Research Article

Fusiform gold nanoparticles by pulsed plasma in liquid method

Saadat Sulaimankulova[1] · Altynai Mametova[2] · Zhypargul Abdullaeva[2]

Abstract

We have synthesized fusiform gold nanoparticles by recovering gold from a solution of gold nanoparticles obtained by dispersing of gold electrodes in hexane by pulsed plasm in liquid method. Formation of fusiform gold nanoparticles with length from 50 to 150 nm, and diameter of 5–15 nm was achieved by pulsed plasma in liquid method. Fusiform gold nanoparticles obtained by our method are different from the spindle-shaped gold nanoparticles which were synthesized from the growth solution, due to their loose structure, while spindle-shaped gold nanoparticles are dense and much larger in size (length 300–400 nm, diameter ~ 100–150 nm). In addition to this work, dumbbell-shaped magnetite/gold (Fe_3O_4/Au) nanoparticles were synthesized by using magnetite nanoparticles obtained in a cetylpyridinium bromide (CpyBr) solution with concentration of 0.1% and a gold nanosolution, where 0.1% sodium citrate solution was applied as a reducing agent. Our proposed method is characterized by the forming of discharge localization during the electrodes erosion process, simpler and does not require use of nuclei in the form of spherical gold nanoparticles and presence of growth solution. Fusiform gold nanoparticles were formed by simple reduction with sodium citrate from gold nano-solution.

Keywords Fusiform · Gold nanoparticles · Magnetite · Synthesis · Pulsed plasma · Nano-solution

1 Introduction

Gold nanoparticles have been attracting big attention due to their extensive application in many fields such as chemistry, physics, material science, electronics, catalysis and bionanotechnology [32]. Medical applications of gold nanoparticles have been tested in biomedicine [20] and surgery [18], in vitro biosensing, in vivo imaging, drug delivery, and tissue engineering [1, 24], therapy and imaging [3]. Other applications of gold nanoparticles are including sensing [11] and antibacterial [34] were also reported recently.

Various methods such as green synthesis oxidation [2, 9, 19, 27, 43], chemical reduction [17, 23, 50], bacterial synthesis [4, 7, 35], fungal synthesis [42], laser ablation [8, 16, 48], CVD method [25, 31, 40, 41, 46], solvothermal [6, 14], halogen-free synthesis [22, 44] and seed-mediated synthesis [26, 36] have been used for the synthesis of gold nanoparticles with different size, shapes and properties depending on the synthesis method and experimental conditions, as shown in the Table 1.

Gold nanoparticles (AuNPs) were synthesized at room temperature by a simple, rapid, and green route using fresh squeezed apple juice as a reducing reagent, where morphology of obtained AuNPs was based on the particle color, stability, and color change suitable for colorimetric detection of cysteine (Cys), are synthesized using 5 mL of 10% apple juice, 1 mL of 10 mM gold precursor solution, and 1 mL of 0.1 M NaOH [10].

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Table 1 Various methods used for synthesis of gold nanoparticles with various size

Preparation method	Au NP size (nm)	References
Green synthesis oxidation	15	Kunoh et al. [8]
Chemical reduction	1–3.5	Yeh et al. [9]
Bacterial (*R. capsulata*)	50–400	Abdullaeva [10]
Laser ablation	7	Amendola et al. [11]
CVD method	5–100	Takagi et al. [12]
Solvothermal method	7–15	Ahmad et al. [13]
Halogen-free synthesis	< 10	Sashuk et al. [14]
Seed-mediated synthesis	18–100	Kumar et al. [15]

Anisotropic gold nanoparticles (GNPs) and nanostructures have been produced [49]; including triangles, stars and rods which are represented in the Fig. 1 below. Gold nanoparticles with fusiform shape have been recently fabricated by controlling the volume of the growth solution with shape evolutions ranging from fusiform nanoparticles to 1-D rods [13]. The term *fusiform* can be referred to a spindle-shaped form of nanostructures with wide area in the center. Previously spindle-shaped gold nanoparticles were successfully prepared in large amount by using a simple wet chemical approach method, where L-ascorbic acid (AA) used as reductant in the presence of cetyltrimethylammonium bromide (CTAB) at room temperature [29].

In this article we are reporting the synthesis of fusiform gold nanoparticles by using the impulse plasma in a liquid method, used for the synthesis of metal oxide, carbon and carbon coated metal nanoparticles [5, 37, 38]. Pulsed plasma in a liquid method is based on a dispersion phenomenon of interfacing metal electrodes under vibration. Characteristic of this discharge form is the localization of the erosion process. A single pulse has an extremely short duration (10^{-3}–10^{-5} s), a high current density (10^{6}–10^{8} A/cm^2) in the impact zone, and very high temperature in the discharge channel (10^{4}–10^{5} K) and a pressure of 3^{-10} kbar. Drawback of this method is sometimes appearing in separation of synthesized nanoparticles, due to various phases and morphology of nanoparticles.

Gold nanoparticles prepared by modified citrate reduction method under sonication exhibited uniform spherical shapes, which was shown by ultraviolet (UV)–Visible absorption spectrum of the two final products represented in the Fig. 2, because the absorption peak was centered between 520 and 540 nm [28].

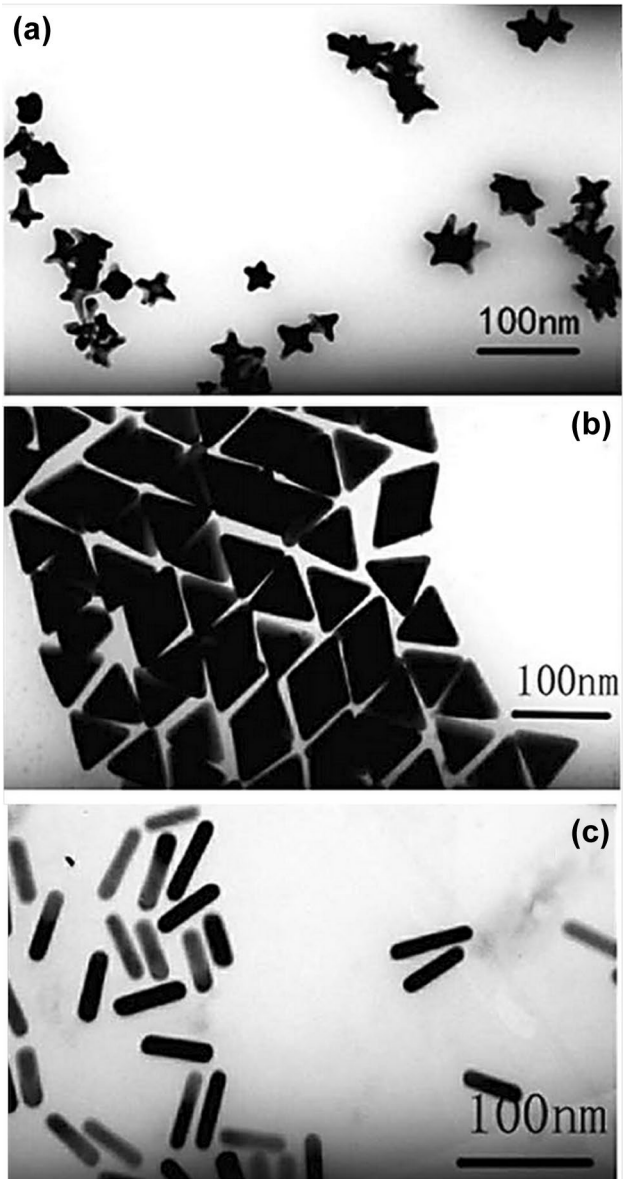

Fig. 1 Anisotropic gold nanoparticles with different shapes in the form of stars, triangles and rods. Reproduced with permission from Xie et al. [49]

2 Experimental method

For the synthesis of fusiform gold nanoparticles, we have applied pulsed plasma which was generated between two gold metal rod shaped electrodes submerged in hexane solution. Energy of the simple impulse was appeid with electrical energy of 0.05 J, capacity of the condensator was 4 µFs (microfarads). Schematic of the experimental setup was shown in the Fig. 3. During the pulsed plasma in liquid physical effects caused by electrical field can be explained by the Cauchy's equation of motion and continuity [30]:

$$\rho \frac{Dv}{Dt} = \rho f - \nabla P + \tau \quad (1)$$

$$\frac{\partial \rho}{\partial t} = \nabla \cdot (\rho v) \quad (2)$$

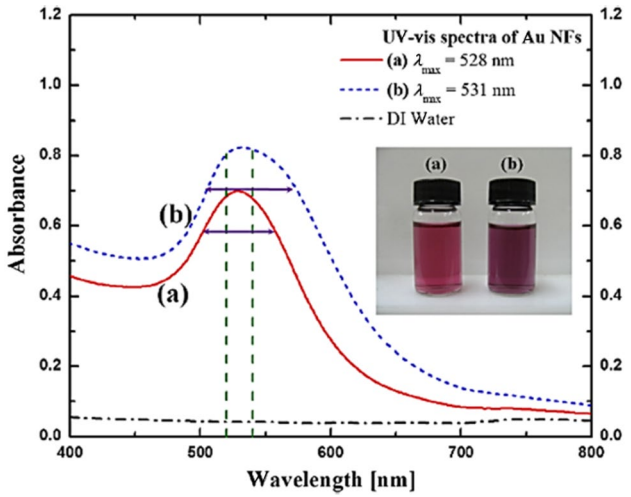

Fig. 2 UV–Visible spectroscopy of synthesized gold suspensions. (a) Gold suspension (maximum absorption peak λ_{max} is 528 nm) prepared with 243 kJ (30 min with 135 W ultrasonic power) of sonication; (b) gold suspension (maximum absorption peak λ_{max} is 531 nm) prepared with 729 kJ (90 min with 135 W ultrasonic power) of sonication. Reproduced from Lee et al. [22] under permission license CC-BY2.0

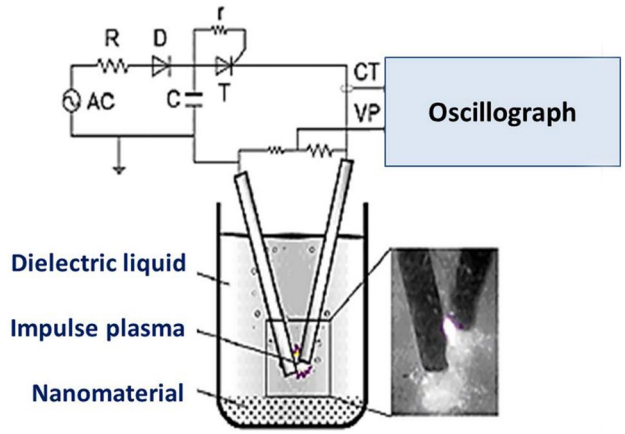

Fig. 3 Schematic of the experimental setup for synthesis of fusiform gold nanoparticles by pulsed plasma in liquid method

here, Eq. (1) is the motion, and Eq. (2) is the continuity equations respectively; where, *v*, and mass density, ρ, in a fluid as functions of the pressure—P, body forces—*f*, and viscous stress—τ.

Pulsed plasma processes in the presence of organic liquids such as hexane may cause the introduction of reactive species (e.g., OH– and H– radicals) into organic compounds through spatial and temporal control of the plasma discharge [47]. Recently, in-liquid plasma methods can be broadly classified into four categories, depending on the electrode configurations and energy sources, such as: (1) *Plasma A*: direct discharge between two electrodes using an AC (plus) power supply; (2) *Plasma B*: contact discharge between an electrode and the surface of the surrounding electrolyte using a DC power supply; (3) *Plasma C*: plasma generated with either radio frequency (RF) or microwave (MW) irradiation; (4) *Plasma D:* plasma generated using the laser ablation technique [21].

In our case, a single impulse discharge was generated between two gold electrodes immersed in a liquid medium (hexane). Pulsed plasma in dielectric liquid resulted from the breakdown of the inter-electrode space with a high potential difference between the electrodes and a relatively small source power, which is insufficient to initiate an arc discharge.

The energy of a single pulse (0.05 J) was regulated by changing the power of capacitors *C* in an electrical circuit. The energy value was selected as a result of numerous experiments conducted by the authors of this work in collaboration with colleagues for many years. Selected energy of a single pulse is sufficient to disperse any even the most refractory conductive material with the formation of nanostructures [45]. During synthesis of fusiform gold nanoparticles electrodes of pure gold (99.99%) were adjusted inside hexane solution and pulsed discharge was applied. Obtained gold nanoparticles were placed in a conical flask and exposed to aqua regia for 20 min. As a result, a 1% gold–yellow–orange solution was obtained. After that, 1 ml of 1% sodium citrate solution was added to 1 ml of the resulting solution. Discoloration of the initial solution was observed. Precipitate was centrifuged at 2000 rpm for 30 min, and then separated from the medium by decantation and filtration. Dried powders were subjected to physicochemical analyses.

2.1 Reagents and materials

Gold metal rod electrodes with purity of 99.99%, diameter of 2 mm and length of 10–15 mm were used. Hydrochloric acid (HCl) with purity of 99.9% and nitric acid (HNO_3) with concentration of 58.9%, hexane solution with concengtration of 97%, sodium citrate in water solution of 5.5% were used for experiments.

2.2 Characterization of gold NPs

X-ray diffraction (XRD) measurement was performed on a Rigaku RINT-2500HV diffractometer with Cu-Kα radiation wavelength of 0.15406 nm. Tube voltage was applied as 40 kV, and the current was 200 mA. XRD patterns were taken in the range of (2θ) 20°–80°. Transmission electron

microscopy (TEM) images of as synthesized fusiform gold nanoparticles were taken using a JEOL 1400 microscope with Specification of maximum accelerating voltage 120 kV, maximum resolution of 0.2 nm installed in the Nanobiomedcenter of the Adam Mickiewicz University, Poznań, Poland.

3 Results and discussions

3.1 Transmission electron microscopy analyses

Transmission electron microscopy (TEM) analysis of resulting fusiform gold nanoparticles was carried out by dropping fusiform gold nanoparticles solution on the copper grid and adjusting the sample holder into the TEM microscope. TEM image of the sample clearly shows TEM images of fusiform gold nanoparticles synthesized by pulsed plasma in a liquid method: (a, d) rough and soft fusiform gold nanoparticles; (b, c) aggregates of fusiform gold nanoparticles in Fig. 4. Fusiform gold nanoparticles have a length of from 50 to 150 nm and diameter of 5 to 15 nm. The fusiform gold nanoparticles obtained by pulsed plasma in liquid method are different from the spindle-shaped nanoparticles synthesized by the growth solution [18]: our nanoparticles are loose, while those obtained in a growth solution are dense and much larger in size (length 300–400 nm, diameter ~ 100–150 nm). Our proposed method is simpler and does not require the use of germs in the form of spherical gold nanoparticles and growth solution. Fusiform gold nanoparticles formed by simple reduction with sodium citrate from gold nano-solution.

3.2 X-ray diffraction analysis

XRD characterization of fusiform gold nanoparticles was taken from dried powder samples revealed that the gold nanoparticles synthesized by pulsed plasma in a liquid method existing predominantly in a face-centered-cubic (FCC) phase, with cell parameters of a = 0.4078 nm and

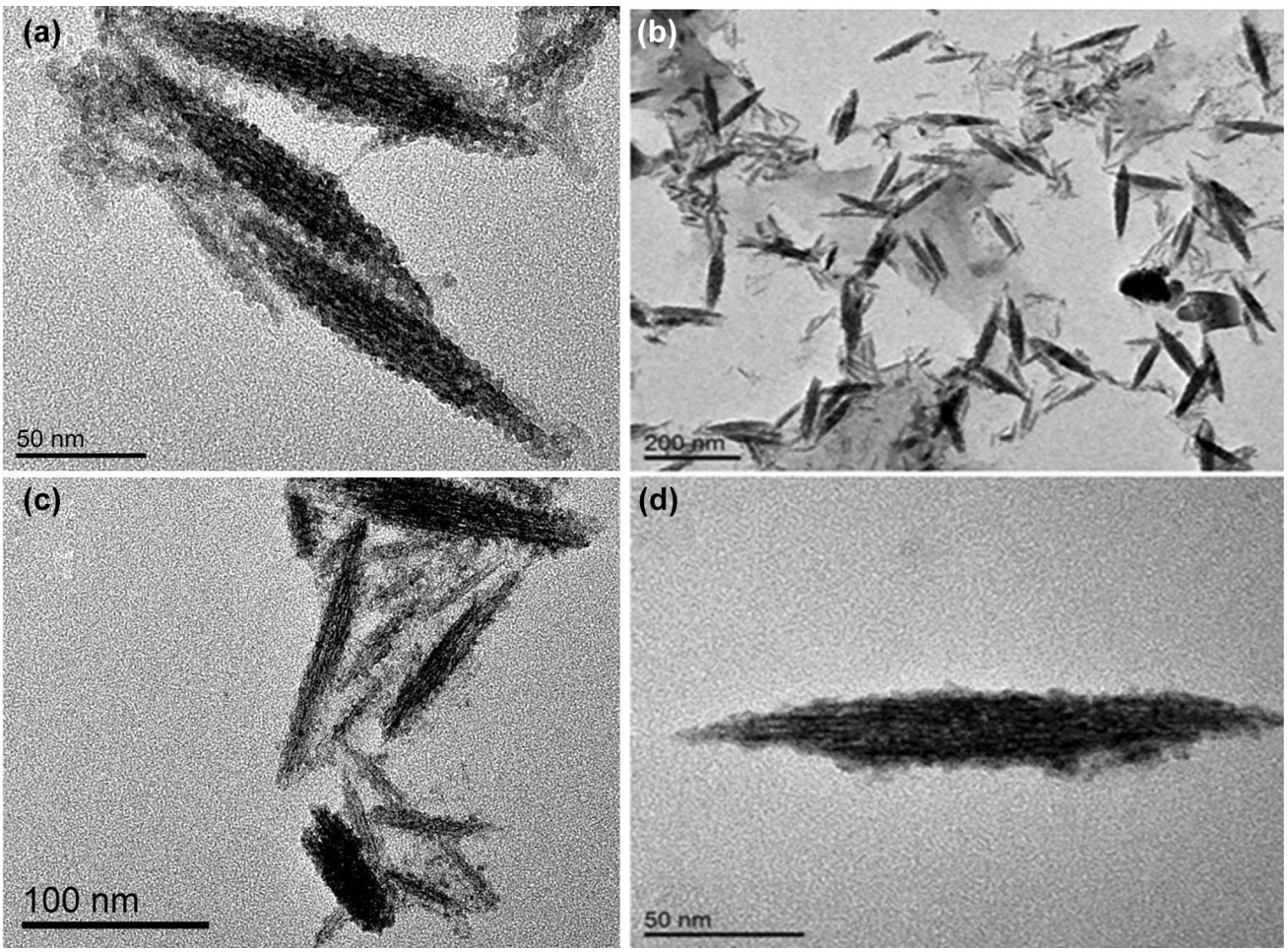

Fig. 4 TEM images of fusiform gold nanoparticles synthesized by pulsed plasma in a liquid method: **a**, **d** rough and soft fusiform gold nanoparticles; **b**, **c** aggregates of fusiform gold nanoparticles

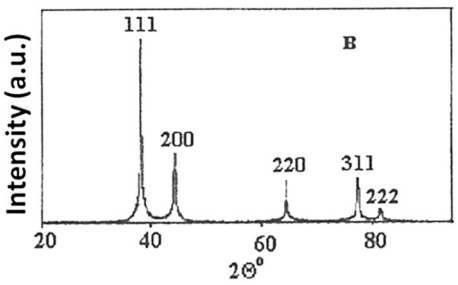

Fig. 5 XRD pattern of fusiform gold nanoparticles synthesized by pulsed plasma in a liquid method

average size of gold nanoparticles equal to 2.5 nm. As it is shown in the Fig. 5, presence of intense peaks corresponding to 2θ values of (111), (200), (220), (311) and (222) in the powder X-ray diffraction pattern agrees with those values reported in literature (JCPDS No. 04-0784), as well as previously reported spindle shaped gold nanoparticles [12].

Supplementary Table 2 is showing interplanar *d* spacing for fusiform gold nanoparticles which were calculated by using the following equation:

$$\frac{1}{d^2} = \frac{h^2 + k^2 + l^2}{a^2} \tag{3}$$

According to these calculations the interplanar *d* spacings for fusiform gold nanoparticles were as 2.36182 for (111), 2.04570 for (200), 1.44497 for (220) and 1.23268 for (311), respectively.

In addition to this work, we have synthesized Fe_3O_4/Au nanoparticles by using magnetite [33] nanoparticles which were obtained in a cetylpyridinium bromide solution of 0.1% CPyBr and a gold nanosolution. Here 0.1% sodium citrate solution was applied as a reducing agent [51]. Figure 6 shows TEM image of samples obtained by treating 0.1 g of magnetite nanoparticles with a solution of sodium citrate, then with a gold nanosolution. Here adsorption of cationic CPyBr on the surface of freshly formed magnetite nanoparticles was carried out in order to stabilize them, prevent oxidation and transition to Fe_2O_3 due to hydrophobization of the nanoparticles surface. Dumbbell-shaped Fe_3O_4/Au nanoparticles were found as shown by arrows in the Fig. 6a, b.

Biomedical applications of such spindle shaped gold nanoparticles have been previously reported [39]. Cetrimonium bromide $[(C_{16}H_{33})N(CH_3)_3]Br$ (CTAB) forms rod-like micelles in the solution above its critical micelle concentration and form bilayers on the surface of gold nanorods, which is resulting in a stable dispersion of gold nanorods. Nanorods then capped with a bilayer of surfactant CTAB have positive charge. However, CTAB is known for its cytotoxicity [15]; hence, it is necessary to mask the CTAB layer for biomedical applications. The CTAB-coated gold nanorods were further covered with poly(sodium 4-styrenesulfonate (PSS) by electrostatic interactions to obtain the negatively charged gold nanorods. ζ-potential analysis of gold nanorods was performed before and after PSS coating. CTAB-stabilized gold nanorods showed a positive charge on the surface due to the presence of quaternary amine hydrophilic head groups from adsorbed CTAB, whereas PSS-capped gold nanorods showed a negative charge on the surface due to the presence of anionic $-SO_3$ groups. Thus, ζ-potential analysis confirmed successful coating of PSS on the surface of CTAB-capped gold nanorods. This surface modification of gold nanorods was found to be very effective in reducing the cytotoxicity of gold nanorods caused by an excess of CTAB and formed a stable dispersion of gold nanorods for further attachment of biomolecules. The bioconjugation of gold nanorods and the detection of IgG are represented schematically

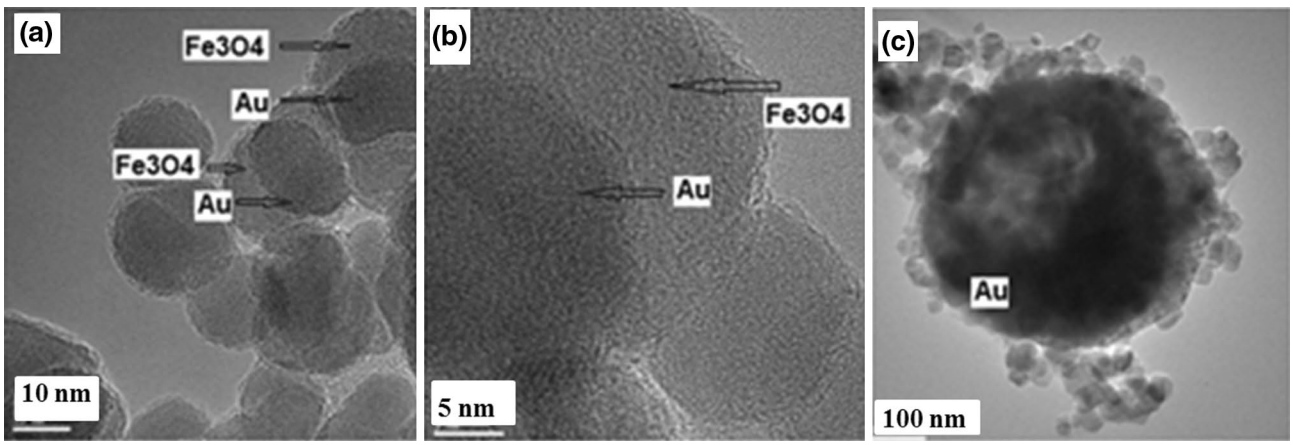

Fig. 6 Dumbbell-shaped Fe_3O_4/Au nanoparticles obtained from magnetite using the pulsed plasma in liquid

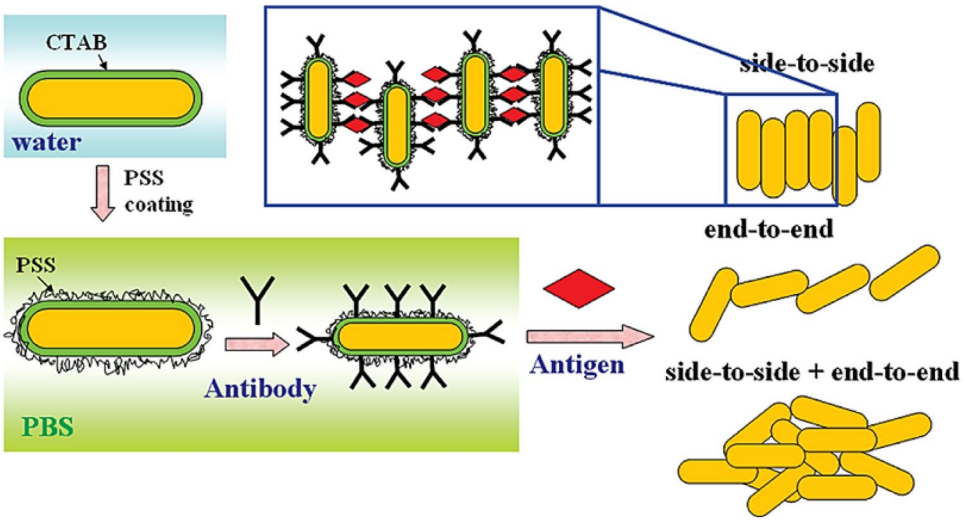

Fig. 7 Schematic representation of bioconjugation of gold nanorods and the detection of g-IgG through the aggregation of gold nanorods. Reproduced with permission from Parab et al. [39] Copyright (2009) American Chemical Society)

in Fig. 7 (reproduced with permission from Parab et al. [39] Copyright (2009) American Chemical Society).

4 Conclusions

Fusiform gold nanoparticles were successfully synthesized by using the pulsed plasm in a liquid method. Pulsed plasma in dielectric liquid resulted from the breakdown of the inter-electrode space with a high potential difference between the electrodes and a relatively small source power, which is insufficient to initiate an arc discharge. A single pulse has an extremely short duration (10^{-3}–10^{-5} s), a high current density (10^{6}–10^{8} A/cm^2), a very high temperature in the discharge channel (10^{4}–10^{5} K), and a pressure of 3^{-10} kbar and spreads in the amount of 10^{-3}–10^{-4} cm^3, i.e. characterized by strong localization effects on the solid. TEM observations of as-synthesized sample clearly showed formation of fusiform gold nanoparticles with a length of 50 to 150 nm and diameter of 5 to 15 nm. As properties, morphology and synthesis of fusiform gold nanoparticles were not researched well enough, our work is original and valuable contribution to the synthesis of fusiform gold nanoparticles.

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Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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