Kinetic study of gold nanoparticle mediated photocatalytic degradation of Victoria blue

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Abstract

In the study, biogenic gold nanoparticles (AuNPs) were used for the photocatalytic degradation of triphenylmethane dyes Victoria blue B (VBB) and R (VBR). The process was found to result in an approximate degradation of 65 and 52%, respectively, for VBB and VBR within a period of 8 h. The relative rate of photocatalytic degradation of VBB and VBR was identified to be $0.0195 \pm 0.0031/\text{min}$ and $0.0295 \pm 0.0025/\text{min}$, respectively, by using the Langmuir–Hinshelwood model. By using the *Vigna unguiculata* model system, the degradation products were demonstrated to have non-toxic effect. Moreover, the less toxic nature of AuNPs used for dye removal highlights its feasibility for large-scale application. Hence, the AuNPs-based photocatalytic dye degradation as described in the study is cost-effective, rapid and environment-friendly.

Keywords Gold nanoparticle · Victoria blue · Photocatalysis · UV–Vis spectroscopy · Phytotoxicity

Introduction

Triphenylmethane-based textile dyes have diverse toxic effects. As some of these are considered as potent clastogens, its removal from water is highly significant (Azmi et al. 1998; Cho et al. 2003). Also its accumulation in water bodies has shown to result in incomplete degradation and formation of carcinogenic aromatic amines (Toh et al. 2003). These dye pollutants are chemically stable and hence traditional water treatment methods have limited application for their removal. The widely used textile dye Victoria blue (VB) is a chloride salt of triphenylmethane. Here the carbon–chlorine bond is present and the chlorine atom is ionized within the aromatic structure of VB. Hence its microbial degradation has previously been suggested to be difficult and time-consuming (Lade et al. 2015; Khan et al. 2012). As the dyes are resistant to microbial oxidation (Padikkaparambil et al. 2013), development of novel and eco-friendly methods for their efficient removal is highly demanding.

Developments in nanotechnology have resulted in the generation of nanoproducts and nanoparticles (NPs) with novel and size-correlated physico-chemical (Mock et al. 2002), antimicrobial (Wani and Ahmad 2013) and catalytic properties (Zhou et al. 2010; Ju-Nam and Lead 2008). Among the various methods for nanoparticle fabrication, microbiological methods are cost effective and are green in approach (Velusamy et al. 2016). This is quite attractive with respect to gold nanoparticles (AuNPs) due to its non-toxic nature. Diverse microorganisms like *Bacillus amyloliquefaciens*, *Bacillus subtilis*, *Streptomyces viridogens* and *Geotrichum candidum* have been reported for the synthesis of gold nanoparticles (Mittal et al. 2013; Nadaf and Kanase 2016; Thirumurugan et al. 2012). Here, the proteins or enzymes of microbial origin are considered to be the likely basis of reduction of metal salts to nanoparticles and its subsequent stabilization (Mittal et al. 2013).

The higher surface to volume ratio of nanoparticles provides its suitability for catalytic reactivity (Song et al. 2009). Its light absorption property is due to the localised surface plasmon resonance (LSPR) effect (Sarina et al. 2013). Both gold (AuNPs) and silver nanoparticles (AgNPs) were identified to combine the light flux to the conduction electrons of nanoparticles (Sarina et al. 2013; Saha et al. 2017), which provide opportunities to explore NPs for photocatalytic remediation. The method can further be made cost-effective and eco-friendly by using biofabricated nanoparticles.

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Hence, gold nanoparticles generated by previously isolated *B. amyloliquefaciens* SJ14 were used in the study to explore its dye degradative capability. The selected organism has been observed to form AuNPs rapidly when compared to previous reports (Nadaf and Kanase 2016; Mondal et al. 2017). Due to its LSPR effect, the AuNPs can have tremendous promises to be used as nano-based open industrial effluent treatment system with automated functioning in presence of sunlight. By further immobilization, its continuous use can be assured with minimal environmental release. Here also the inherent non-toxic nature of AuNPs provides added advantage as it is safer to the environment even if get released. The nano-based system can have comparative advantage over microbial enzymatic degradation due to its suitability to harsh conditions of treatment. The tailor-made design of nano-based treatment strategies enable effective utilization of treated water also. In the current study, combination of catalytic and light absorption properties of AuNPs was used to mold an efficient method for the degradation of VBB and VBR dyes. The less toxicity nature of AuNPs, its least explored application in nanoremediation and its biogenic origin make the study highly attractive.

Methods

Dye

The dyes VBB and VBR ($\lambda_{\max} = 615$ nm) were purchased from Hi-Media (RM7614) and Sigma-Aldrich (234176), respectively. Stock solutions were prepared by dissolving 10 mg of dye in 100 mL distilled water and the required concentrations were further prepared by dilution.

Synthesis, purification, and characterization of gold nanoparticles

B. amyloliquefaciens SJ14 was used for the synthesis of AuNPs as per the previously described method (Roshmi et al. 2015). The biomass of the overnight culture of SJ14 was collected by centrifugation at 10,000 rpm for 10 min. Then 100 mL of filter sterilized 1 mM HAuCl₄ was added into 2 g wet biomass followed by incubation at 30 °C in the presence of light for 24 h. The bacterial biomass incubated without HAuCl₄·3H₂O was maintained as the control. After AuNPs synthesis, the reaction mixture was centrifuged at 15,000 rpm for 15 min and the pellet was washed and resuspended in 50 mM Tris buffer (pH 7). The bacterial cells were further disrupted by ultrasonication (Sonics-Vibra Cell). Then the solution was further filtered through 0.22 µm filter, and the obtained purified AuNPs were used for further characterization (Roshmi et al. 2015) by using UV–Vis spectrophotometer at a wavelength range of 400–700 nm

(UV–Visible spectrophotometer (SHIMADZU UV-2600 series) and HR-TEM (JEOL, JEM-2100 instrument) at 80 kV. Powder X-ray diffraction (pXRD) was used for the analysis of crystalline nature of purified biogenic AuNPs. The spectrum was recorded on a model D8-Advance of Bruker (Germany), in the 2θ range of 5°–80° (Thomas et al. 2015).

Photocatalytic degradation of VBB and VBR

The degradation process was carried out in Erlenmeyer flasks containing 0.4 mg VBB or VBR in 100 mL distilled water in presence of 10 mg AuNPs. The flasks were kept for eight hours in static condition under direct sunlight. An aliquot of 5 mL was removed from each of the flasks at a time interval of 1 h, and the absorbance was analyzed using UV–Visible spectrophotometer (Aswathy Aromal et al. 2012).

Percentage of decolorization was calculated by using the formula:

$$\% \text{Decolorization} = \frac{\text{Initial absorbance} - \text{Final absorbance}}{\text{Initial absorbance}} \times 100$$

Kinetics of photocatalytic degradation

The kinetics of photocatalytic degradation was analysed by following Langmuir–Hinshelwood model

$$r_i = -\frac{dC_i}{dt} = \frac{kKC_i}{1 + kKC_i} \quad (1)$$

where C_i is the concentration of the textile dye solution, k represents reaction rate constant, and K is the adsorption coefficient of the textile dye to the catalyst. When C_i is small ($C_i < 10^{-3}$ M), $kKC_i \ll 1$ and Eq. (1) will be simplified to pseudo-first-order equation:

$$r_i = -\frac{dC_i}{dt} = kKC_i \quad (2)$$

on integrating Eq. (2):

$$\ln \left(\frac{C_0}{C} \right) = k_{\text{obs}} t \quad (3)$$

where C_0 is absorbance at the initial concentration of the dye and $k_{\text{obs}} = kK$ is the observed pseudo-first-order reaction rate constant. Thus, the reaction rate k_{obs} can be obtained from the slope of $\ln(C_0/C)$ versus t . The concentration of the dye solution during the photocatalytic degradation was determined from the $\lambda_{\max}$ using a spectrophotometer based on the Lambert–Beer law, where the absorbance was proportional to the concentration. The reaction rate k_{obs} was calculated from fitted linear plots of VBB and VBR (Cui et al. 2013).

Effect of different factors on dye degradation

To investigate the effects of dye concentration, AuNPs concentration and pH on the photocatalytic activity, different concentrations of VBB and VBR (0.2, 0.4 and 0.6 mg/100 mL), different concentrations of AuNPs (5, 10 and 20 mg/100 mL) and different pH conditions such as 3, 5 and 7 were done in triplicates. The progress of reaction in each case was measured as per the above described method.

Phytotoxicity study

Assessment of eco-toxicity of AuNP degraded products of VBB and VBR was carried out using seeds of *Vigna unguiculata*. 2% sodium hypochlorite solution was used for the initial surface sterilization of selected seeds. This was followed by soaking seeds in sterile distilled water for overnight for its germination (Soghra Younesikelaki et al. 2016). Then ten germinated seedlings were placed in soil containing pots. Each set of seedlings were treated with (5 mL per day) dyes of 0.4 mg/100 mL concentration, AuNPs treated dyes and distilled water as control. All treatments were triplicated and the length of shoot, root, and number of roots were measured after 5 days.

Statistical analysis

Means obtained from triplicated experiments were used for statistical analysis by using one-way analysis of variance (ANOVA) by Origin Pro7 SRO (Northampton, MA, USA), with the Tukey post hoc multiple comparison tests to conclude the significant difference in various groups at 5% level of significance.

Results and Discussion

Biosynthesis and characterization of AuNPs

A color change from pale yellow to dark purple was observed in the flasks containing bacterial biomass and aurochloric acid mixture and this indicated the AuNP formation as per previous report (Malarkodi et al. 2013). The formation of AuNPs was further monitored by UV–Vis spectrophotometric analysis, where a strong narrow peak was identified at 540 nm (Fig. 1a). TEM analysis of the AuNPs proved their shape to be spherical and triangular and they also had a non-aggregate nature (Fig. 1b) (Malarkodi et al. 2013). The AuNPs were identified to have a size range of 9–32 nm as per the particle size histogram analysis (Fig. 1c). Four intense peaks with 2θ values ranging from 20° to 80° were observed in the XRD spectrum of AuNPs (Fig. 1d) and were at 38.2° , 44.3° , 64.5° and 77.4° . These could be assigned

to (1 1 1), (2 0 0), (2 2 2) and (3 1 1) planes, respectively. The result was in accordance with the pure crystalline gold structure database of the Joint Committee on Powder Diffraction Standards (JCPDS) file No. 04-0783. The mean particle diameter of gold nanoparticle was also calculated from the XRD spectrum according to the line width of the (220) refraction peak using the Scherrer equation:

$$D = \frac{0.9\lambda}{\beta(\cos \theta)}$$

where β is the full width at half maximum (FWHM), λ is the X-ray wavelength, and θ is the reference peak width at angle; D is the average crystallite area size perpendicular to the sparkly planes. The size of the gold nanoparticles as calculated by Debye–Scherrer equation was found to be 16 nm.

Photocatalytic degradation

Photocatalytic degradative activity of the AuNPs was studied with VBB and VBR. The visual observation (Fig. 2a, b) showed a decrease in intensity of blue color with extension of time. Pure VBB and VBR showed its absorption peak at 615 nm and the same was found to decrease upon treatment with AuNPs due to catalytic degradation (Fig. 3b, d). However, there was no change for the absorption peaks of VBB and VBR control (Fig. 3a, c). This clearly revealed the remarkable influence of AuNPs on the degradation of VBB and VBR in presence of sunlight. Because of this, there was visually observable change in colour from deep blue into light. Importantly, 65% (VBB) and 52% (VBR) degradation was found to occur within 8 h which was a rapid and completely eco-friendly process. Previous report on photocatalysis of AuNPs has proved the relation of its catalytic activity with the crystallographic structure and size (Mata et al. 2016). The rate of catalytic reaction is considered to increase with decrease in nanoparticle size. By decreasing the size of NPs, it may favor increased number of co-ordinate atoms which ultimately mediate enhanced photocatalytic degradation of adsorbed dye (Wei et al. 2010; Saharan et al. 2016). Gold nanoparticles can act as donor as well as acceptor of electrons which help the electron transmit in redox reaction (Wunder et al. 2011; Mallick et al. 2006) and this catalytic activity of gold nanoparticles may also be favored by its small size which provides greater surface area for adsorption of dye. As there are only very few reports on exploration of microbially fabricated AuNPs for dye degradation, the result presented in the paper is highly significant. The reaction rate k_{obs} for VBB and VBR was found to be $0.0195 \pm 0.0031/\text{min}$ and $0.0295 \pm 0.0025/\text{min}$, respectively. The k_{obs} values showed the photocatalytic activity to be high in the case of VBR. The VBR might have adsorbed on the surface of AuNPs more effectively than VBB to result in elevated degradation (Fig. 4a, b, Table 1).

Fig. 1 Characterization of AuNPs synthesized by *B. amyloliquefaciens* SJ14 **a** UV-Vis absorption spectrum of AuNPs, **b** TEM image of AuNPs, **c** Histogram of particle size distribution of biosynthesized AuNPs, **d** XRD pattern of AuNPs

Fig. 2 Visual observation on photocatalytic degradation of **a** VBB and **b** VBR by AuNPs. All reaction vials contained 0.4 mg dye and 10 mg AuNPs per 100 mL reaction mixtures. The degradation process was carried out in presence of sunlight up to a time period of 0–8 h

Effect of different factors on degradation

In the current study, the dye concentration was found to have an inverse relation to the photocatalytic activity of AuNPs. At higher dye concentration, optimal dye adsorption on the

surface-active sites of nanoparticles might have affected (Mai et al. 2008) and a reduction in availability of light to nanoparticle surface might also have occurred (Nikazar et al. 2008; Bharati et al. 2017). Because of this, AuNPs treatment with VBB and VBR at a concentration of 0.2 mg/100 mL

Fig. 3 UV–Vis absorption spectra of dye treatment under different conditions **a** Victoria blue B in the absence of AuNPs and **b** in presence of AuNPs **c** VBR in the absence of AuNPs and **d** in presence

of AuNPs. The reaction was carried out in presence of sunlight at a dye concentration of 0.4 mg and AuNPs concentration of 10 mg per 100 mL reaction volume

in presence of sunlight showed 70 and 60% of degradation after 8 h (Fig. 5a, b).

From an application point of view, it is essential to evaluate the effect of concentration of AuNPs on the photocatalytic degradation. So AuNPs at concentrations of 5, 10 and 20 mg were used for the treatment of VBB and VBR (0.4 mg/100 mL) at pH 7. The results (Fig. 6a, b) showed a positive effect of increased AuNPs concentration on the photocatalytic degradation. Here 67 and 62% degradation of VBB and VBR was observed with 20 mg AuNPs. The

increased concentration of AuNPs might have provided more reactive surfaces for the degradation.

In the study, 0.4 mg dye and 10 mg AuNPs were also subjected to photocatalytic degradation studies under three different pH values (3, 5 and 7). The results (Fig. 7a, b) confirmed pH 7 as the optimal value for photocatalytic degradation of VB. Changes in pH have been reported to have remarkable impact on the ability of the dye to get adsorbed onto the surface of nanoparticles (Mai et al. 2008). The decrease in photocatalytic degradation of VB in lower pH might be due to the aggregation of AuNPs also (Wang et al. 2015).

Fig. 4 Relative rate of photocatalytic degradation of **a** VBB and **b** VBR. The degradation process was carried out in presence of sunlight at a dye concentration of 0.4 mg and AuNPs concentration of 10 mg per reaction mixture

Table 1 Relative rate of photocatalytic degradation of VBB and VBR

Dye (catalyst)	k_{obs} (min^{-1})	R^2
VBB (AuNPs)	0.0195 ± 0.0031	0.90
VBR (AuNPs)	0.0295 ± 0.0025	0.97

k_{obs} reaction rate, R correlation coefficient, VBB Victoria blue B, VBR Victoria blue R

Phytotoxicity study

The release of dye containing wastewater onto land even after treatment can have a direct impact on soil fertility and environment. Thus, it is very significant to evaluate the toxicity of the dyes before and after degradation on a living system for the verification of non-toxic or less toxic effect of the degradation products. So comparative sensitivity response of *V. unguiculata* (cowpea) towards the dye and its degradation products was carried out by using seed germination and plant growth assays (Rajan and David Noel 2015).

Fig. 5 Percentage of decolorization of **a** VBB and **b** VBR at different dye concentrations by AuNPs in presence of sunlight. The dye concentrations used were 0.2, 0.4 and 0.6 mg and AuNPs were 10 mg per 100 mL reaction volume

Fig. 6 Percentage degradation of **a** VBB and **b** VBR at different AuNPs concentrations in presence of sunlight. The dye concentration used was 0.4 mg per 100 mL reaction mixture. The AuNPs concentrations for same reaction volume were 5, 10 and 20 mg/100 mL

Fig. 7 Percentage of degradation of **a** VBB and **b** VBR by AuNPs at pH of 3, 5 and 7. AuNPs used was at a concentration of 10 mg per 100 mL reaction mixture at a dye concentration of 0.4 mg for same the volume

The application of VBB and VBR dyes showed a significant toxic effect on plant growth than their degradation products, representing non-toxic nature of degradation products as compared to dye (Fig. 8a, b). Plants treated with VBB and VBR showed retardation in growth which implies toxicity. However, plants treated with degradation products exhibited normal growth when compared to control plants. The data obtained were statistically analyzed by comparing it with the control samples and it showed VBR to be more toxic (Fig. 8c, d). Most of the earlier dye

removal studies have targeted only on the color removal while excluding the fact that the processes can also result in the formation of toxic products. Hence, it is very significant to evaluate degradation effect with ecotoxicity consideration. In the current study, phytotoxicity analysis has confirmed the photocatalytic degradation products of VBB and VBR to have less toxic or non-toxic effect, which thereby ensure environmental safety with the release of water containing the treated product to land (Rajan and David Noel 2015).

Fig. 8 Effect of untreated and treated **a** VBB and **b** VBR with AuNPs on *Vigna unguiculata* plants. 1—untreated VBB, 2—treated VBB with AuNPs, 3 and 6—distilled water, 4—untreated VBR and

5—treated VBR with AuNPs, **c** Statistical analysis on the effect of untreated and treated VBB and **d** VBR by AuNPs on growth of *Vigna unguiculata* (SL shoot length, RL root length, RN number of roots)

Conclusions

The current study explored the catalytic and light absorption properties of biogenic AuNPs for the removal of VBB and VBR. Under the experimental conditions used, 65 and 52% degradation of VBB and VBR was found to take place within a period of 8 h in presence of sunlight. The reaction rate k_{obs} for VBB and VBR was $0.0195 \pm 0.0031/\text{min}$ and $0.0295 \pm 0.0025/\text{min}$, respectively. Remarkably, the phytotoxicity study on *V. unguiculata* revealed non-toxic nature of the degradation product as the growth parameters of plants treated with degradation product and control were comparable. For the effective removal of dyes from textile industry effluents, the method presented here can be used alone or can be incorporated into other methods to develop hybrid systems.

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Compliance with ethical standards

Conflict of interest The authors have declared no conflict of interest.

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