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Studies on absorption spectra of uniform gold nanoparticles prepared in Triton X-100 reverse micelles

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Abstract

A procedure for preparation of uniform gold nanoparticles in the ternary Triton X-100/water/cyclohexane system in the presence of sodium sulfite is suggested. The formation and stabilization of nanoparticles in Triton X-100-based reverse micelles in the above ternary system and in the quaternary system (additionally containing *n*-hexanol) have been explored. The mechanism for formation of Au(0) and growth of gold clusters was found to depend on the initial concentration of $HAuCl_4$ and Na_2SO_3. The mean size of nanoparticles formed in reverse micelles in the ternary system was markedly smaller (less 5 nm) than of those formed in the quaternary system (from several to dozens nanometers).

Keywords: Optical absorption spectra; Reverse micelles; Gold nanoparticles; Preparation; Stabilization; Triton X-100

1. Introduction

Interest in studies on nano-sized particles stems from their potential use as new smart materials in various fields of modern science and technology. Of especial interest is the preparation of metal nanoparticles (NPs), in particular gold NPs, with the mean size of several nanometers. The most popular method for preparation of such particles is the reduction of hydrogen tetrachloroaurate(III) trihydrate ($HAuCl_4 \cdot 3H_2O$) into gold metal with such reducing agents as hydrazine, sodium tetrahydroborate, etc. Stable gold colloids were first prepared long ago by Faraday (using phosphorus as a reducer) [1] and then by Zsigmondy (reduction with hydrogen peroxide and formaldehyde) [2]. High stability of such systems is ensured by the presence of additives – such as sodium citrate [3,4], thiols [5–9], phosphines [10], Triton X-100 (TX, octylphenol-polyethyleneglycol ether) [11–15], dendrimers [16,17], chitosan [18], polyvinyl pyrrolidone [4,19], polyethylene glycol [20], etc. – that passivate the surface of gold particles and prevent from physical contact between the particles. Some of these compounds (e.g. sodium citrate and TX) can also act as reducing agents.

Gold NPs can also be prepared through radiochemical or photochemical reduction of $HAuCl_4$. Gold particles 1.5–2.5 nm in size are known to form upon radiolysis of $HAuCl_4$ in aqueous solutions containing polyvinyl alcohol or polyvinyl pyrrolidone [19]. Metal NPs were also formed upon UV irradiation of $HAuCl_4$ solutions containing poly(amidoamine) dendrimers [16]. The photochemical reduction of $HAuCl_4$ in aqueous solutions containing TX was reported in Refs. [11–14]. In Ref. [15], the synthesis of gold NPs in TX aqueous micellar solutions was carried out (without irradiation) in the presence of dopamine hydrochloride as a reducing agent and TX as a stabilizer.

A promising method for preparation of metal NPs is their synthesis in reverse micelles (RMs). The size of metal NPs formed in the micellar water pool can be expected to be confined by the pool size, while the shell of surfactant molecules ensures their stabilization [21]. But in reality, the mean size of gold particles prepared in the RMs based on cetyltrimethylammonium bromide [22,23] or sodium bis-(2-ethylhexyl)sulfosuccinate (AOT) was found [24–26] to be markedly larger than the diameter of micellar pool. Therefore, some additional measures are seemingly needed in order to ensure a small size of synthesized particles along with their narrow size distribution. For example, addition of dodecanethiol to an RM solution led [23] to formation of stable gold NPs about 5 nm in size.

According to [25], a low stability of gold NPs in the AOT-based RMs can be associated with their oxidation in micellar pools. The stability of such micellar solutions was found to increase either upon addition of surface-passivating agents or after removal of dissolved oxygen. It was also found that the use

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of the TX-based RMs (hereinafter TX-RMs) led to a marked decrease in the mean particle size, increase in their yield, and rise in the stability of loaded RM solutions (compared to AOT-based ones).

In this paper, we report the data of our recent studies on the mechanism for formation and stabilization of gold NPs in TX-based reverse micelles.

2. Experimental

RMs were prepared from TX, $HAuCl_4$, dodecanethiol, and *n*-hexanol (all reagent grade, as purchased from Sigma), double-distilled water, and chromatographically purified *n*-hexane (or cyclohexane). Other reagents (Na_2SO_3, $N_2H_4{\cdot}H_2SO_4$) were of reagent grade and used without further purification. RM solutions were prepared as described elsewhere [25,27]. In the absence of *n*-hexanol (as a co-surfactant), the mole ratio $W = [H_2O]/[\text{surfactant}]$ was 1:1 (hydrodynamic radius of micelles 30.5 nm [27]). Gold NPs were prepared by mixing two RM solutions, one containing $HAuCl_4$ in the aqueous phase while another, either reducing agent or Na_2SO_3.

Concentrations of water-soluble components in RMs are given for the aqueous phase. Formation of gold NPs was monitored by absorption spectroscopy (Shimadzu UV-3101PC spectrophotometer and/or Ocean Optics HR-2000 spectrometer). Samples for IR spectroscopy were prepared just before measuring by placing a drop of micellar solution between two zinc selenide glass plates. IR spectra were taken with an Infra LUM FT-02 spectrometer. Samples for electron microscopy (transmission electron microscope JEM-100CX) were additionally stabilized with dodecanethiol. Stabilized NPs were precipitated from RM solutions by centrifuging. A precipitate was dispersed in cyclohexane, and the suspension was then deposited onto a copper grid covered with carbon.

3. Results and discussion

Colloidal gold NPs exhibit a well-pronounced band of plasmon absorption in the range 500–550 nm. Location and width of this absorption band are known to depend on the size/shape of particles [28,29]. For a wide range of Au particles (from subnano-sized clusters to particles up to several dozen nanometers in diameter), the size dependence of absorption spectra has been well confirmed by both theoretical and experimental studies [5,30]. The effect of environment on position of the plasmon band was reported in Ref. [31].

According to [25], the mean size of gold nanoparticles synthesized in TX-RMs in the presence of sodium sulfite is markedly smaller than that of gold particles synthesized in AOT-based RMs. Stable TX-RMs are normally formed in the presence of co-surfactant [32] (e.g. *n*-hexanol).

Fig. 1 presents the absorption spectra of TX/*n*-hexanol/water/*n*-hexane mixture ($W = 2$, $[HAuCl_4] = 0.02$ M) containing sodium sulfite as taken in 3, 60, 120 min and 23 h after preparation of micellar solution. It follows that the feeble band belonging to gold NPs (peaked around 515–520 nm) begins to manifest itself in 60 min after mixing starting solutions (curve 2). With time, the mixture became turbid, which was followed by precipitation of dark deposit. This is indicative of system instability. With increasing *W* and/or concentration of starting reagents, destabilization of microemulsion became faster.

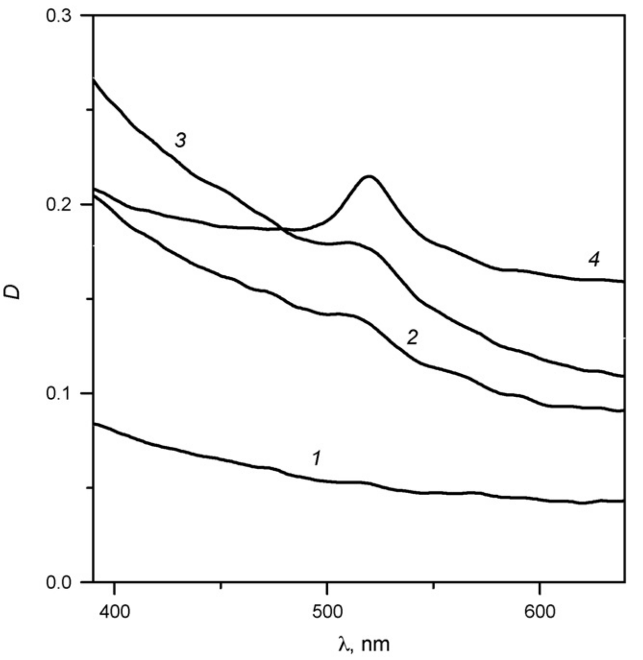

Fig. 1. The absorption spectra of TX/*n*-hexanol/water/*n*-hexane mixture ($W = 2$, $[HAuCl_4] = 0.02$ M, $[Na_2SO_3] = 0.24$ M, $[TX] = 0.34$ M, $[TX]/[n\text{-hexanol}] = 1{:}4.7$) as taken in 3 min (1), 60 min (2), 120 min (3) and 23 h (4) after preparation of micellar solution.

The use of cyclohexane as a dispersion medium afforded the preparation (at low values of *W*) of TX-RMs in the absence of co-surfactant [27]. Such micellar solutions exist only within a very narrow range of *W* and also are thermodynamically unstable. At room temperature and for $W \leq 1$, such RMs remains stable only within a few hours. Nevertheless, it was this system in which stable gold NPs with an extremely small size have been recently synthesized [25].

In a few hours, the TX-RM solutions containing gold NPs became turbid and gradually separated into two layers: a transparent upper layer rose in color (typical of gold NPs) and a slightly turbid colorless underlayer. The separation rate grows with decreasing temperature. Upon stirring, the solution became rose and transparent again. All this was not accompanied by any broadening and red shift of the absorption band, thus suggesting that no coarsening of gold particles took place.

The absorption spectra of gold NPs formed in TX-RMs in the presence of Na_2SO_3 (curve 1) and $N_2H_4{\cdot}H_2SO_4$ (curve 2) and taken (at other equal conditions) in 30 min after mixing are presented in Fig. 2. Curve 1 is seen to exhibit a narrow absorption band peaked at 514 nm whose intensity is greater by a factor of ten compared to that in curve 2. Such absorption is typical of uniform gold particles about 5 nm in their size [4], which is also confirmed by the data of TEM (Fig. 3a). In case of hydrazine sulfate as a reducing agent (Fig. 2, curve 2), the plasmon absorption of gold particles represents a broad

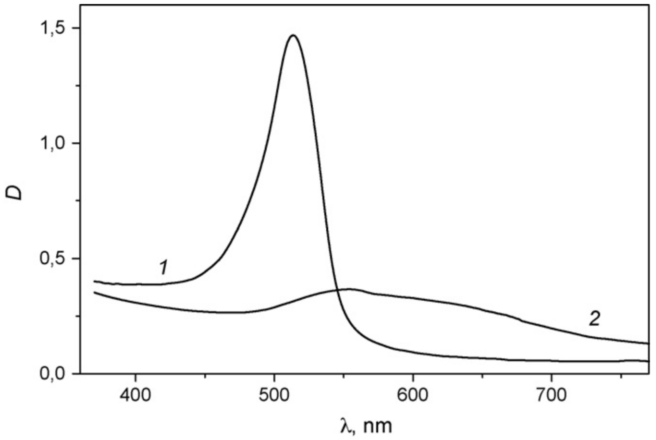

Fig. 2. The absorption spectra of gold NPs obtained in TX/water/cyclohexane mixtures ([$HAuCl_4$] = 0.02 M) in the presence of Na_2SO_3 (1) and $N_2H_4 \cdot H_2SO_4$ (2) as taken in 30 min after mixing the starting solutions.

band in the range 500–700 nm. The TEM image of the sample (Fig. 3b) shows the presence of particles from several to dozens nanometers in their size.

Similar results were obtained for TX-RMs with $HAuCl_4$ in the absence of sodium sulfite and any other reducing agent (Fig. 4). The broad band with a maximum at 540 nm (curve 3) is seen to appear in 24 h after mixing the starting solutions, which is indicative of a broad size distribution of gold NPs formed in the system. Therefore, it can be inferred that, in the presence of sodium sulfite, the yield of NPs increases by a factor of several times, the mean particle size decreases, and the stability markedly increases even in the absence of stabilizing agents.

Position of the plasmon band was also found to depend on [$HAuCl_4$] and [Na_2SO_3]. The higher is [$HAuCl_4$]$_0$, the greater are the yield of gold NPs and red shift of the plasmon band [25], that is, the larger gold NPs are formed.

As follows from Fig. 5, the action of Na_2SO_3 is most pronounced at the initial stages of NPs formation. At low [Na_2SO_3], the plasmon band initially peaked at 522 nm undergoes a grad-

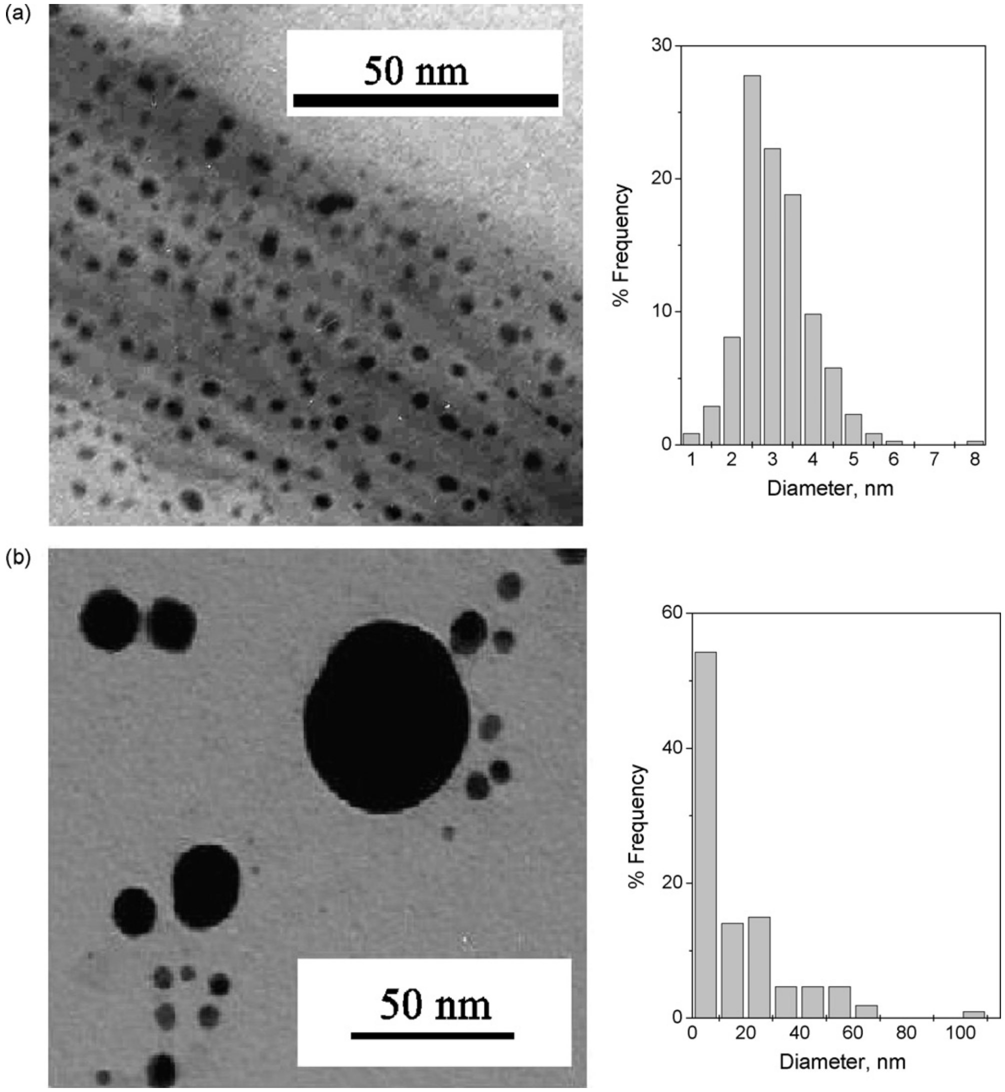

Fig. 3. TEM images and size distribution of gold nanoparticles prepared in conditions corresponding to curve 1 (a) and curve 2 (b) in Fig. 2. Respective histograms were plotted for an ensemble of 300–400 particles in several micrographs of the same sample.

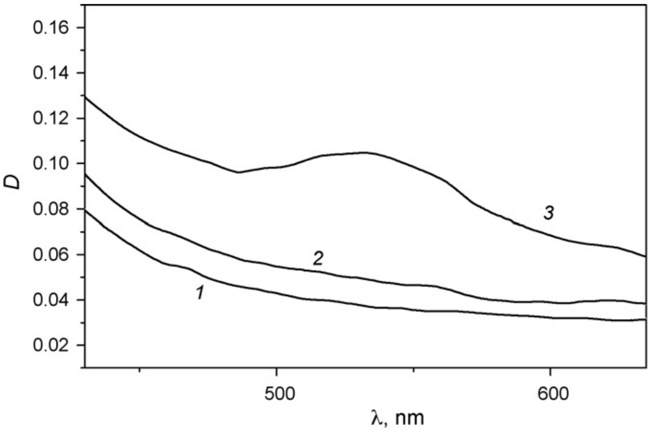

Fig. 4. The absorption spectra of TX/water/cyclohexane mixture ($[HAuCl_4] = 0.02$ M) in the absence of Na_2SO_3 and other reducing agents as taken in 1–3 min (1), 280 min (2), and 24 h (3) after mixing the starting solutions.

ual blue shift and then (in $t = 20$ min) levels off at 510 nm (curve 1). On going too higher $[Na_2SO_3]$, the effect becomes opposite (curves 2 and 3): the initial value of $\lambda_{max} = 503$ nm gradually shifts to longer wavelengths and then levels off around 510–512 nm.

As is known, nano-sized gold aggregates may undergo oxidation under the action of dissolved atmospheric oxygen. Sodium sulfite is known to bind oxygen and thus to suppress oxidation [25]. In addition, this compound may react with $HAuCl_4$ to give sodium aurate which (at pH 7–13) gradually transforms into $[Au(OH)_4]^-$ [33]. According to [34], the reaction of $HAuCl_4$ with sodium sulfite is accompanied be conversion of Au(III) to Au(I) resulting in formation of the relatively stable colorless complex as shown below:

$$2HAuCl_4 + 5Na_2SO_3 + 2H_2O = 2Na_3[Au(SO_3)_2] + 2NaCl + 6HCl + Na_2SO_4 \tag{1}$$

The $SO_3{}^{-2}$ ions are known to exhibit nucleophilic properties and thus to compete with OH^- [35]. In this context, the $SO_3{}^{-2}$ ions present in water pools of TX-RMs act as carriers of charge to TX molecules, due to which the latter ones acquire the properties of gold reducer in the following reaction:

$$\underset{\text{I}}{\text{R–OCH}_2\text{CH}_2\text{OH}} + \text{Au(I)} \rightarrow \text{Au(0)} + \underset{\text{II}}{\text{R–OCH}_2\text{CHO}} + 2\text{H}^+, \tag{2}$$

where $R = (CH_3)_3CCH_2C(CH_3)_2C_6H_4(OCH_2CH_2)_9$.

According to [12,14], formation of gold NPs in $HAuCl_4$ solutions proceeds in two stages. One involves the formation of very small gold clusters which then act as seeds for subsequent aggregation. At the second stage, the nucleation stops, and NPs begin to grow on these seeds. This process takes place due to adsorption and subsequent reduction of ions on the surface of clusters which catalyze the growth of NPs. The size of resultant NPs depends on the size of seeds and on the number of ions reduced on their surface [36]. The smaller size of starting clusters, the higher probability for formation of NPs with a minimal size and narrow size distribution.

The size of NPs in RMs is controlled by the size of micellar pools, while their growth is governed by the process of intermicellar exchange. Collisions between micelles give rise to exchange by molecules and ions, so that the properties of micellar shells are of utmost importance. In the presence of co-surfactant molecules, the mobility and permeability of micellar shells are known to increase [37], which makes possible the exchange not only by individual molecules but also by their aggregates [38,39]. In this case, the formation of clusters will be accompanied by their aggregation, which will accelerate the consumption of seeds and, as a result, lead to a broad size distribution of resultant NPs. Accordingly, all this will manifest itself in appearance of a broad plasmon band in the absorption spectra.

The micellar shells formed by TX molecules contain a large number of oxyethylene groups which are capable of binding gold-containing ions [20]. In the presence of sodium sulfite, charged TX molecules can reduce Au(I) to Au(0). In reaction (2), the molecules of TX I are oxidized to compound II in which the alcohol group is replaced by carbonyl. In the IR spectrum of the sample top layer (Fig. 6, curve 1), the band at 1735 cm^{-1} (C=O

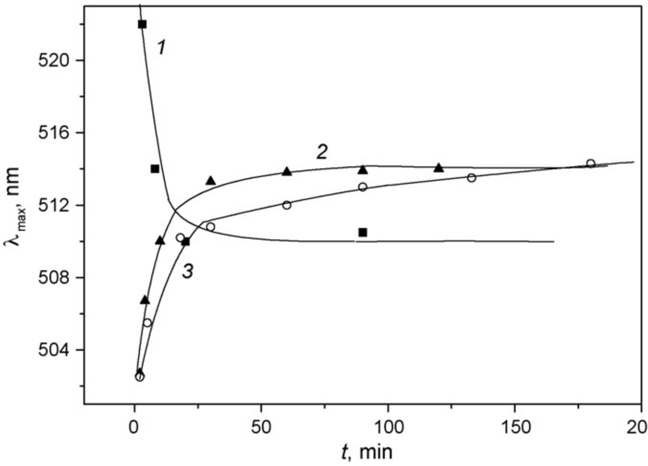

Fig. 5. Position of λ_{max} for the plasmon band as a function of time t for $[Na_2SO_3] = 0.12$ M (1), 0.24 M (2), and 0.72 M (3); $[HAuCl_4] = 0.02$ M.

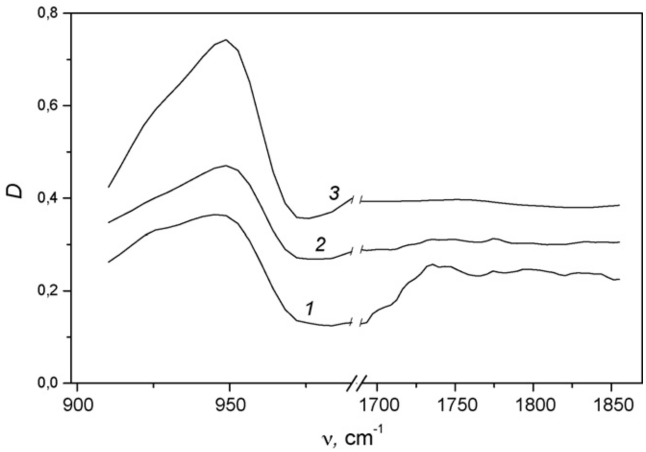

Fig. 6. The IR spectra of the top layer (1), underlayer (2) of delaminate TX/water/cyclohexane mixture, and of pure TX (3).

stretching mode) can be regarded as an evidence for formation of compound II since such a band is absent in the spectrum of pure TX (curve 3) [17]. Reduced Au atoms are assembled into clusters and located amidst oxyethylene groups of surfactant molecules, i.e. in the micellar shells rather than in micellar water pools. In the presence of sodium sulfite in water pools, gold NPs could be expected to convert into $[Au(OH)_4]^-$. However, our data show that the obtained Au particles are very stable (and also to oxidation). Therefore, TX molecules not only form micellar shells but also act as reducing agents and stabilizers for gold NPs.

Keeping in mind that pH of micellar water pools depends on the $[HAuCl_4]/[Na_2SO_3]$ ratio, the hypsochromic shift of λ_{max} observed for low $[Na_2SO_3]$ (Fig. 5, curve 1) can be explained as follows. Gold ions are distributed between the water pools and shells of micelles (where they are captured by oxyethyl groups of TX). At low $[Na_2SO_3]$, the medium of water pools becomes acidic, which is a necessary condition for reduction of gold ions into Au(0) under the action of sodium sulfite. In this case, the reduction of gold is pH-dependent in the aqueous phase and pH-independent within the shells. In these conditions, gold ions in water pools are rapidly reduced with sodium sulfite. Apparently, such a process was reported in Ref. [26] where $HAuCl_4$ was reduced with K_2SO_3 in AOT-RMs. As a result, the size of gold NPs is restricted by the pool diameter, while gold ions in the aqueous phase are completely consumed. Further formation of gold NPs takes place in the micellar shells due to slow reduction of captured ions with TX (see reaction scheme (2)) yielding small NPs exhibiting plasmon absorption at shorter wavelengths (as compared to NPs localized in water pools). Gradual accumulation of small NPs eventually makes them predominant, which gives rise to an apparent blue shift of their absorption.

At high $[Na_2SO_3]$, the role of sodium sulfite is only confined to the transfer of charge to TX molecules. In this case, the reduction of gold ions takes place largely within the micellar shells to give gradually accumulating small gold NPs. This process manifests itself as a 'normal' red shift of the plasmon absorption (Fig. 5, curves 2 and 3).

Surfactant molecules are present in TX-RM solutions either as well-ordered aggregates in micellar shells or as monomers in dispersion medium, both in dynamic equilibrium [27]. It can be assumed that, when gold particles are formed in micellar shells, some surfactant molecules bound with NPs are present in their oxidized form II (see reaction scheme (2)). Since the interaction between hydroxy groups is much stronger than that between carbonyl groups, hydroxyl-substituted molecules can form their associates in solution while carbonyl-containing compounds are present as individual molecules. As-prepared RM solutions remain transparent and homogeneous for over several hours, and during this period surfactant molecules undergo exchange between micellar shells and cyclohexane. Since carbonyl groups of oxidized TX molecules only weakly interact with hydroxyl groups of the neighboring TX molecules and with the micellar water pool, they are gradually displaced by TX molecules. Gold NPs stabilized with several $R–OCH_2CHO$ molecules turn out in the organic phase where their further aggregation occurs very slowly. These aggregates remain in the suspended state in the top layer for a long time. Bicontinuous structures consisting of disordered or locally ordered particles of water and cyclohexane separated by surfactant molecules are probably formed in the underlayer [40]. The gold NPs from the underlayer are unstable and undergo rapid aggregation followed by precipitation of aggregates to the cell bottom. This is indirectly confirmed by the following observations: (1) after shaking the delaminated microemulsion, the mean size of gold NPs remained unchanged and (2) the rate of thermodynamic separation of TX-RM solutions grew with increasing $[HAuCl_4]$.

Table 1
Position (ν_{OH}) and full width at half maximum (FWHM) of absorption bands assigned to the stretching mode of the OH group in the oxyethylene chain of TX molecules

Phase	ν_{OH} (cm^{-1})	FWHM, (cm^{-1})
Top layer	3474	225
Underlayer	3484	264
Triton X-100	3477	234

Additional information about the top layer and underlayer of delaminated TX-RM solutions can be inferred from the IR data presented in Table 1 and Fig. 6.

According to [41], the IR band around 3480 cm^{-1} belongs to the terminal OH group in the oxyethylene chain of TX molecules. As follows from Table 1, the absorption band of the underlayer formed in delaminated RM solution is broader and shifted to higher ν in comparison with the top layer and pure Triton X-100. According to [42], interaction between oxygen atom from the oxyethylene chain and water gives rise to the absorption band at 3506 cm^{-1} which can overlap the band peaked at 3480 cm^{-1}; as a result, this leads to broadening of the latter and its shift to higher ν. Since the ν_{OH} values for the top layer and pure TX (containing no water) are close, it can be concluded that the top layer contains no water.

Another support for such an interpretation can be deduced from Fig. 6 which shows a fragment of IR spectrum (around 949 cm^{-1}) for the three samples of Table 1. According to [43], this band belong to the coiled conformation GTTG (here T stands for *trans* while G, for *gauche*) of the oxyethylene chain in a TX molecule. Curves 1 and 3 in Fig. 6 exhibit a shoulder 925 cm^{-1} which is absent in curve 2. The shoulder was attributed [43] to another conformation (GGTG and probably GGGG) which appears due to some disordering of the oxyethylene chain. It has been found [42] that the shoulder at 925 cm^{-1} is most pronounced in the absence of water and disappears when even trace amounts of water are present. Therefore, the above IR data clearly demonstrate that the top layer of delaminated TX-RM solutions contains gold NPs stabilized by surfactant molecules and contains no water.

4. Summary

The gold NPs formed in ternary TX/water/cyclohexane mixtures in the absence of co-surfactant (*n*-hexanol) are markedly smaller and more homogeneous in their size than those synthesized in quaternary TX/*n*-hexanol/water/*n*-hexane mixtures.

This is because in the former case gold NPs are generated in micellar shells where, upon attaining some certain size, they are stabilized by TX molecules in the organic phase. In the presence of Na_2SO_3 in the ternary mixtures, gold NPs can be prepared even without using an inert atmosphere. In the quaternary mixtures, the formation and growth of gold NPs proceed in micellar water pools, while the co-surfactant molecules present in micellar shells promote the aggregation of NPs.

Acknowledgments

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