Carbohydrate Polymers 89 (2012) 1–6

Contents lists available at SciVerse ScienceDirect

Carbohydrate Polymers

journal homepage: www.elsevier.com/locate/carbpol

Investigation of silver nanoparticles synthesis using aminated β-cyclodextrin

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ARTICLE INFO

Article history:
Received 7 December 2011
Received in revised form
31 December 2011
Accepted 16 January 2012
Available online 2 February 2012

Keywords:
Nanotechnology
Silver nanoparticles
β-Cyclodextrin

ABSTRACT

Aminated β-cyclodextrin was prepared through the reaction of 2-chloroethylamine with β-cyclodextrin. The preparation was carried out under different conditions (time, temperature, concentration of NaOH, and concentration of 2-chloroethylamine). The aminated β-cyclodextrin was used as reducing and stabilizing agent for the preparation of silver nanoparticles from $AgNO_3$. Factors (pH, temperature, time, extent of amination and concentration of aminated β-cyclodextrin) affecting the preparation of silver nanoparticles were studied. The prepared silver nanoparticles were evaluated by UV–visible spectral analysis and transmission electron microscopy (TEM). The results obtained indicate that the optimum conditions for preparation of silver nanoparticles with size ranged from 1 to 9 nm could be produced using 0.6 g β-cyclodextrin derivative, 0.1 M $AgNO_3$ at pH 12, 70 °C for 20 min.

1. Introduction

According to environmental restrictions, a continuous demand for biodegradable materials for industrial application is very important. Cyclodextrins and their derivatives constitute a group of chemicals belonging to such type of materials (Buschmann, Knittel, & Schollmeyer, 1990; Knittel, Buschmann, & Schollmeyer, 1991). Cyclodextrins are oligosaccharides produced by Bacillus macerans during the enzymatic degradation of starch and related compounds (Fouda, 2005; Huang & Tonell, 1998).

The first reports of the naturally occurring cyclodextrins (CDs) appeared in the late nineteenth and early twentieth (Easton & Lincoln, 1999). Since discovery of cyclodextrin an investigations of CD chemistry have shown an amazing increase (Bender, & Komiyama, 1978; Easton & Lincoln, 1999; Huang & Tonell, 1998). Cyclodextrins are cyclic (α-1,4)-linked oligosaccharides of α-gluco-pyranose containing a relatively hydrophobic central cavity and hydrophilic outer surface. Cyclodextrins are cone shaped molecules. The primary hydroxyl groups are located on the narrow side of the cone while the secondary hydroxyl groups are located on the wider edge. This special conformation of the molecules results in external hydrophilicity and internal hydrophobicity.

There are 3 types of cyclodextrins, α-, β-, and γ-cyclodextrin, which consist of six, seven and eight glucopyranose units respectively (Fig. 1). The common CDs differ not only in their diameter, related to the number of glucopyranose units, but also in their water solubility. The water solubility of α- to β- to γ-CD at 25 °C is 14.5, 1.85 and 23.2 g/100 ml respectively. Thus, β-CD, which for many years has been the most easily available and commonly employed, is the least water-soluble of the series. However, many other derivatives have been prepared and present high water solubility (Fouda, 2005). The most important characteristics of CDs and their derivatives are their ability to include various or part of molecules inside their hydrophobic cavity, leading to an inclusion complex exhibiting new physicochemical and biological properties (Hirayama & Uekama, 1987). One of the most important application of CDs is in the field of pharmaceutical (wound healing, drug delivery, etc.) (Stella & Rajewski, 1997; Uekama, Hirayama, & Irie, 1998). CDs can play a major role in environmental science in terms of removal highly toxic substances from industrial effluent by inclusion complex formation. Textile finishing is another area in which CDs are increasingly attracting attention (Abou-Okeil & El-shafie, 2011; Denter, Buschmann, Knittel, & Schollmeyer, 1997).

Today nano-scale materials are considered one of the interesting fundamental sciences. The interest in nanoparticles of these typical sizes is due to the fact that the magnetic, optical and electronic behaviors of bulk materials can be modified when their size approaches the nanometer scale (one nanometer is 10^{-9} of a meter). The properties of materials can be different on nanoscale for two main reasons: first, nano-materials have, relatively, a larger surface area than the same mass of material, this can be make materials more chemically reactive, and the second reason, is below 50 nm, the laws of classical physics different from these of the same material at a larger scale (Dowling, 2004). Nano-materials have wide-ranging applications including physics, chemistry, electronics, optics, material science, the biomedical sciences and textile finishing.

Ag nanoparticles can be synthesized using various methods: chemical, electrochemical (Vorobyova, Lesnikovich, & Sobal, 1999), γ-radiation (Choi, Zhang, Gopalan, Lee, & Kang, 2005)

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doi:10.1016/j.carbpol.2012.01.038

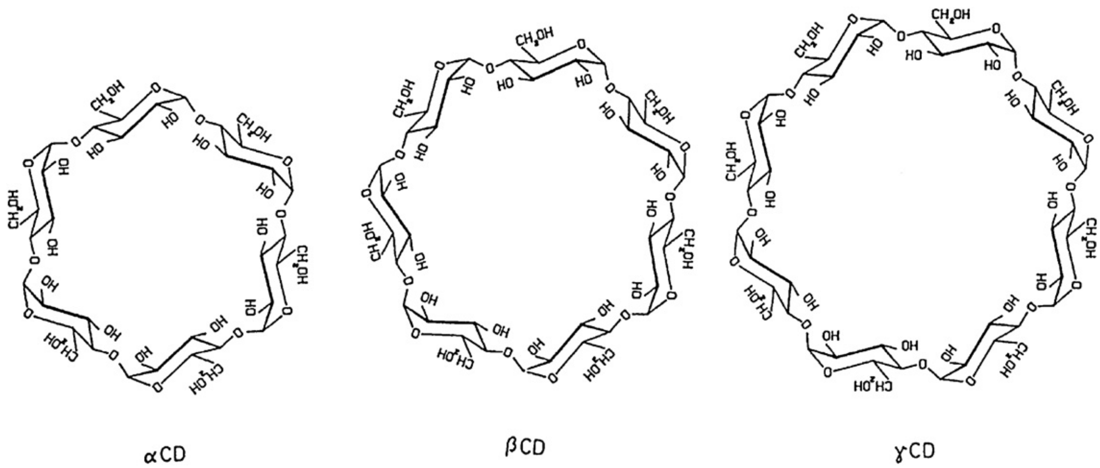

Fig. 1. structure of cyclodextrins.

photochemical (Li, Li, Qian, Yin, & Zhu, 2005), laser ablation (Tsuji, Watanabe, & Tsuji, 2003), etc. The most popular preparation of Ag colloids is chemical reduction of silver salts by sodium borohydride or sodium citrate. This preparation is simple, but the great care must be exercised to make stable and reproducible colloid. The purity of water and reagents, cleanliness of the glassware are critical parameters. Solution temperature, concentrations of the metal salt and reducing agent, reaction time influences particle size.

For imparting anti-bacterial properties, nano-sized silver, titanium dioxide and zinc oxide are used (El-Shafie & Abou-Okeil, 2011; Xin, Daoud, & Kong, 2004; Xiong, Gu, You, & Wu, 2003; Yang, Zhu, & Pan, 2004). Silver nanoparticles have an extremely large relative surface area, thus increasing their contact with bacteria or fungi, and improving their bacteria and fungicidal effectiveness. Nano-silver is very reactive with proteins. When contacting bacteria and fungus, it will adversely affect cellular metabolism and inhibit cell growth of those bacteria and fungi which cause infection, odor, itchiness and sores. Hence nano-silver particles are widely applied to socks and healthcare products in order to prohibit the growth of bacteria.

The present work was undertaken with a view to study the major factors affecting the amination of β-cyclodextrin, the work was extended for using the so-obtained aminated β-cyclodextrin derivative as reducing and stabilizing agent for the preparation of silver nanoparticles.

2. Materials and methods

2.1. Materials

β-Cyclodextrin (β-CD) was supplied by Wacker–Chemie GmbH, Munich, Germany. 2-Chloroethyl amine ($Cl–C_2H_4–NH_2$) was supplied by Merck Schuchardt OHG, Hohenbrunn, Germany. Silver nitrate ($AgNO_3$) was obtained from Sisco Research Laboratories PVT. LTD., Mumbai, India. All other reagents were of laboratory grade.

2.2. Synthesis of aminated β-cyclodextrin

$$\beta\text{-CD} + Cl–C_2H_4–NH_2 \xrightarrow{NaOH} \beta\text{-CD–O–}C_2H_4–NH_2 + NaCl + H_2O$$

β-Cyclodextrin (β-CD) was first soaked in aqueous solution of 2-chloroethylamine (5–25%) (on weight of β-CD) for 30 min, and dried at 65 °C till solvent (water) was evaporated. The product was immersed in NaOH solution (5–25%) concentrations for 15–90 min at different temperatures (50–90 °C). The aminated β-cyclodextrin was obtained by precipitated in methyl alcohol. The aminated β-CD washed several times and dried at 65 °C for 10 min. The extent of reaction was expressed as nitrogen content.

2.3. Preparation of silver nanoparticles

Aminated β-cyclodextrins having different nitrogen content were dissolved in distilled water. After complete dissolution, the pH of the solution was adjusted within the range pH 7–12, followed by raising the temperature to the desired temperature (30–70 °C). At the end, certain amount of silver nitrate (to give 0.1 M $AgNO_3$ in 100 ml total volume) solution was added drop wise (keeping in mind that the total volume of the reactant is 100 ml). The reaction mixture was kept under continuous stirring for different durations (5–60 min). The resulting solution acquired a clear yellow in color indicating the formation of silver nanoparticles.

2.4. Testing and analysis

2.4.1. Nitrogen content (N%)

Nitrogen content was determined according to the Kjeldahl method (Vogel, 1975).

2.4.2. FTIR

FTIR spectra were recorded using FTIR Raman model Nexus 670 (Nicollet-Madison, WI).

2.4.3. UV–Vis spectra (UV–Vis)

Ultra violet–visible spectra have been proved to be quite sensitive to the formation of silver colloids because silver nanoparticles exhibit in intense absorption peak due to the surface Plasmon excitation. UV–Vis spectrum of Silver nanoparticles was recorded on Perkin Elmer Lambda 3B UV-Vis spectrometer.

2.4.4. Transmission electron microscope (TEM)

TEM was measured using Zeiss-EM10-Germany.

3. Results and discussion

3.1. Effect of different parameters on synthesis of aminated β-cyclodextrin

3.1.1. Effect of 2-chloroethylamine (CEA) concentration

Modification of β-cyclodextrin with CEA was highly affected by the concentration of 2-chloroethylamine. CEA concentrations

Table 1
Effect of CEA concentration on the amination of β-cyclodextrin.

CEA concentration (%)	Nitrogen content (N%)
5	0.27
10	0.28
15	0.28
20	0.50
25	0.63

Reaction conditions: NaOH, 15%; temperature, 70 °C; 30 min.

Table 2
Effect of NaOH concentration on the amination of β-cyclodextrin.

NaOH concentration (%)	Nitrogen content (N%)
5	0.63
10	0.64
15	0.50
20	0.44
25	0.28

Reaction conditions: CEA, 20%; temperature, 70 °C; 30 min.

in the range (5–25%) based on the weight of β-cyclodextrin were used. Table 1 shows the effect of the concentration of CEA concentration in the range studied on the extent of amination of β-cyclodextrin (expressed as N%). Obviously the extent of amination increases significantly by increasing CEA concentration from 5% to 25%. Increasing of amination with increasing the concentration of CEA could be associated with increasing the functional groups present in CEA which has the ability to react with the hydroxyl groups of β-cyclodextrin.

3.1.2. *Effect of NaOH concentration*

β-Cyclodextrin was subjected to the reaction with CEA at different NaOH concentrations in the range (5–25%). The effect of NaOH concentration on the N% (extent of amination) is shown in Table 2. It is seen from Table 2 that the amination extent, expressed as N%, relies on the NaOH concentration. The effect of NaOH concentrations on the extent of amination follows the following order: 5–10% > 15% > 20% > 25%. It can also be seen from Table 2 that increasing of the NaOH concentration has a decremental effect on the extent of amination, which can be explained by increasing the decomposition effect of the high concentrated NaOH on the formed aminated β-cyclodextrin as shown by the following equation

$$\beta\text{-CD–O–}C_2H_4\text{–}NH_2 \xrightarrow[H_2O\Delta]{NaOH} \beta\text{-CD–O–H} + HO\text{–}C_2H_4\text{–}NH_2.$$

3.1.3. *Effect of reaction temperature*

Table 3 shows the effect of amination temperature on the extent of amination of β-cyclodextrin. The reaction of CEA with β-cyclodextrin was performed at temperature range from 50 °C to 90 °C. It is clear that increasing the reaction temperature from 50 °C to 80 °C leads to increasing in the N% (extent of amination), further increase in the reaction temperature over 80 °C has an adverse effect on the N%. Evidently amination displays its maximal at 70–80 °C; this finding signifies that by increasing the temperature, the ability of the CEA to reach the OH groups of β-cyclodextrin increases and the extent of reaction increases. Reaction temperature above 80 °C may lead to a partial deamination of the formed aminated β-cyclodextrin.

Table 3
Effect of reaction temperature on the amination of β-cyclodextrin.

Temperature (°C)	Nitrogen content (N%)
50	0.32
60	0.35
70	0.50
80	0.54
90	0.36

Reaction conditions: NaOH, 15%; CEA, 20%; 30 min.

Table 4
Effect of reaction time on the amination of β-cyclodextrin.

Time (min)	Nitrogen content (N%)
15	0.37
30	0.50
45	0.57
90	0.23

Reaction conditions: NaOH, 15%; temperature, 70 °C; 30 min.

3.1.4. *Effect of the reaction time*

The effect of reaction time on the amination of β-cyclodextrin by CEA was investigated. The amination was performed at 70 °C, 20% CEA, and 15% NaOH, for time ranging from 15 to 90 min. Table 4 shows the N% of the formed aminated β-cyclodextrin samples as a function of time. It is clear (Table 4) that the extent and rate of amination, expressed as N% exhibit an initial fast rate by increasing the reaction time from 15 to 45 min followed by slower rate by increasing the reaction time to 90 min. The favorable effect of the reaction time could be associated with longer contact between β-cyclodextrin and CEA. Decreasing the N% at reaction time longer than 60 min can be attributed to the effect of high temperature at high contact time, which may partially decompose the produced aminated β-cyclodextrin.

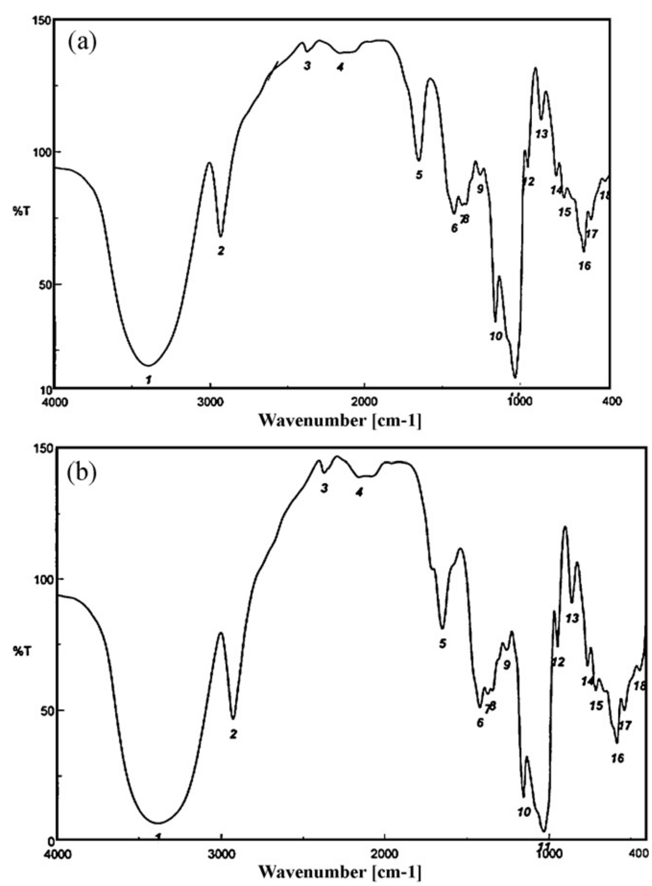

Fig. 2. FTIR spectra of (a) β-cyclodextrin, (b) aminated-β-cyclodextrin.

3.1.5. IR spectra of β-cyclodextrin and aminated β-cyclodextrin

Fig. 2a and b shows the IR spectra β-cyclodextrin and aminated β-cyclodextrin. It can be seen that the characteristic absorption peak of OH group of β-cyclodextrin is located at 3396.99 cm^{-1}. This peak becomes wider and broader and shifts from 3396.99 cm^{-1} to 3387.02 cm^{-1} in the case of aminated β-cyclodextrin. This can be attributed to the presence of absorption band of NH_2 stretching at 3300 cm^{-1} that overlaps with the absorption of OH group (in the range of 3200–3500 cm^{-1}).

4. Preparation of Ag nanoparticles using aminated β-cyclodextrin as reducing and stabilizing agent

Aminated β-cyclodextrin was prepared using the following conditions: NaOH, 15%; temperature, 70 °C; for 90 min. This sample which has 0.23 N% was used along all the study except in case of studying the effect of N% on the preparation of Ag nanoparticles, different conditions were used to give aminated β-cyclodextrin with N%, 0.44, 0.54, and 0.63 (Tables 1–4). Fig. 3a–e shows the effect of different factors on the preparation of Ag nanoparticles using aminated β-cyclodextrin.

4.1. Effect of pH on the preparation of Ag nanoparticles using aminated β-cyclodextrin

Ag nanoparticles were prepared using 0.6% aminated β-cyclodextrin, 70 °C, 10 min at different pH values (7, 9, and 12) the pH value was adjusted using NaOH or H_2SO_4. Fig. 3a shows the UV–Vis spectrum of Ag nanoparticles prepared at different pH values. It can be seen from Fig. 3a that pH has a very important effect on the preparation of Ag nanoparticles using aminated β-cyclodextrin as reducing and stabilizing agent. Only preparing Ag

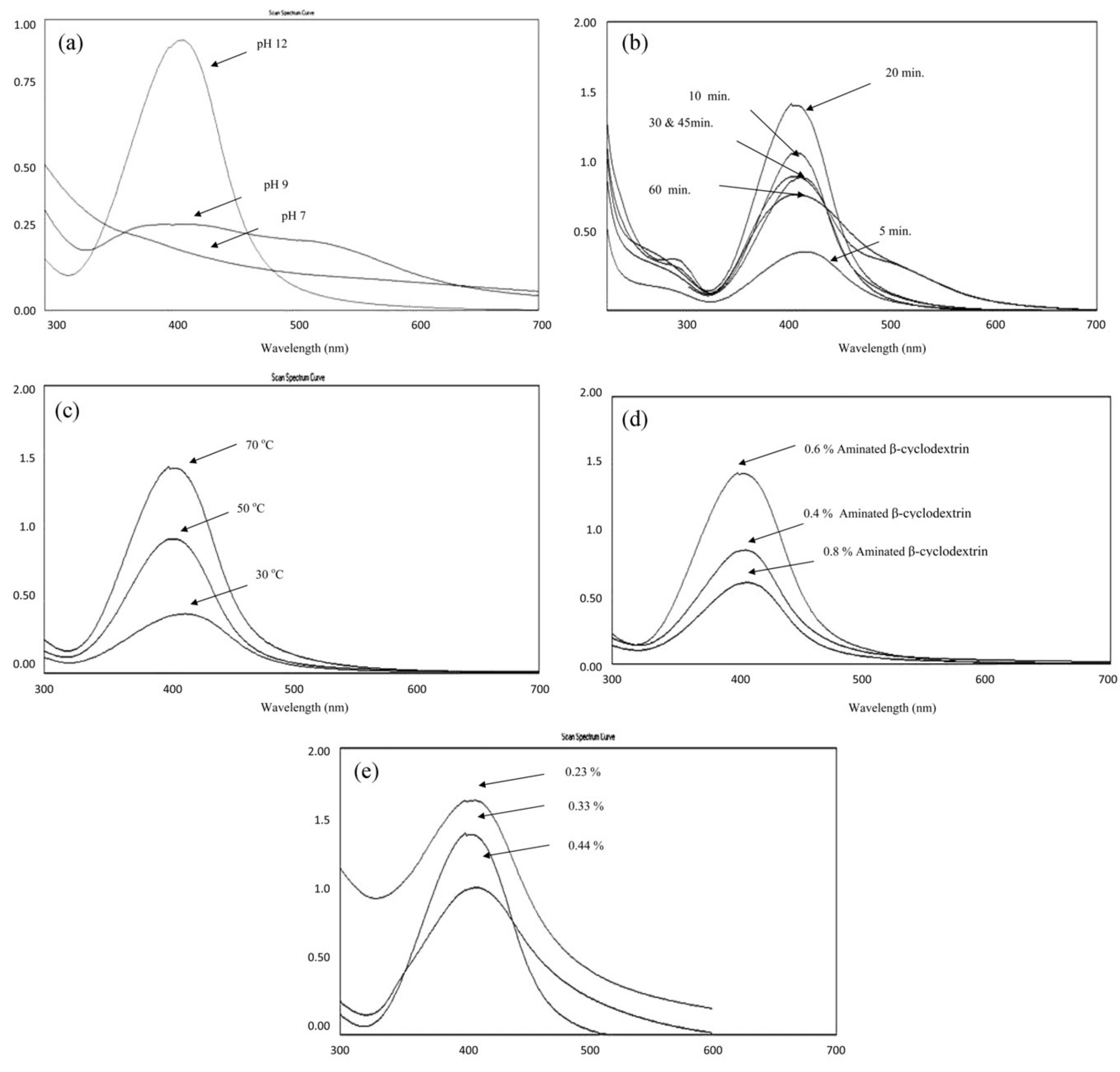

Fig. 3. UV–Vis spectrum of Ag nanoparticles (a) effect of pH, (b) effect of time, (c) effect of temperature, (d) effect of concentration of aminated-β-cyclodextrin, (e) effect of N%.

nanoparticles at pH 12 has clear and sharp surface Plasmon band at around 411 nm which is characteristic for Ag nanoparticles. Preparation of Ag nanoparticles at pH 7 has no characteristic band while at pH 9 there is a very broad band around 400 nm. These results indicate that the preparation of Ag nanoparticles needs to take place at higher pH value which can be attributed to the ability of NaOH to increase the rate of reducing reaction of Ag+ ions to Ag (Wang, Qiao, Chen, & Ding, 2005).

4.2. Effect of time on the preparation of Ag nanoparticles using aminated β-cyclodextrin

Fig. 3b shows UV–Vis spectrum of Ag nanoparticles prepared using, 0.6% aminated β-cyclodextrin, pH 12, 70 °C at different time intervals. Fig. 3b depicts that Ag nanoparticles surface Plasmon band occurs at around 410 nm for all time intervals (only a few nm differences between all time intervals). The intensity of the absorbance increases steadily as function of time and reaches its maximum at 20 min. The intensity starts to decrease by increasing the time of preparation over 20 min. These results can be explained by increasing the concentration of Ag nanoparticles along with the ability of aminated β-cyclodextrin to stabilize the formed Ag nanoparticles at the beginning (higher intensity of absorbance). Prolonging the preparation time over 20 min gives a higher concentration of Ag nanoparticles which accompanied by aggregation as a result of inability of aminated β-cyclodextrin to stabilize this higher quantity of Ag nanoparticles (lower intensity of absorbance) supporting polydispersity of Ag nanoparticles.

4.3. Effect of temperature on the preparation of Ag nanoparticles using aminated β-cyclodextrin

Fig. 3c shows the UV–Vis spectrum of Ag nanoparticles prepared by using, 0.6% aminated β-cyclodextrin, pH 12, and 20 min at different temperature (30 °C, 50 °C, and 70 °C). As shown by Fig. 3c increasing temperature is accompanied by increasing the intensity of the absorbance at around 410 nm. Increasing temperature may affect the ability of adsorption of Ag on the surface of aminated β-cyclodextrin and facilitate the approach of Ag to the surface of aminated β-cyclodextrin as well as increasing the accessibility of the OH and NH_2. This leads to a very good stabilizing behavior of aminated β-cyclodextrin and increasing the concentration of Ag nanoparticles which reflects on the intensity of the Plasmon band.

4.4. Effect of concentration of aminated β-cyclodextrin on the preparation of Ag nanoparticles

Fig. 3d gives an idea about the effect of the concentration of aminated β-cyclodextrin in the range studied (0.4%, 0.6% and 0.8%) on the UV–Vis spectrum of Ag nanoparticles prepared by the following conditions: different amount of aminated β-cyclodextrin, pH 12, and 20 min 70 °C. As shown by Fig. 3d, increasing N% of aminated β-cyclodextrin from 0.4% to 0.8% has an effect on the intensity of the surface Plasmon band; this effect can be divided into two stages. First stage is by increasing the concentration of aminated β-cyclodextrin from 0.4% to 0.6%, this increase of concentration of aminated β-cyclodextrin is drastically accompanied by increasing in the intensity absorbance from 0.857 to 1.435 at around 410 nm, which can be explained by increasing the concentration of Ag nanoparticles. The second stage is by increasing the concentration of aminated β-cyclodextrin from 0.6% to 0.8% which has an adverse effect on the intensity of absorbance, the intensity of the absorbance decreased from 1.435 to 0.615. Increasing the concentration of β-cyclodextrin to a concentration higher than 0.8%, leads to a sudden aggregation. This finding can be understood by the inability of aminated β-cyclodextrin to stabilize Ag nanoparticles at high concentration.

4.5. Effect of N% of aminated β-cyclodextrin on the preparation of Ag nanoparticles

Fig. 3e shows the UV–Vis spectrum of Ag nanoparticles as a function of N% of aminated β-cyclodextrin. Ag nanoparticles were prepared by the following conditions: 0.6% of aminated β-cyclodextrin, pH 12, and 20 min at 70 °C. In this study different samples of aminated β-cyclodextrin having different N% (0.23, 0.33, and 0.44) were used. The effect of N% on the surface Plasmon band was almost the same as the effect of the concentration of aminated β-cyclodextrin. We have also 2 stages, the first one is by increasing N% from 0.23 to 0.33 which is accompanied by decreasing in the intensity of absorbance from 1.655 to 1.435 at around 410 nm. This effect is due to the increasing of Ag nanoparticles concentration. The second stage is by increasing N% to 0.44 which gives a drastic decrease in the intensity (1.075). Increasing the N% over 0.44

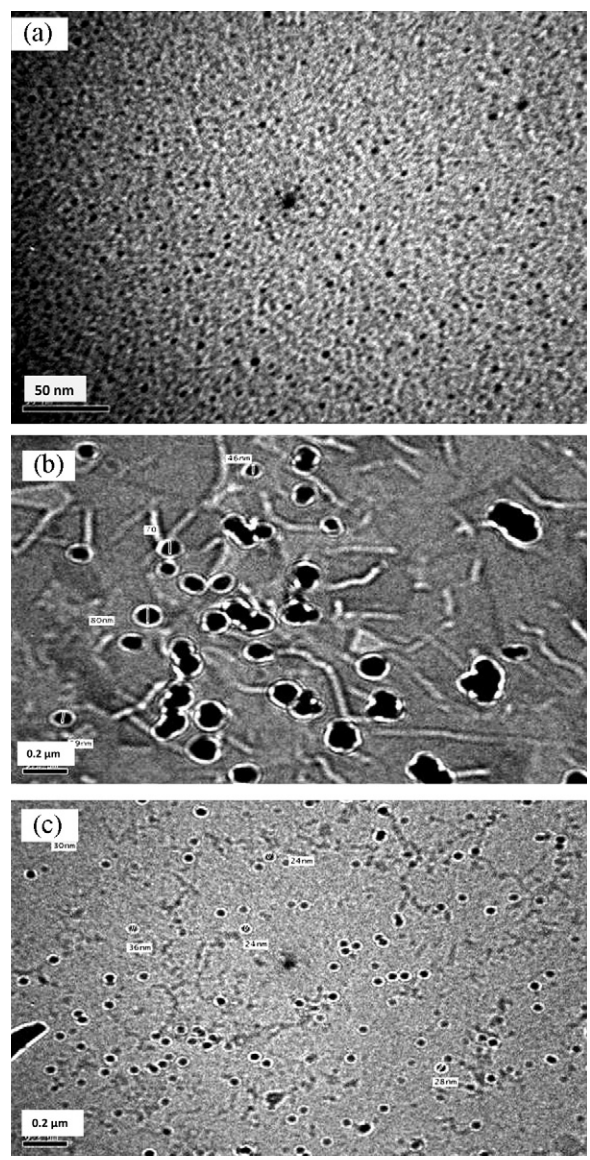

Fig. 4. TEM of Ag nanoparticles prepared by (a) 0.6% of aminated β-cyclodextrin, pH 12, and 20 min at 70 °C, (b) 0.6% of aminated β-cyclodextrin, pH 9, and 20 min at 70 °C, (c) 0.6% of aminated β-cyclodextrin, pH 12, and 20 min at 30 °C.

induces a very rapid precipitation of Ag nanoparticles. These results support the results of the effect of aminated β-cyclodextrin concentration which is the inability of aminated β-cyclodextrin to stabilize Ag nanoparticles at higher concentration of Ag nanoparticles.

4.6. TEM of Ag nanoparticles prepared using aminated β-cyclodextrin

Fig. 4a–c gives an idea about the TEM of Ag nanoparticles prepared using aminated β-cyclodextrin as reducing and stabilizing agent. Fig. 4a and b shows the TEM image of Ag nanoparticles prepared at pH 12 and 9 respectively, It can be indicated from Fig. 4a and b that the shape and size of Ag nanoparticles are significantly affected by the pH of the reaction medium. The shape of Ag nanoparticles prepared at pH 12 seems to be spherical and there is no appearance to any aggregations on contrary to the shape of Ag nanoparticles prepared at pH 9, the shape of nanoparticles is not spherical and there are some aggregations appeared. Also the particle size of the Ag nanoparticles prepared at pH 12 is smaller than that prepared at pH 9; the particle size at pH 12 was in the range of 1–4 nm while at pH 9 the particle size was in the range 40–80 nm. Fig. 4 shows also the TEM of Ag nanoparticles prepared at 50 °C (Fig. 4c); Comparison between Fig. 4a and c gives an idea about the effect of temperature on the particle shape and size. It is clear from Fig. 4a and c that the temperature play an important role in the shape and particle size of Ag nanoparticles, as show from Fig. 4a and c the particle size decreased with increasing the temperature from 50 °C to 70 °C, also Fig. 4c shows a certain aggregation at 50 °C. All the data in Fig. 4 are in accordance with the results of UV–Vis spectrum.

5. Conclusion

Aminated β-cyclodextrin was prepared through the reaction of 2-chloroethylamine with β-cyclodextrin. The preparation was carried out under different conditions (time, temperature, concentration of NaOH, and concentration of 2-chloroethylamine). The aminated β-cyclodextrin was used as reducing and stabilizing agent for the preparation of silver nanoparticles from $AgNO_3$. From the results obtained it can be concluded that the extent in amination of β-cyclodextrin increases significantly by increasing CEA concentration from 5% to 25%. The effect of NaOH concentrations on the extent of amination follows the following order: 5–10% > 15% > 20% > 25% NaOH. Increasing the reaction temperature from 50 °C to 80 °C leads to increasing in the N% (extent of amination). Increasing the reaction time from 15 to 45 min leads to increasing in the rate of amination. Only preparing Ag nanoparticles at pH 12 has clear and sharp surface Plasmon band at around 411 nm which is characteristic for Ag nanoparticles. Plasmon band occurs at around 410 nm for all time intervals (only a few nm differences between all time intervals). The intensity of the absorbance increases steadily as function of time and reaches its maximum at 20 min increasing temperature is accompanied by increasing the intensity of the absorbance at around 410 nm. Increasing N% of aminated β-cyclodextrin from 0.4% to 0.8% has an effect on the intensity of the absorbance and also increasing the concentration of aminated β-cyclodextrin has an important effect on the absorbance intensity.

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