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RESEARCH ARTICLE

Biosynthesis and characterization of Pt and Au-Pt nanoparticles and their photo catalytic degradation of methylene blue

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Abstract

A green synthesis strategy to prepare both monogeneous platinum nanoparticles (PtNps) and bimetallic heterogeneous platinum-aurum nanoparticles (PtAuNPs) using hot water extract of green tea as a reducing, stabilizing and protecting agent is reported. The nature of both PtNPs and PtAuNPs were characterized using UV/vis spectroscopy (UV–vis), transmission electron microscopy (TEM), Fourier transform infrared spectroscopy (FT-IR), thermal gravimetric analysis (TGA) and energy dispersive X-ray (EDX). TEM images revealed that the biosynthesis of PtNPs were in the size range of 2–4 nm and PtAuNPs in the size range of 10-40 nm and both are crystallized in face centered cubic symmetry. Photocatalytic degradation property of Pt and Pt-Au nanoparticles were investigated by degradation of Methylene blue dye under UV light irradiation. The results show that PtNps has higher activity of degradation of Methylene blue than PtAuNPs. So, this eco-friendly, simple, cost effectiveness method may be utilized for removal of waste (pollutants) of water and other industrial purposes for better environment.

Introduction

The noble metal nanoparticles such as gold, silver and platinum have been playing an important role in the fields of organic catalysis, bioelectronics and pharmaceuticals (Polsky et al., 2006; Eklund and Cliffel, 2004; Kang et al., 2008; Castaneda et al., 2007; Han et al., 2007). Platinum nanopatrticles, with a high melting point (1769 ˚C) and a good resistance to corrosion and chemical attack, plays a major role in many industrial applications. Platinum nanoparticles are enjoying increasing applications in organic catalysis, fuel cells, hydrogen storage, electrocatalysis, and bioelectronics (Xu and Yang, 2008; Wen et al., 2008; Wang and Yang, 2008; Jena and Raj, 2008; Kang et al., 2008).

On the other hand, gold nanoparticles have received great attentions for the fascinating size and shape related electronic, magnetic, and optical properties. They have important applications in biological tagging, surface-enhanced Raman spectroscopy, optoelectronics, and catalysis (Kuo and Huang, 2006; Abdelrahman et al., 2006; Liu et al., 2010; Huang et al., 2010). Bimetallic alloys such as platinum- gold (PtAu) alloy has an excellent catalysis and resistance to deactivation due to the high synergistic action between gold and platinum (Xiao et al., 2009).

Biosynthesis of nanoparticles as an emerging highlight of the interaction of nanotechnology and biotechnology has received increased attention due to growing need to develop environmentally benign technologies in material synthesis (Bhattacharya et al., 2005). With the growing need to minimize or eliminate the use of environmental-risk substances, as the green chemistry principles describe (Anastas and Kirchhoff, 2002), the synthesis of nanoparticles using biological entities has received increasing attention in the last decade (Mohanpuria et al., 2008). The

biosynthetic procedures involve either living organisms such as bacteria (Joerger et al., 2000), fungi (Bhainsa and D'Souza, 2006) and plants (Khalil et al., 2012; Kavitha et al., 2013, Mittal et al., 2013**)**. Biological synthetic processes have emerged as a simple and viable alternative to more complex physicochemical approaches to obtain nanomaterials with adequate control of size and shape (Shankar et al., 2004). Biosynthesis of platinum nanoparticles using *Plectonema boryanum* UTEX 485 (Lengke et al., 2006)), horse spleen apoferritin (Deng et al., 2009), hydrogenase enzyme from *Fusarium oxysporum* (Govender et al 2009) enzyme (Riddin et al., 2010) and *Dipyros kaki* leaf extract (Song et al., 2010) has been reported.

The dry leaves of the green tea plant *Camellia sinensis* (L.) are known to contain flavanols with antioxidant capacity such as catechin, (−)-epicatechin, (−)-epicatechin-3-gallate, (−)-epigallocatechin and (−)-epigallocatechin-3-gallate (Henglein, 1989; Alivisatos, 1996). The consumption of green tea is therefore associated with protective effects against coronary diseases and cancer of the lung, fore-stomach, esophagus, duodenum, pancreas, liver, breast, colon and skin, induced by chemical carcinogens (Henglein, 1989; Alivisatos, 1996).

We reported a bio-directed, facile, greener and economically feasible method for the fabrication of PtNps and Pt-AuNPs bimetallic alloy by using the extract of green tea as antioxidant. Moreover, their application in photocatalytic degradation of methylene blue dye under UV light irradiation is investigated.

2. Materials and Methods

2.1. Materials

$HAuCl_4$. H2O (Au content: 49%) was obtained from Electron Microscopy Sciences and H_2PtCl_6. H_2O (Pt content: 40%) was obtained from Alpha Chemika are used as received. Methylene blue ($C_{16}H_{18}C_lN_3SC_l$) which was obtained from Qualigens Fine Chemicals (India). Deionized water was used throughout the reactions. Green tea was obtained from local market.

All glass wares were washed with concentrated sulphuric acid H_2SO_4 and deionized water then dried in hot air oven. A stock solution of H_2PtCl_6 was prepared by dissolving 1.0 g in 50 ml deionized water (4.67×10^{-2}M). Similarly, a stock solution of $HAuCl_4$ (1.0 g in 50ml deionized water (5.59×10^{-2}M)) was prepared. .

2.2. Instrumentation

The UV–vis spectra were recorded at room temperature using a λ-Helios S Pye-Unicam spectrophotometer. Transmission electron microscopy (TEM) studies were performed using a JEOLJEM 1200 electron microscope operating at an accelerating voltage of 90 KV. For the TEM measurements, a drop of a solution containing the particles was deposited on a copper grid covered with amorphous carbon. After allowing the film to stand for 2 min, the extra solution was removed by means of blotting paper and the grid allowed drying before the measurement. Fourier transform infrared (FTIR) spectra were recorded at room temperature on a Nicolet 6700 FTIR spectrometer. For the FTIR measurements of capped nanoparticles, a small amount of Pt or PtAu nanoparticles (0.01 g) dried at 60 ^{0}C for 4 h was mixed with KBr to form a round disk suitable for FTIR measurements. To obtain the FTIR spectrum of the extract, an appropriate amount of the extract was mixed with KBr. Thermo-gravimetric analyses were carried out with a heating rate of 10 °C/min using a Shimadzu DT-50 thermal analyzer. X-ray diffraction (XRD) pattern was obtained using a Shimadzu XRD-6000 diffractometer with Cu Ka (λ= 1.54056 Å) to confirm the biosynthesis of nanoparticles. The energy-dispersive X-ray (EDX) data were obtained on a F20 Tecnai High Resolution microscope (Philips, Netherlands). For photocatalytic experiments, the irradiation source was a 75W Xenon arc lamp (PTI-LPS-220 Photon Technology International), situated above the reactants mixture beaker in the reactor.

2.3. Synthesis of nanoparticles

2.3.1 Preparation of the extract

1.0 g of green tea leaves was boiled for 15 min, filtered and completed to 50 ml to get the extract. The filtrate that was used as reducing agent was kept in the dark at 10^0C to be used within 1 week.

2.3.2. Synthesis of platinum nanoparticles

For the synthesis of platinum nanoparticles, 50 ml of plant extract (1g/ 100 ml deionized water) was added to 4mL of H_2PtCl_6 solution (1.0 g/50 ml). The final concentration of Pt was 3.4×10^{-3} M. By stirring at 100 ^{0}C for 1 h ,The reduction process of Pt^{+6} to Pt^0 nanoparticles was followed by the color change of the solution from yellow to brownish-yellow to deep black. The solid nanoparticles were obtained by centrifugation and dried at 100 ^{0}C.

2.3.3. Synthesis of PtAu bimetallic nanoparticles

For the synthesis of PtAu nanoparticles, 50 ml of plant extract (1g/ 100 ml deionized water) was added to 4ml of H_2PtCl_6 solution (1.0 g/50 ml) and 3 ml of $HAuCl_4$ solution (1.0 g/50 ml). The final concentration of Pt was 3.2 x 10^{-3} M and the final concentration of Au was 3.35 x 10^{-3} M. By stirring at 100 ^{0}C for 1 h, The reduction process Pt^{+6} to Pt^0 nanoparticles and Au^{+3} to Au^0 was followed by the color change of the solution from yellow to brownish-yellow to violet to deep black.

2.4 Photocatalytic degradation using PtNPs and Pt-AuNPs

A 0.05g of PtNPs or PtAuNPs prepared in **2.3.2** or **2.3.3** was mixed with 50ml of methylene blue (3 x 10^{-5} M) and the resulting solution was irradiated and the absorbance was measured at pre-determined time intervals. At given irradiation time intervals, samples were taken from the suspension and immediately centrifuged at 4000 rpm for 20 min, then the absorbance was measured for the clear solution.

3. Results and discussion

3.1. UV–vis spectroscopy characterization

Upon mixing aqueous green tea extract with H2PtCl6 at room temperature for more than 6 hours no change in color was observed. By boiling the mixture at 100 ^{0}C, the bright yellow colour of H_2PtCl_6 change to dark brown or black on completion of the bioreduction was monitored suggesting that the extract reduced H_2PtCl_6 resulting in the production of platinum nanoparticles. Huang et al. (2004) observed that colour changes could be used as an indicator of platinum nanoparticle formation. Soundarrajan et al.(2012) synthesized PtNPs using *Ocimum sanctum* at 100 ^{0}C. The high temperature is required for platinum reduction rate faster. The gold nanoparticles was found to form at room temperature. The rapid conversion of gold was noted within 10 min respectively at a temperature of 90 ^{0}C using olive leaf broth (Khalil et al. 2012)..

The formation of Pt^0 nanoparticles and PtAu bimetallic nanoparticles alloy was followed by UV–vis spectrophotometer. Fig. 1 shows the UV–visible spectra of platinum nanoparticles and PtAu bimetallic nanoparticles. In aqueous solution, literature has reported that Pt(0) nanoparticles have an absorbance spectrum that stretches across the whole of the ultraviolet–visible region (Creighton and, Eadon 1991). Consequently there is no specific wavelength that could be used to accurately determine their concentration. The peak at 330 nm can be taken as indication for Pt^0 formation its UV–vis spectrum and 536 nm for Au^0 as shown in Fig. 1

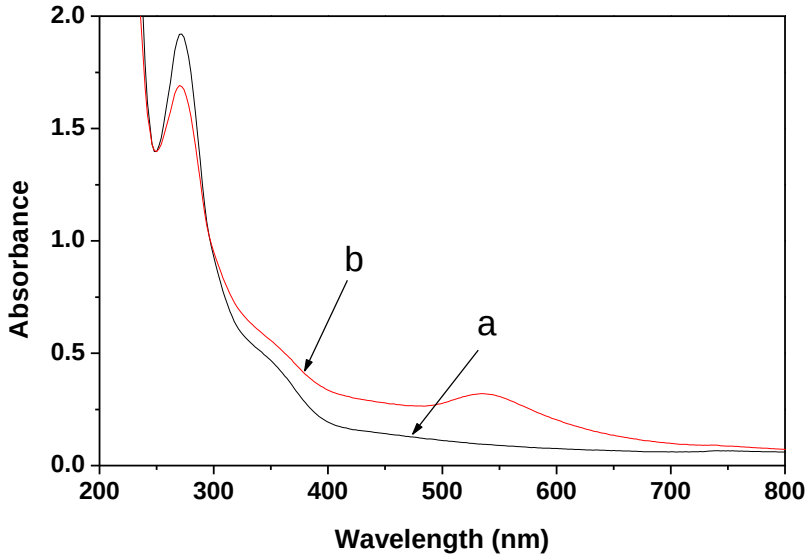

Fig. 1.UV-Vis absorption spectra (a) of Pt nanoparticles absorption (b) Pt and of Au nanoparticles absorption.

3.2. TEM characterization

Pt and PtAu nanoparticles were further confirmed by TEM images, Fig. 2. The results indicate that 2 nm Pt nanoparticles were produced as shown in Fig. 2.a; the formed platinum- gold nanoparticles were predominantly spherical with some triangular and hexagonal shaped, with diameters ranging from 10 to 50 nm as shown in Fig. 2b.

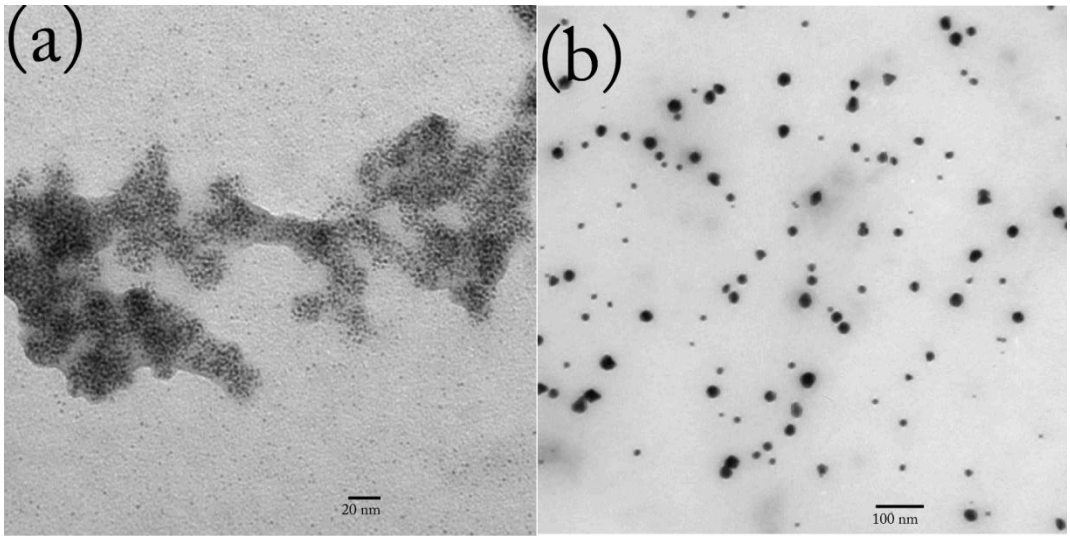

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Fig. 2.Typical TEM images of green tea leaves extract mixed with (a) Pt nanoparticles (b) PtAu nanoparticles.

3.3. X-ray diffraction (XRD)

Fig. 3a shows the X-ray diffraction (XRD) patterns of dried PtNps synthesized using green tea leaf extract at 100 ^{0}C. Diffraction peaks at 2θ = 39.89, 46.40 and 67.97◦ correspond to the indexed planes (111), (200) and (220), respectively were consistent with the crystalline Pt face-centered cubic (*fcc*) phase, (Fu et al., 2003). Fig. 3b. shows XRD of PtAu bimetallic nanoparticles synthesized using green tea leaf extract at 100 ^{0}C extract after the complete reduction of Au^{3+} to Au^0.Other than platinum diffraction peaks, a number of Bragg reflections were present which can be indexed on the basis of the face centered cubic (fcc) structure of gold. The diffraction peaks at = 38.31 (111), 44.46(200), 64.67(220) and 77.45(311) obtained are identical with those reported for the standard gold metal (Au^0) with crystalline structure (Joint Committee on Powder Diffraction Standards-JCPDS, USA).

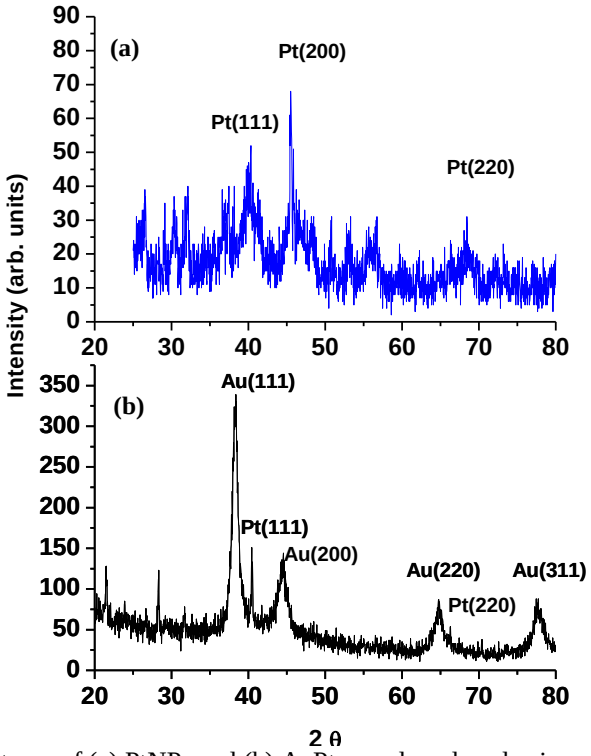


Fig.3. X-Ray diffraction patterns of (a) PtNPs and (b) AuPt sample reduced using green tea leaf extract at 100 ◦C.

3.4. Energy dispersive X-ray (EDX)

The obtained platinum nanoparticles were verified to contain the platinum according to EDX studies. Fig. 4a shows the EDX spectrum of green tea extract mediated nanoparticles, which confirms the presence of platinum demonstrating that the platinum nanoparticles were formed by reduction of the $PtCl_6^{-2}$ ions.

Figure 4b displays the EDX spectrum of Au-PtNPs. The peaks of both Au and Pt peaks are clearly visible. The EDX spectra demonstrated that the metal nanoparticles consist of an alloy. Hence, EDX demonstrates that the nanoparticles seen in the TEM images (Fig. 2) are true alloys because Au and Pt exhibit a miscibility gap in bulk.

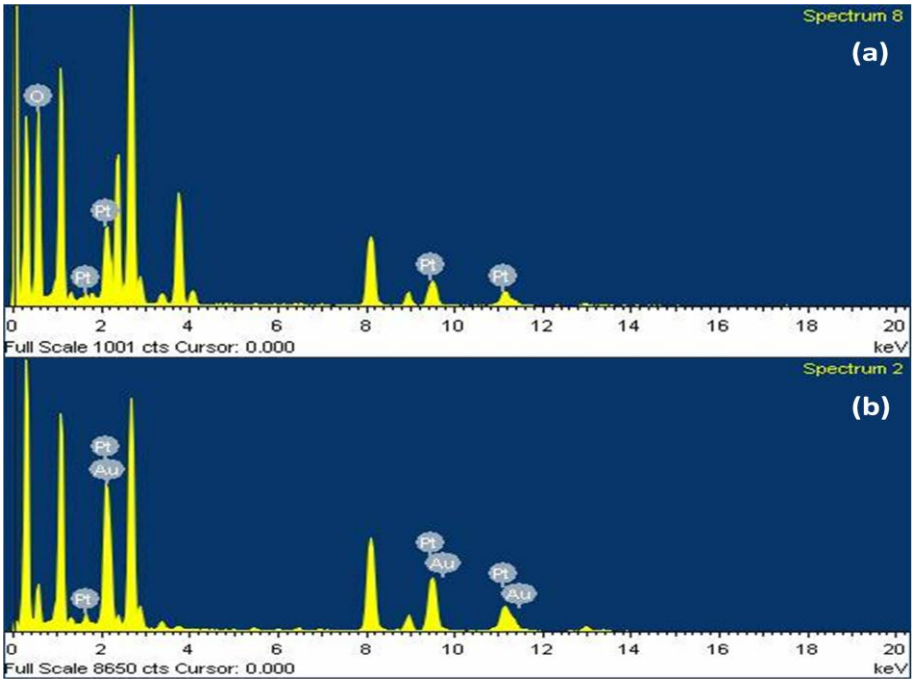


Figure 4 .EDX spectrum of one single Pt-NP (a) and Au-Pt-NP (b) on the surface of the carrier particle.

3.5. FTIR characterization

FT-IR spectroscopic studies were carried out to analyze the presence of polyphenols in the biosynthesized nanoparticles. the FTIR bands for green tea extracts showed that the bands at 3398 cm^{-1}, 1700 cm^{-1}, 1647 cm^{-1},1450 cm^{-1} and 1036 cm^{-1} refer to O-H, C=O, C-N(amide), C=C and C-C stretching vibration respectively, indicating that water-soluble polyphenols and caffeine in the green extract were responsible for the bio-reduction and capping of the Pt NPs as shown in Fig. 5(A) (Kumar et al., 2013). Fig. 5(B) and Fig. 5(C) shows that the FTIR bands for GT-Pt NPs and GT-AuPt NPs respectively, state that the Pt^0 surface and Au^0 surface were capped by the green tea extracts because they matched well with Fig. 5(A) except for some differences. First, the stretching vibration of hydroxyls shifted to longer wavelength. Second, the stretching vibration of C-N amide, C=O, C=C, C-C shifted to shorter wavenumbers in case of Pt NPs and to longer wavelenumbers in case of AuPt NPs. Those shifts indicate that polyphenols and caffeine (shown below) are responsible for capping and stabilizing of the nanoparticles.

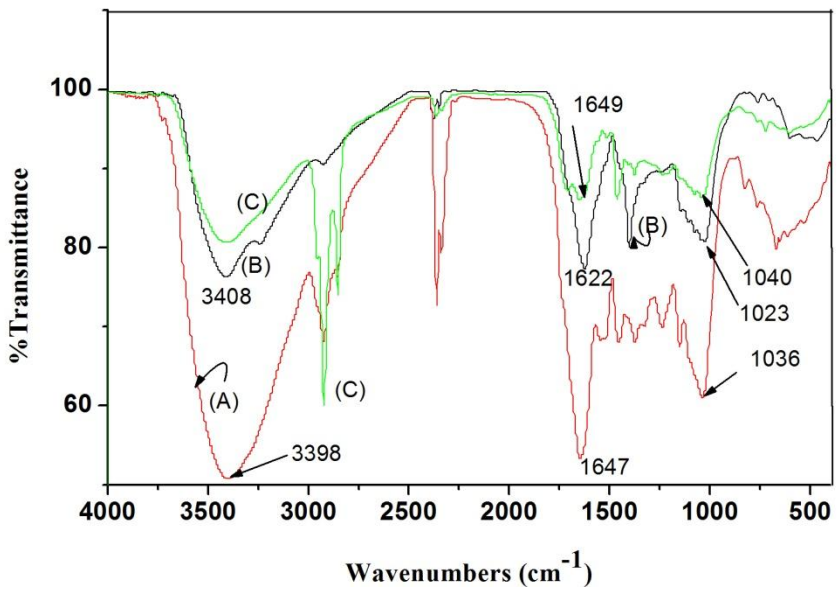
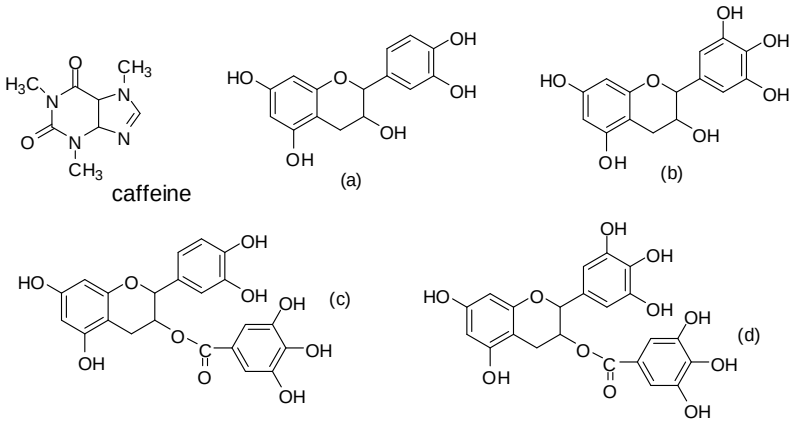


Fig.5. FTIR spectra of (A) green tea extract (B) capped platinum nanoparticles (C) capped Gold-Platinum nanoparticles.



Main component of grean tea leaves extract: caffeine and catechin that composed of (a)EC (b)EGC (c)ECG (d)EGCG.

3.6. Thermal gravimetric analysis

The TGA plot of the capped Pt NPs and capped AuPt NPs prepared using green tea extract (Fig. 6a,b) showed a steady weight loss in the temperature range of 50-700 ^{0}C. The weight loss of the nanopowder due to desorption of bioorganic compounds in the Pt NPs and AuPt NPs were 48.4% and 50.07% respectively .

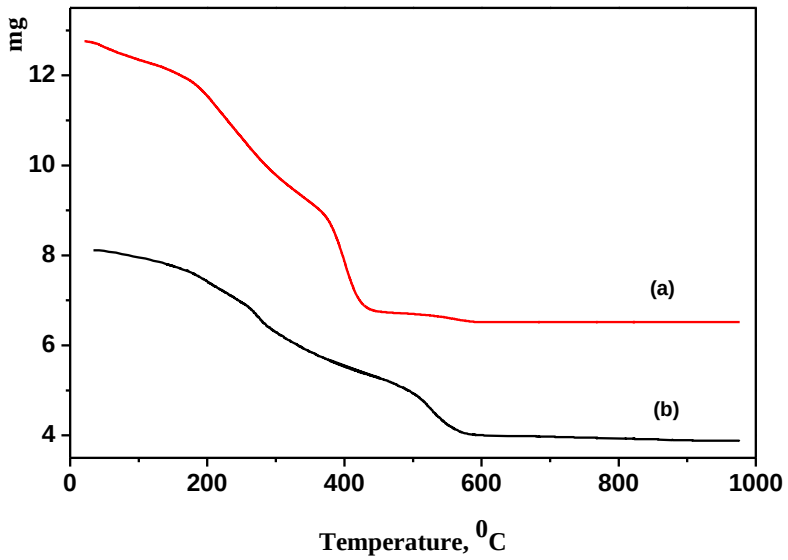


Fig.6. TGA of (a) capped platinum nanoparticles using green tea extract (b) capped PtAu NPs using green tea extract.

3.7. Application

The photocatalytic activity of the Pt and PtAu nanoparticles were investigated by choosing the photocatalytic degradation of Methylene blue. The characteristic absorption peak at 662 nm of Methylene blue was used for monitoring the catalytic degradation process. The absorption spectra of aqueous solution of Methylene blue tested at different time intervals in the presence of Ag nanoparticles is shown in Fig. 7. As seen from Fig, 7 that the main absorption peak of Methylene blue decreased gradually with the extension of the exposure time, indicating the photocatalytic degradation of Methylene blue dye. In the absence of nano-catalyst (control) the reaction did not have any progress. Also, the photocatalytic activity of Pt nanoparticles is higher than that of PtAu nanoparticles. This can be attributed to the difference in the size of the nanoparticles from 2-4 nm of PtNPs to 10-40 nm of PtAuNPs. Literature in photocatalysis research reveals that photocatalytic activity can be strongly dependent on the

crystallographic structure, morphology, and size of the particles (Kamat, 1993; Balazs et al., 2008). Further work is going on toward study the effect of impregnation of the Pt and PtAu nanoparticles on TiO_2 on the photodegradation of methylene blue.

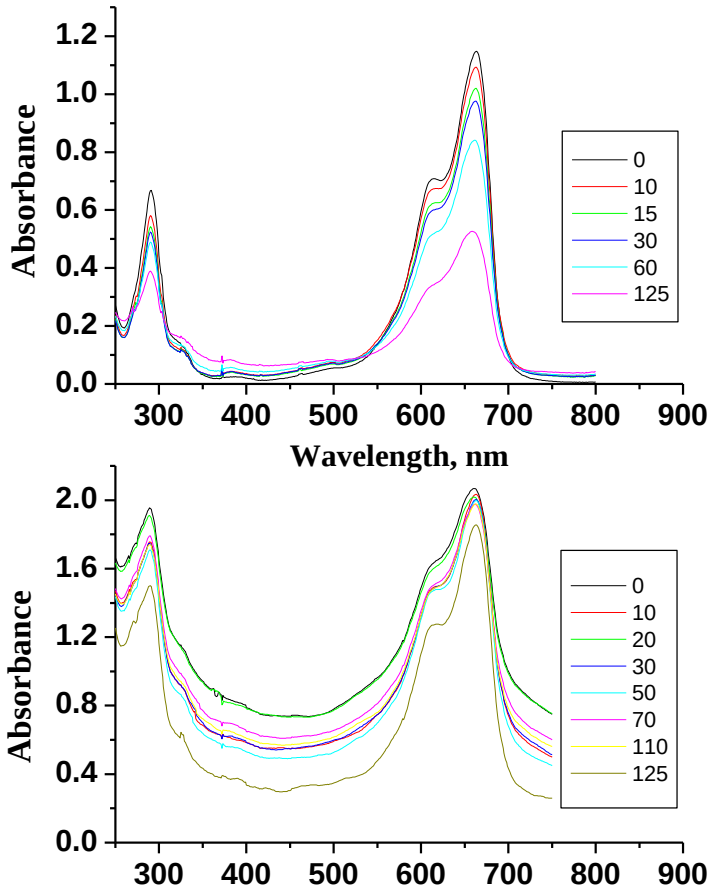


Fig. 7. Photodegradation of methylene blue using a) PtNPs and b) PtAuNps.

4.Conclusion

A simple, eco-friendly, green rapid biosynthesis of stable Pt and PtAu nanoparticles using green tea leaves was demonstrated. The formation of metal nanoparticles by reduction of the metal ions is possibly facilitated by caffeine and catechin present in green tea extract that is responsible for reduction, stabilization and capping of Pt and PtAu nanoparticles. The biological synthesis of metal nanoparticles using plant extracts provides a simple and efficient route for the synthesis of nanomaterials. Photocatalytic property of the Pt and PtAu nanoparticles was investigated by degradation of Methylene blue under UV light. From the study it was found that the Pt and PtAu nanoparticles exhibit suitable photocatalytic activity and rate of the reaction increase. The model further can be utilized for purification of pollutant from water and other industrial purposes.

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