

A green chemical approach for synthesis of shape anisotropic gold nanoparticles

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Abstract A complete green chemical reaction between aurochloric acid and tea polyphenols resulted in the reduction of $\text{Au}^{3+} \rightarrow \text{Au}^0$. The reaction was carried out in a Teflon-coated bomb digestion vessel at 200 °C. It was observed that with increasing the reaction time from 1 to 5 h, the shape of the nanoparticles changed from spherical to rod-like structures. The reaction was followed with the help of UV–vis spectrometer, which showed a single absorption peak at 548 nm for 1-h reaction product and two peaks for a 5-h reaction product at 533 and 745 nm corresponding to the transverse and longitudinal surface plasmon resonance bands. Microstructures obtained from transmission electron microscope revealed that the samples obtained after 1-h reaction are predominantly spherical in shape with an average size of 15 nm. Whereas samples obtained after 5 h of reaction exhibited rod-like structures with an average size of 45 nm.

Keywords Gold nanorods · Green chemical synthesis · Tea polyphenols · UV–vis spectroscopy

Introduction

Synthesis of gold nanoparticles (Au NPs) has drawn the attention of researchers because of their extensive

applications in the development of new technologies in areas such as chemistry, catalysis, electronics, medicine and biotechnology [1–3]. As both size and shape of Au NPs affect optical and electronic properties, a number of methods have been reported for the preparation of Au NPs with varying sizes and shapes [4–6]. The surface electrons of Au NPs exhibit a phenomenal behavior of collective oscillation and absorption of light, which is known as surface plasmon resonance (SPR). The remarkable shape-dependent optical property of gold nanorods and spheres is a typical example, wherein gold nanorods exhibit two absorption peaks arising from the transverse and longitudinal surface plasmon resonances (SPRs), whereas the spherical particles display only a single SPR peak [4, 5]. Au NPs can be synthesized by conventional chemical and physical methods [7–9]. Many of the synthetic routes for development of nanomaterials use toxic reagents that make them unsuitable for biological use and also have adverse effect on the environment. Furthermore, these methods are expensive as they require high energy, long time and sophisticated equipments to carry out the reactions. These concerns over several chemical and physical synthetic techniques have resulted in attempts to develop biological approaches. Therefore, development of green processes for the synthesis of NPs has evolved into an important branch of nanotechnology. Green nanotechnology uses biomolecules present either in plant extracts or microorganism as novel reducing and capping agents. They not only suffice as environmentally friendly routes but also as economically sustainable alternatives to chemical and physical methods.

Recently, many biosynthetic routes have been reported for synthesis of Au NPs through micro-organisms such as algae, fungi and bacteria. However, due to their cumbersome procedures such as maintaining cell cultures under specified laboratory conditions have made them less

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attractive, when compared to using plant products for synthesis of nanomaterials. Many plants or plant products have been employed for the synthesis of Au NPs, some of which include, *Emblia officinalis*, *Acacia nilotica* (Babool), alfalfa biomass, geranium, *Azadirachta indica* leaves, gum kondagogu, gram-beans and tea leaves [10–17]. *Camellia sinensis* commonly known as tea is a rich source of polyphenolic compounds, which exhibits a variety of health benefits such as protection against heart diseases, cancer and the paralytic actions of botulinum neurotoxins [18, 19]. These attractive health benefits apart from its complete green approach have turned tea extract as an important species for synthesis of NPs. However, tea extract has been utilized only for the synthesis of spherical NPs with SPR bands in the visible region. To enhance its biocompatibility, the SPR band needs to be tuned in such a way that it lies close to the NIR region, where biological tissues tend to absorb minimally. Therefore, synthesis of Au NPs with rod-like structures using green processes becomes crucial. Many reports have earlier been published to control the shape of Au NPs to form nanorods, but they require multi-pronged and cumbersome experimental procedures such as seed-mediated growth [20–22]. In this work, we report a novel, simple, single-stage selective biosynthesis route for production of Au NPs in the form of spheroids and rods. To obtain the desired shape of Au NPs, aqueous chloroaurate ions were reacted with an extract of *Camellia sinensis* (tea) leaves in a stainless steel bomb digestion vessel for different time intervals.

Methods

Hydrogen tetrachloroaurate ($\text{HAuCl}_4 \cdot 3\text{H}_2\text{O}$, 99.9 % pure) was obtained from Aldrich chemicals, Germany. Commercially available tea powder from Tata tea Limited, India, was used for preparing the tea aliquot. Ultra high pure water with a resistivity of 18.2 $\text{M}\Omega\text{-cm}$ was used as reaction medium for the synthesis Au NPs. 2.0 g of tea leaves was weighed and transferred into a 100-ml volumetric flask and made up to the mark with water. The contents of the flask were dispersed by agitating it in a shaking water bath for 30 min and then left over night to collect the released *Camellia sinensis* leaf extract. The reddish brown color extract was decanted after centrifuging

at 200 rpm for 30 min to separate any un-dissolved ingredients. A dilute solution of gold (0.05 M) was prepared by dissolving suitable amount of $\text{HAuCl}_4 \cdot 3\text{H}_2\text{O}$ in water. To carry out the $\text{Au}^{3+} \rightarrow \text{Au}$ reaction, 8 ml of tea extract and 2 ml of 0.05 M $\text{HAuCl}_4 \cdot 3\text{H}_2\text{O}$ solution were pipetted out into a Teflon-coated stainless steel bomb digestion vessel and the container was sealed tightly. The bomb digestion vessel along with its reactants was then placed in an oven at 200 °C for different time periods of 1 and 5 h as shown in Table 1. A PG instrument (model T90+), UV–vis spectrometer was used to analyze samples collected from different reactions. To study the effective capping of biomolecules on the Au NPs surface, a Tensor 27 Fourier transform infrared spectrometer (FTIR) was used. Crystal structure of as-synthesized Au NPs was obtained using a Philips PW3020 X-ray diffractometer (XRD) consisting of an X-ray source of $\text{CuK}\alpha$ with 0.15405 nm wavelength. A transmission electron microscope (TEM) of FEI TECHNAI G2 was used for both imaging the Au NPs and obtaining their selected area electron diffraction (SAED) patterns. For TEM imaging, a small drop of the sample solution was dispersed onto a specific 3-mm carbon-coated copper grid and was dried in a desiccator at room temperature before TEM imaging.

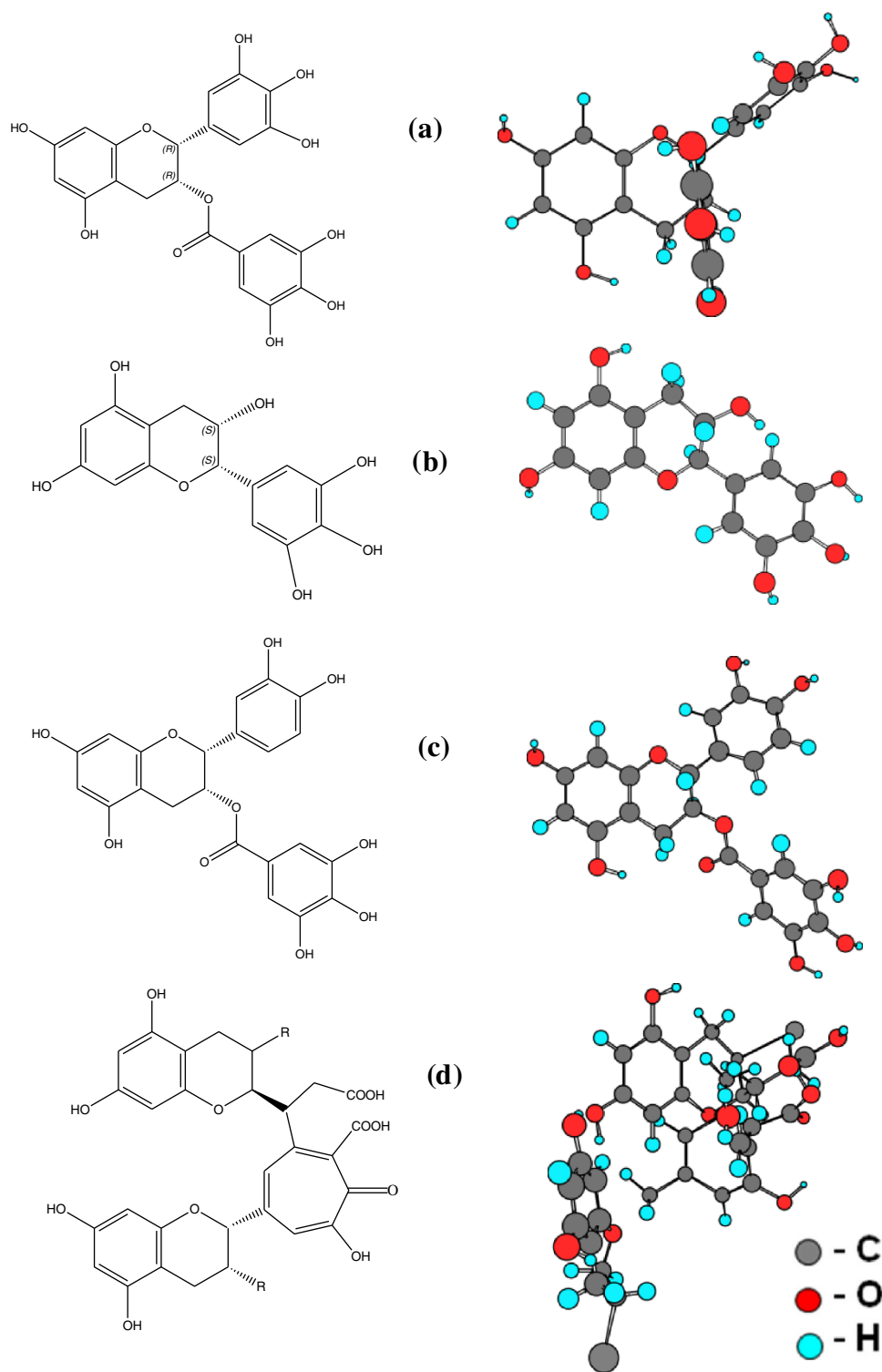
Results and discussion

The chemical reduction of aqueous HAuCl_4 solution under different experimental conditions was followed visually. The reactant solution from experiment 1 remained yellow even after 1 h of reaction in the bomb vessel, which indicates that the Au^{3+} ions did not get reduced. A similar reaction carried out in experiment 2 with 8 ml of tea leaf extract changed its color from yellow to pale pink indicating the reduction of Au^{3+} ions to Au. These results indicate that aurochloric acid does not reduce hydrothermally in the bomb digestion vessel under the given conditions in experiment 1. It also proves that the tea polyphenols are very essential for the reduction of Au^{3+} ions to Au NPs. Tea consists of two major categories of polyphenolic compounds, namely thearubigins and theaflavins. The three major constituents of theaflavins are Epigallocatechin gallate, Epigallocatechin and Epicatechin gallate [17]. Figure 1a–c depicts the chemical structures of

Table 1 Experimental conditions for synthesis of Au NPs of selective shapes upon reaction of *Camellia sinensis* with 0.05 M Au^{3+} species in a bomb digestion vessel at 200 °C

| Experiment | $\text{HAuCl}_4 \cdot 3\text{H}_2\text{O}$ (mL) | <i>Camellia sinensis</i> extract (mL) | Time (h) | Particle size (nm) | Shape |
|------------|-------------------------------------------------|---------------------------------------|----------|--------------------|-----------|
| 1 | 2 | 0 | 1 | – | – |
| 2 | 2 | 8 | 1 | 15 | Spherical |
| 3 | 2 | 8 | 5 | 45 | Rods |

Fig. 1 Chemical structures along with their 3D models of the three major theaflavin compounds **a** Epigallocatechin gallate, **b** Epigallocatechin, **c** Epicatechin gallate and **d** a typical Thearubigin



the three major theaflavin compounds and from their structures it could be observed that all the chemical compounds consist of hydroxyl (OH) groups adjacent to each other. This arrangement is similar to 1, 2 diols, which is

essential for the polyphenols to act as reducing agents. Therefore, as shown in Table 1, these polyphenolic compounds act as novel reducing agents for the synthesis of Au NPs in the above reactions. Whereas the structure shown in

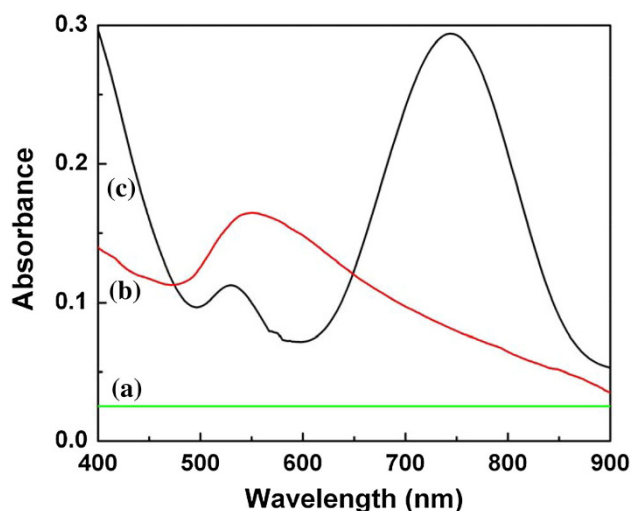


Fig. 2 UV-vis absorption spectra obtained from aqueous HAuCl_4 solution upon reaction in a bomb digestion vessel at 200°C under different reaction conditions **a** without tea extract for 1 h, **b** with tea extract for 1 h and **c** with tea extract for 5 h

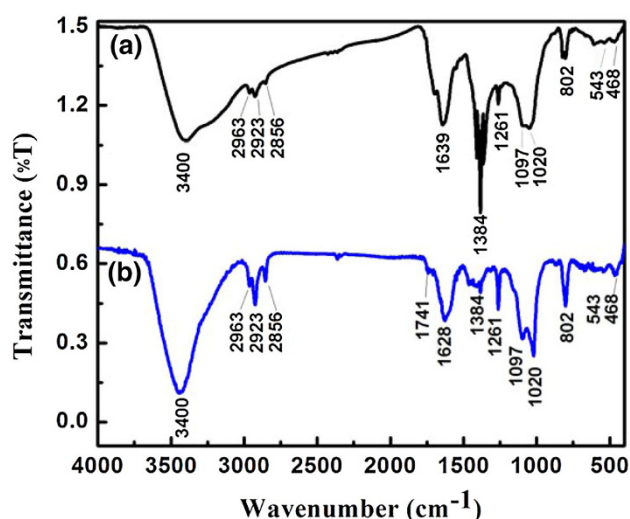


Fig. 3 FTIR spectra of **a** un-reacted tea extract and **b** colloidal solution of Au NPs obtained after a 5-h reaction

Fig. 1d is that of a typical thearubigin possessing the carboxylic acid (COOH) functional group, well known for their capping abilities [18, 23]. Therefore, the tea extract performs the dual role of novel reducing agent as well as an efficient bio-surfactant.

The reaction products were further characterized by UV-visible spectroscopy. Figure 2a–c shows three typical UV-vis spectra obtained from aqueous HAuCl_4 solution upon reaction in a bomb digestion vessel at 200°C under different reaction conditions. Figure 2a shows the spectrum recorded for the solution from experiment 1. It could be observed that only a flat line exists in the range of

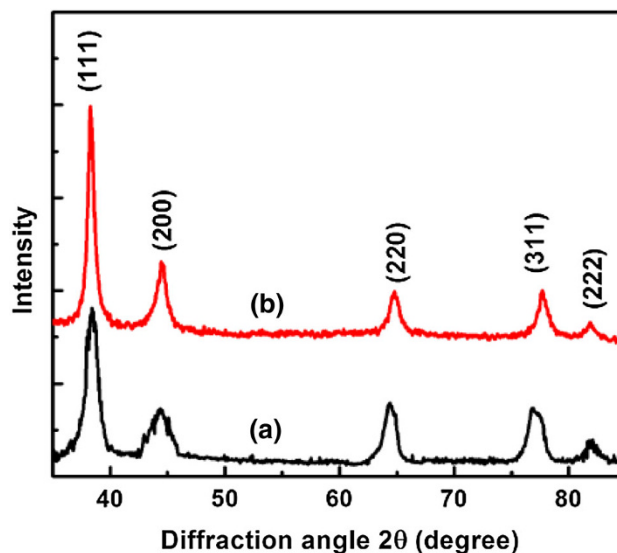


Fig. 4 X-ray diffraction patterns obtained from Au NPs **a** experiment 2 and **b** experiment 3

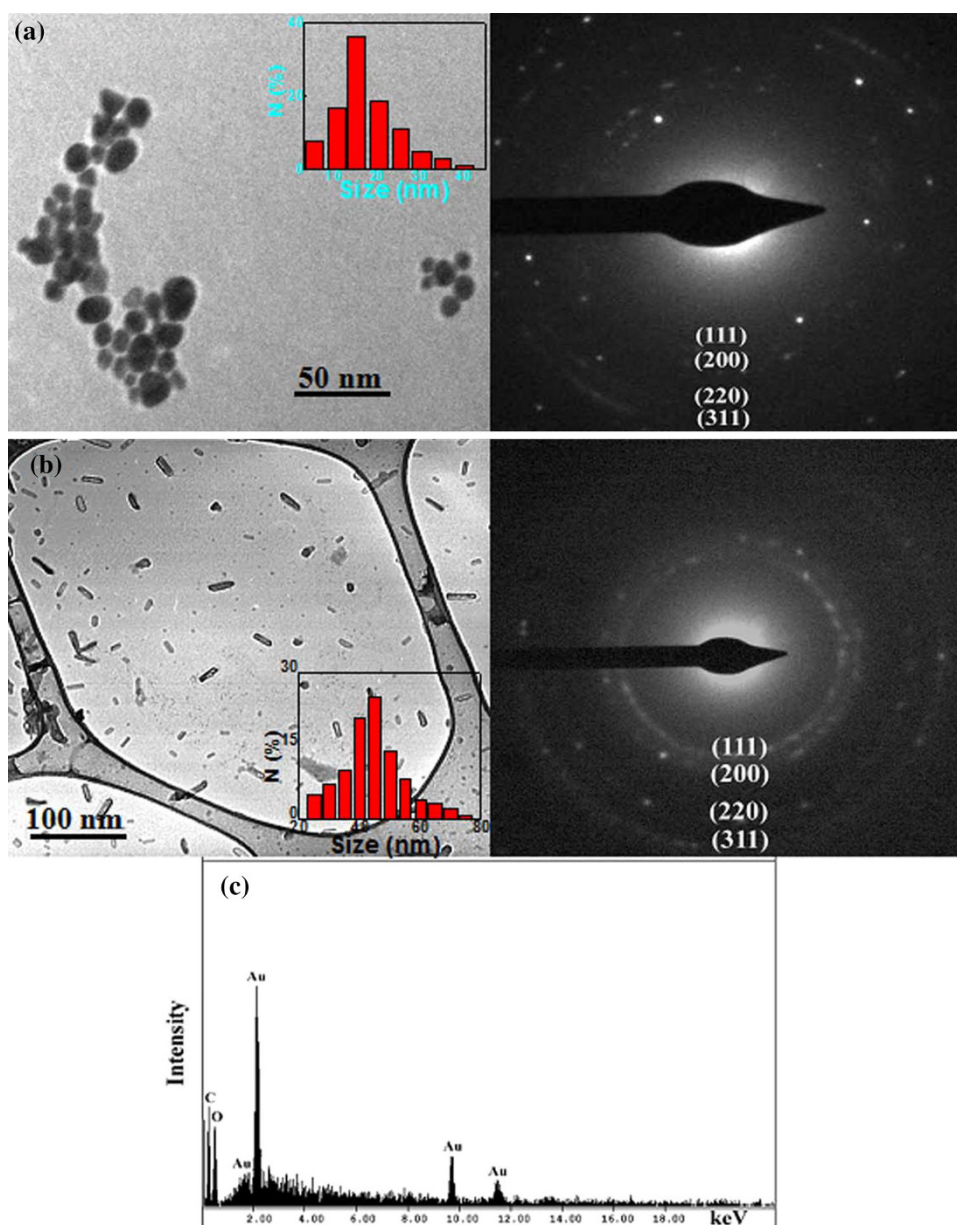
400–900 nm and the absence of any peaks in this region indicates that there is no change in the metal oxidation state of Au^{3+} ions. Figure 2b shows the UV-vis spectra recorded for the sample solution from experiment 2. Upon reaction for 1 h with 8 ml of black tea extract, the appearance of a single absorption peak centered at about 548 nm was observed. This could be attributed to the transverse SPR band of Au NPs, which indicates that upon reaction with the tea polyphenols, through the reduction of Au^{3+} ions, metallization of Au occurs. A single broad peak also leads to an inference that the Au NPs are isotropic in shape and uniform in size. As the reaction time was increased from 1 to 5 h, a drastic change in the UV-vis spectra was observed as shown in Fig. 2c. The peak at 548 nm diminished in its intensity and slightly blue shifted to 533 nm. A new broad peak with λ_{max} at 745 nm was observed, which could be attributed to a typical signal arising from the longitudinal SPR of Au nanorods. The above result indicates that upon extended heating, the spherical Au NPs convert into rod-like structures. However, a small peak persisting at 533 nm indicates that a minor fraction <10 % of the particles still remains in the form of nanospheres. The presence of typical SPR bands in the UV-vis region for Au samples indicates that the particles are in the nanometric range. This also proves that the polyphenolic constituents of tea not only act as good reducing agents, but also perform the function of stabilization of Au NPs by capping on to their surfaces.

Figure 3 shows the FTIR spectra of (a) un-reacted tea extract and (b) colloidal solution of Au NPs obtained after a 5-h reaction. To understand the nature of interaction between the tea extract and Au^{3+} ions, FTIR spectra were

analyzed in detail. From Fig. 3a, it could be observed that the un-reacted tea extract consists of a broad peak in the region of $3,200\text{--}3,500\text{ cm}^{-1}$ corresponding to the O–H stretch and the peaks at $2,963$, $2,923$ and $2,854\text{ cm}^{-1}$ could be attributed to the sp^3 and sp^2 C–H stretching vibrations of the organic moieties. Peak at $1,639\text{ cm}^{-1}$ could be attributed to the C=O stretch of the acid groups present in thearubigins and the peak at $1,384\text{ cm}^{-1}$ could be assigned to the stretching vibration of carboxylate ion ($-\text{COO}-$). A peak at $1,261\text{ cm}^{-1}$ shows the –OH deformation vibrations in the polyphenolic compounds and peaks at $1,097$ and $1,020\text{ cm}^{-1}$ could be attributed to the C–O and C–OH single-bond vibrations. The peaks present at 802 , 543 and 468 cm^{-1} confirm the presence of aromatic-substituted

rings. Except for a new peak at $1,741\text{ cm}^{-1}$ and a slight shift in the C=O stretch from $1,639$ to $1,628\text{ cm}^{-1}$, rest of the peaks remain unchanged in the spectrum obtained from that of Au NPs after the reaction with tea extract as shown in Fig. 3b. The new peak at $1,741\text{ cm}^{-1}$ could possibly be due to the conversion of C–OH group to C=O group during the reduction reaction of $\text{Au}^{3+} \rightarrow \text{Au}$. The very low intensity of this peak also suggests that most of the C–OH groups remain as such and only a small fraction of the them converts to C=O group, which is in good agreement with the fact that tea aliquot is taken in large excess to carry out the reaction. A red shift in the carbonyl frequency by 11 cm^{-1} indicates a weak coordination between the carbonyl group and the surface of Au NPs, which proves that

Fig. 5 TEM micrographs of Au nanoparticles obtained after reaction in a bomb digestion vessel at $200\text{ }^\circ\text{C}$ under different reaction conditions **a** with tea extract for 1 h and **b** with tea extract for 5 h. Respective selected area electron diffraction patterns are shown on the right. **c** Energy dispersive X-ray spectra obtained from 5 h reaction product



the Au NPs are protected by the polyphenolic compounds present in tea.

Figure 4 shows typical XRD patterns from recovered Au NPs (a) experiment 2 and (b) experiment 3. They consist of five characteristic peaks at 2θ values of 38.27° , 44.49° , 64.69° , 77.73° and 81.91° , which can be discerned to (111), (200), (220), (311), and (222) lattice reflections of a face-centered cubic (fcc) structure of Au (JCPDS JCPDS 01-1172), respectively. From the XRD patterns, the lattice parameter (a) was calculated for both (a) and (b) samples as 0.4076 and 0.4069 nm, respectively. Compared to peaks obtained from sample (b), Fig. 4a shows slightly broader peaks. Average crystallite size was calculated from the peak broadening using the Debye–Scherrer relation, which presents a value of 12 nm for sample (a) and 18 nm for sample (b).

Figure 5a shows representative TEM images recorded from the drop-coated sample of the Au NPs, synthesized by treating the aurochloric acid solution with an extract of black tea after 1 h in a bomb digestion vessel at 200°C (experiment 2). As shown in the inset, TEM images obtained from the above sample exhibit predominantly spherical particles of 15 nm size with narrow size distribution. However, few triangles and rods were also observed in the same sample. Selected area electron diffraction (SAED) pattern obtained from the above sample is shown on the right. The SAED pattern consists of four concentric rings, which could be assigned to (111), (200), (220) and (311) planes of fcc structure, which is in good agreement with the XRD pattern observed in Fig. 4a. Figure 5b shows TEM images for samples obtained from experiment 3. The micrograph depicts predominantly (>90 %) rod-like structures with an average size of 45 nm. This result corroborates well with the UV–vis spectrum observed in Fig. 2c, where two peaks were found with the major one at 548 nm and the minor peak at 533 nm, indicating that the major portion of the Au NPs exists in the form of nanorods and only <10 % of them retains their spherical shape. This is evident from the inset in Fig. 5b, which shows the size distribution graph depicting a small fraction of Au NPs in the range of 20–25 nm size. The SAED pattern obtained from the rods is shown on the right, which also exhibits a typical fcc structure of Au. Figure 5c shows the energy dispersive X-ray (EDX) spectra obtained from the gold nanorods, which confirm the presence of gold as major constituent and carbon and oxygen as trace impurities. The traces of carbon and oxygen could be attributed to the polyphenolic coating over the Au NPs.

The above results prove that Au NP formation occurs as early as 1 h after reaction in the bomb digestion vessel, whereas a typical reaction takes 4–5 h to

complete in an open vessel [16]. This shows that the rate of reaction between Au^{3+} ions and polyphenols increases rapidly when heated in a closed container. The 5-h reaction product, however, shows a distinct microstructure comprising predominantly of nanorods throughout the grid. The phenomenal change in microstructure from experiment 2–3 could be attributed to the fact that a lot of pressure develops inside the bomb upon constant heating at 200°C for 5 h. This excess pressure leads to the conversion of spherical Au NPs into rod-like structures. Similar results were also observed in the case of cobalt NPs prepared by a chemical reaction in bomb digestion vessel [24].

Conclusions

We have successfully demonstrated a complete green chemical route for the synthesis of Au NPs with different shapes (spheres and rods). Shape anisotropy of Au NPs could be achieved by simply carrying out the reaction in a bomb digestion vessel for different reaction times. Spherical Au NPs from experiment 2 were of 15 nm in size and exhibited a single SPR band at 548 nm, whereas the Au nanorods from experiment 3 were of 45 nm in size and contained two SPR bands in the UV–vis region. The longitudinal SPR band of gold nanorods at 745 nm is of great importance for biological applications. An added advantage of synthesizing Au nanorods here is that the NPs are capped with biomolecules (tea polyphenols) which not only are bio-compatible but also exhibit other health benefits.

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Conflict of interest Authors declare no competing financial interests. The authors have no other relevant affiliations or financial involvement with any organization or entity with a financial interest in or financial conflict with the subject matter of materials discussed in the manuscript apart from those disclosed. No writing assistance was utilized in the production of this manuscript.

Authors' contributions SSKK, JV and PKS hypothesized and executed the experiments, collected and interpreted the data, and wrote the manuscript. PG assisted in collecting, analyzing and interpreting the TEM micrographs. SR and LD were involved in writing and editing the manuscript and interpreting the results. All authors have read and approved the final manuscript.

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