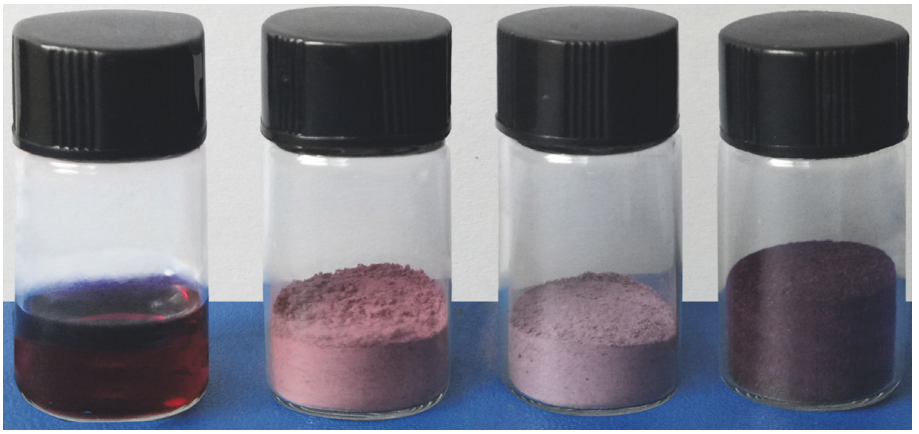

Figure 7: Photograph of AuNPs/dextran solution (a), spray drying (b), coagulation (c), and centrifugation (d) of AuNPs powders.

dose of 6 kGy and with $f = 0.35$, then the production rate of AuNPs/dextran solution calculated according to (1) will be of 932 kg/h (~932 litters/h). Besides mass production, the obtained colloidal AuNPs solution is of high purity without residues of excessive chemical reductant and by-products from chemical reductant after reduction process that were typically analyzed for using trisodium citrate as reducing agent [40].

3.4. Characteristics of AuNPs Powders. Photograph of AuNPs/dextran solution and powders was presented in Figure 7. The AuNPs powders are brownish-pink to dark-brownish in color depending on the powdering method.

Figure 8 presented the UV-Vis spectra of original AuNPs/dextran solution and AuNPs solutions from AuNPs powders. The values of λ_{max} and the sizes of AuNPs were shown in Table 1.

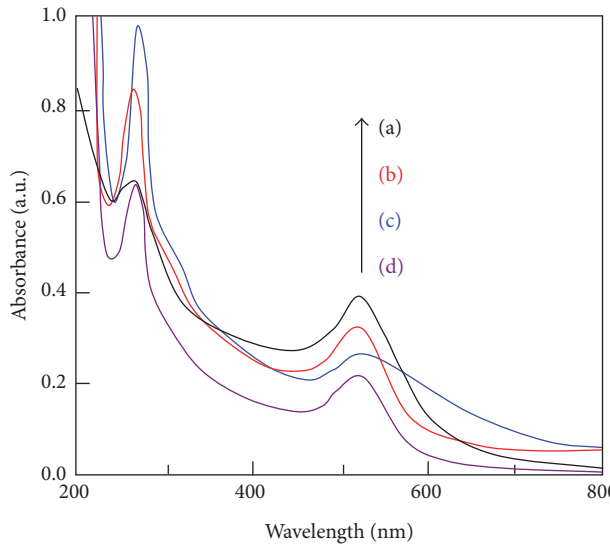

FIGURE 8: UV-Vis spectra of original AuNPs/dextran solution (a), and AuNPs/dextran solutions from AuNPs/dextran powders: spray drying (b), centrifugation (c), and coagulation (d).

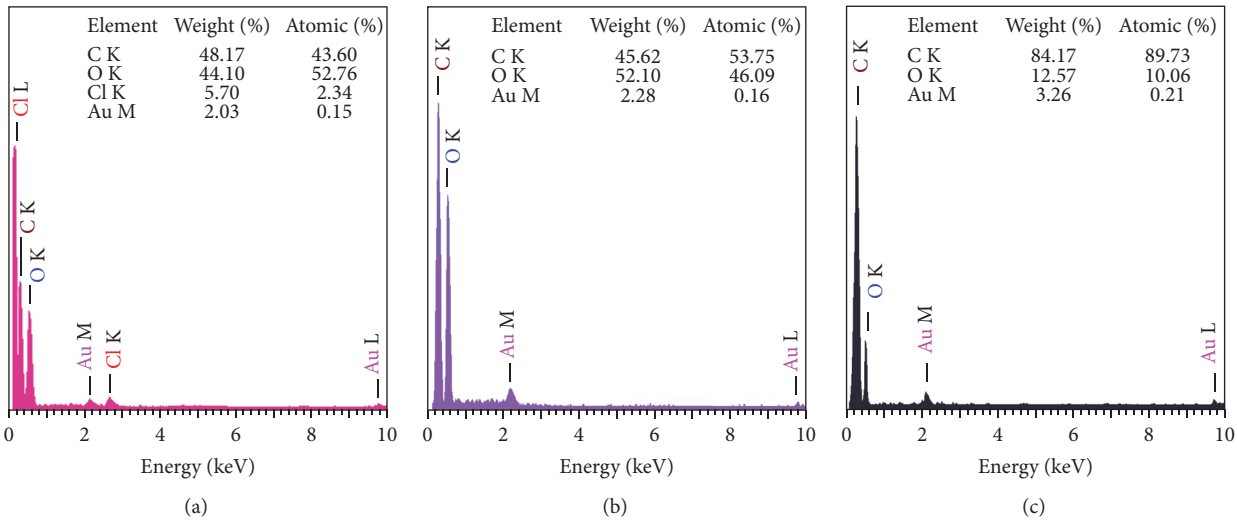

FIGURE 9: EDX spectra of AuNPs/dextran powders: spray drying (a), coagulation (b), and centrifugation (c).

TABLE 1: The values of λ_{max} and the size of AuNPs (d) from AuNPs/dextran solution and powders prepared by different methods.

AuNPs sample	λ_{max}, nm	d, nm
Solution	520.5	6.2 ± 0.4
Spray drying	522.0	7.5 ± 0.5
Coagulation	522.5	8.0 ± 0.3
Centrifugation	523.5	11.2 ± 0.5

It is interesting to note that the value of λ_{max} and the size of AuNPs powders made from spray drying and coagulation were almost not much changed in comparison with those of original AuNPs/dextran solution, but these parameters increased for the AuNPs made from centrifugation (Table 1). The reason may be due to aggregation of AuNPs which occurred during centrifugation with ultrahigh speed (30,000 rpm). To the best of our knowledge, no research of the effect of AuNPs powder preparation methods on the characteristics of AuNPs, particularly λ_{max} and the size, has been reported yet except the study of the purity of AuNPs synthesized by using trisodium citrate as reducing agent and purified by centrifugation method which was reported [40]. Nevertheless, the final form of AuNPs in this study was not powder but referred to as "treated AuNPs suspension."

Results of the EDX spectra in Figure 9 indicated that the AuNPs powders prepared by coagulation and centrifugation did not contain chlorine (Figures 9(b) and 9(c)); however the AuNPs powder prepared by spray drying was contaminated by 5.7% chlorine (Figure 9(a)). Thus, it can be deduced that AuNPs powders prepared by coagulation and centrifugation

were of high purity. In other words, the AuNPs/dextran powders obtained by coagulation and centrifugation methods were effectively purified. However, chlorine content in AuNPs powder should be analyzed with more precise methods.

The stability of colloidal AuNPs solution depends on various factors such as pH, dielectric constant, and concentration of ligand around the particles [19]. Furthermore, the stability of colloidal AuNPs solution stored at low temperature is better than at high ones [40]. In general, polysaccharides such as chitosan, hyaluronan, alginate, and dextran with oxygen rich structures in hydroxyl and ether groups lead to tightly binding polysaccharides to nanoparticles via steric and electrostatic interaction [38]. However, for a long time of storage, the AuNPs in colloidal AuNPs solution will be gradually aggregated to form bigger sizes due to sedimentation phenomenon of AuNPs in colloidal system. In addition, storage and transportation of colloidal AuNPs solution are not always convenient. In fact, it was observed that several samples of the synthesized AuNPs/dextran solutions stored at normal condition were randomly attacked by fungi. Therefore, in those aspects AuNPs powder is much more suitable. Besides, AuNPs/dextran powder holds great potential for biomedical and pharmaceutical applications; especially those AuNPs/dextran powders can be sterilized suitably by radiation method (gamma Co-60 ray, electron beam) [41] for use in intravenous administration for X-ray contrast imaging, photothermal cancer therapy, and in other purposes of application as well.

4. Conclusions

Dextran stabilized AuNPs with diameter in the range of 6–35 nm were synthesized by gamma Co-60 ray irradiation of Au^{3+} in dextran solution. The gamma Co-60 ray irradiation method for production of AuNPs can be favorably carried out on large scale. The AuNPs sizes can be controlled by varying pH, Au^{3+}, and dextran concentrations. AuNPs powders with the almost unchanged size were also prepared by spray drying, coagulation, and centrifugation methods. Due to the biocompatibility of dextran, the purity of AuNPs/dextran colloidal solution as well as the AuNPs/dextran powders and unique attributes of AuNPs, AuNPs/dextran can be potentially applied in biomedicine and pharmaceutics especially in X-ray contrast imaging and photothermal cancer therapy.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

Acknowledgments

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