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Nanogold Loaded, Nitrogen Doped TiO₂ Photocatalysts for the Degradation of Aquatic Pollutants Under Sun Light

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1. Introduction

Along with the rapid development of industry, various issues related to energy and environment got generated which are now grown into a significant level. The hazardous waste materials with high concentrations are being discharged directly or indirectly into water bodies without adequate treatment to remove harmful compounds. The World Bank estimates that 17 to 20 percent of industrial water pollution comes from textile dyeing and treatment causing a major global problem. A facile and cheap method for removing inorganic and organic pollutants from wastewater has much relevance in modern world. Dyes are an important class of aquatic pollutants. Its complexity and variety makes it difficult to find a unique treatment procedure that entirely covers the effective elimination of all types of dyes. Particularly, biochemical oxidation suffers from significant limitations since most dyestuffs commercially available have been intentionally designed to resist aerobic microbial degradation and also they may get converted into toxic or carcinogenic compounds. The physical methods such as flocculation, reverse osmosis and adsorption on activated charcoal are nondestructive and merely transfer the pollutant to other media, thus causing secondary pollution (Binitha, 2009, as cited in Belver, 2006) Heterogeneous photocatalysis with various oxide semiconductor photocatalysts is an efficient and rapidly expanding purification technique for water and air. Semiconductor-oxides are a popular class of materials because of their functionalities and applications in the field of photocatalysis and generation of photoelectricity.

There has been greater attention on the photocatalytic activity of nanocrystalline TiO₂ after the discovery of photodecomposition of water on Titania [Ali, 2009; Hao, 2008; Parida, 2007;

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Xiang, 2008; Dambar, 2007; Wang, 2004; Colmenares, 2006; Kolenko, 2005; Shengli, 2006; Baiju, 2007]. Titania (titanium oxide) is considered as one of the most promising heterogeneous photocatalyst owing to the facts such as high photocatalytic activity, strong oxidizing power, low cost, chemical and thermal stability, resistance to photo corrosion and non-toxicity. In addition, TiO_2 possess favorable optoelectronic properties which makes it a well accepted photocatalyst for the degradation of various environmental pollutants (Meenal, 2009; Pirkanniemi; 2002).

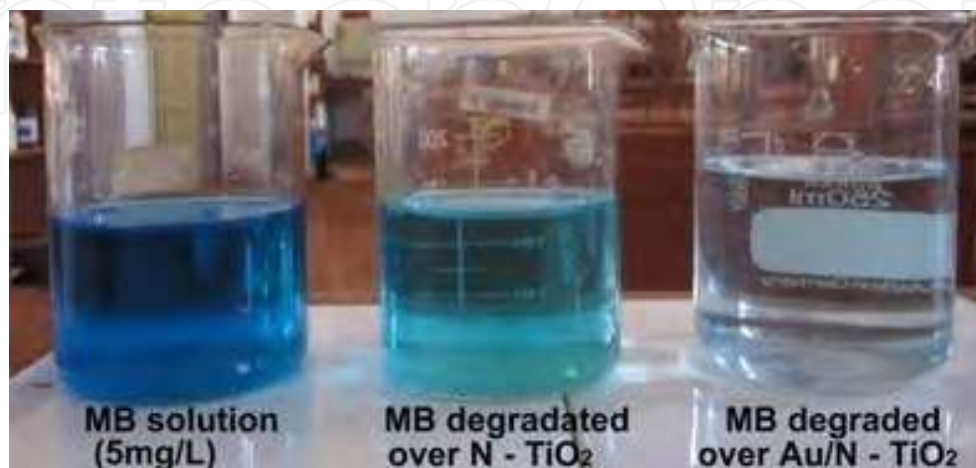


Fig. 1. Degraded methylene blue solution under 1 h exposure to sun light over N doped TiO_2 and Au loaded N doped TiO_2

However, the high intrinsic band gap energies of major crystalline forms of TiO_2 (3.2 eV for anatase phase and 3.0 eV for rutile) makes them effective photocatalysts only when the wavelengths of light is shorter than 387 nm. Thus only a small part of solar light is harvested if we use bare TiO_2 photocatalysts (Haijian, 2008, as cited in Fujishima & Honda, 1972; Li, 2000). It is known that the UV part of the solar spectrum accounts only for about 4% of the incoming solar energy while the major part of the rest is visible light (Binitha, 2010). It is therefore of great significance to adjust the band structure of TiO_2 to improve the photoreaction rate for the efficient use of solar energy for photocatalysis. There are several studies in recent years attempting the incorporation of the visible range of solar spectrum also in the photocatalytic process, which include dye sensitization, metal ion doping, nonmetal doping, etc. (Meenal, 2009, as cited in Choi, 1994; Shockley & Read, 1952; Asahi, 2001). The incorporation of specific dopants in TiO_2 should improve the efficiency of the photocatalytic behavior by creating new band structures or by suppressing the recombination of photogenerated electron-hole pairs resulting in improved quantum efficiency (Zhiqiao, 2009).

The most feasible method for improving the photocatalytic performance of titania are considered as doping with metals as well as non metals. Recent researches concerning TiO_2 -doped with nonmetal elements such as nitrogen (Yohei, 2004, as cited in Sato, 1986; Asahi, 2001; Morikawa, 2001), fluorine (Yohei, 2004, as cited in Hatori, 1998), sulfur (Yohei, 2004 as cited in Umebayashi, 2002) and carbon (Yohei, 2004, as cited in Khan, 2002) have been reported. Among the different anion dopants, nitrogen is observed to be the most effective one and is widely studied.

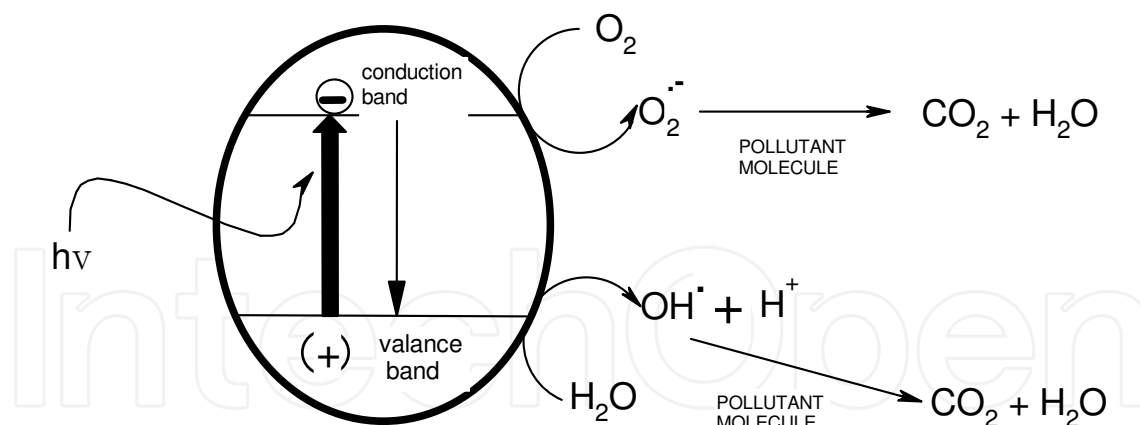


Fig. 2. Band structure of semiconductor titania: Making it suitable for pollutant degradation

Sato (Sato, 1986) was the first person who reported N-doped titania by annealing the mixtures of a commercial titanium hydroxide and NH₄Clor NH₄OH, which showed higher photocatalytic activity in the visible-light region. Asahi et al. (Asahi, 2001) reported that nitrogen-doped titania could induce the visible-light activity in which nitrogen atoms substitute a few oxygen atoms (0.75%), and the doped nitrogen was responsible for the visible-light sensitivity. These two studies had kicked off a new area of research to extend the absorbance of TiO₂ into visible-light region by means of nitrogen-doping. There has been several methods reported thereafter for the preparation of N doped TiO₂, such as high temperature treatment of TiO₂ under NH₃ flow (Hao, 2008, as cited in Asahi, 2001; Irie, 2003; Diwald, 2004), hydrolytic process (Hao, 2008 as cited in Ihara, 2003; Noda, 1986; Salthivel, 2003), mechanochemical (Hao, 2008 as cited in Yin, 2003; Wang, 2004), reactive DC magnetron sputtering (Hao, 2008, as cited in Lindgren, 2003; Chen, 2004), sol-gel (Hao, 2008, as cited in Burda, 2003), solvothermal process [(Hao, 2008, as cited in Aita, 2004), calcination of a complex of Ti ion with a nitrogen containing ligand (Hao, 2008, as cited in Sano, 2004), calcination in nitrogen atmosphere etc.

Among the different preparation procedures, sol gel route is the preferred one because of its endowed nature. The temperatures required for all stages of the process involved in the conversion of sol to gel apart from densification are low, avoiding material degradation and resulting in high purity and stoichiometry of the products. The fact that the precursor metal alkoxides are volatile in general and thus are easily purifiable, substantiates the formation of high purity products. Also, since the organometallic precursors involving different metals are normally miscible with each other, a homogeneous controlled doping can be achieved easily. In addition, during sol gel synthesis the chemical conditions are mild and thus even biological species including enzymes and whole cells may be entrapped retaining their functions after doping on sol gel prepared metal oxides. Besides, the formation of highly porous and nanocrystalline materials can be achieved by sol gel method, by means of appropriate chemical modification of the precursors, adequate control over the rates of hydrolysis and condensation, resulting in the formation of colloid particles of suitable size, porosity and the pore size, and thus achieving a fine control over the pore wall chemistry of the final material. However, there are only a few reports on the anion-doped photocatalysts prepared using wet-methods such as sol-gel and co-precipitation owing to the difficulties involved in the procedure (Hao,2008). Still there are some reports on the N doped sol gel titania which shows good visible light activity for the degradation of pollutants (Dewi, 2010; Hu, 2010; Jian, 2006; Liu, 2005; Min, 2008)).

In the previous studies, the shifting of the absorbance of TiO_2 to visible light upon nitrogen doping is explained in two ways. One proposed mechanism is the narrowing of the band gap by mixing the $\text{N}2p$ and $\text{O}2p$ states. Another explanation is the existence of two absorption edges in the UV visible spectra, the one around 400 nm, the resultant of the band structure of original TiO_2 and second one around 530 nm which is attributed to the newly formed $\text{N}2p$ band located above the $\text{O}2p$ valence band. The incorporation of nonmetal dopant atoms into the lattice structure of titania is believed to decrease the band gap, and shift its response to the visible part of the solar spectrum (Xin & Quingquan, 2008, as cited in Asahi, 2001; Khan, 2002; Ohno, 2004; Lin, 2007).

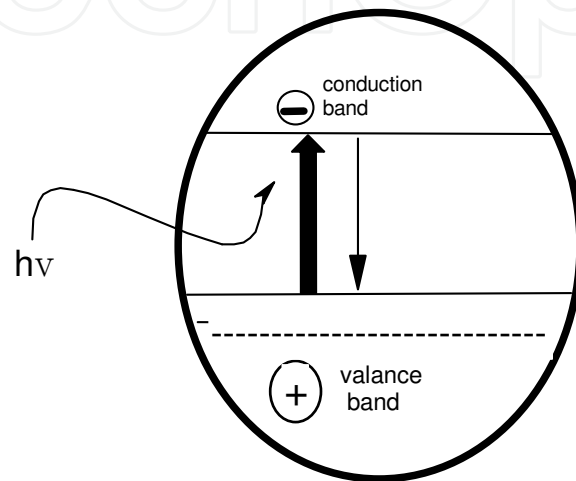


Fig. 3. Schematic picture of reduced band gap of titania as a result of mixing of $\text{N}2p$ and $\text{O}2p$ stages

As clear from the pictorial representation, the hole and electron pair separation is small when the electron is excited by the visible light after N doping and thus they can recombine easily, which will reduce the efficiency of photons. Thus suppression of the recombination of hole-electron pairs is a necessity for visible light active photocatalysts.

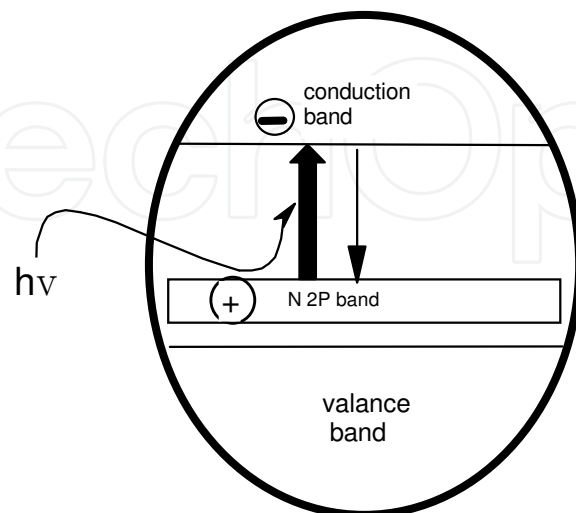


Fig. 4. The schematic diagram representing the shifting of TiO_2 absorption to visible region as a result of the newly formed $\text{N}2p$ band above the TiO_2 valance band

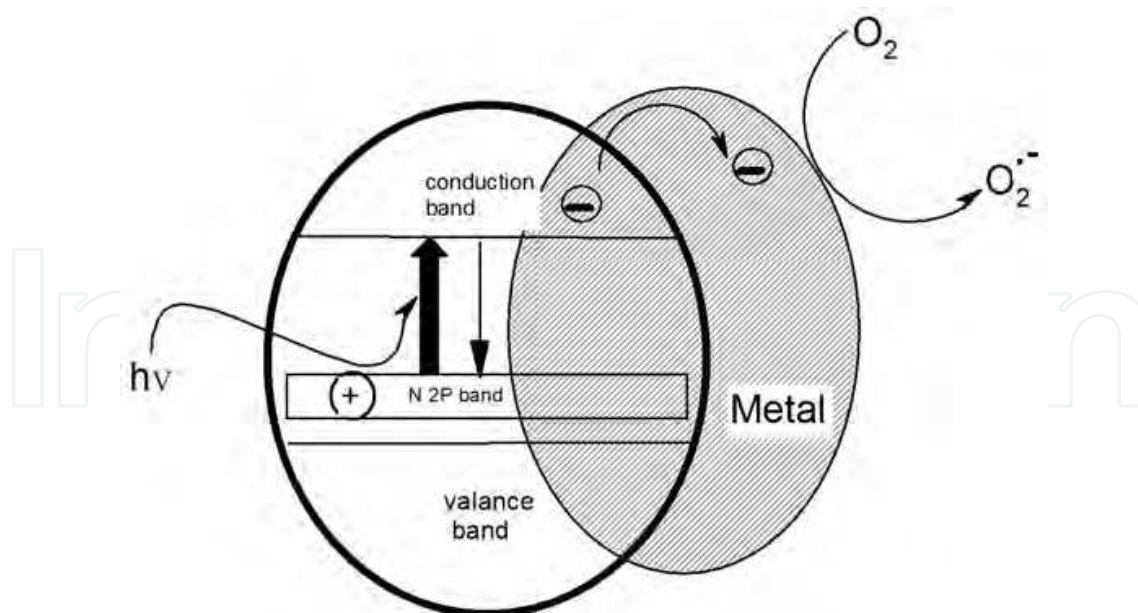


Fig. 5. The schematic diagram showing the role of doped metal nanoparticles in the photocatalytic activity. Metal nanoparticles act as an electron sink, supporting interfacial charge transfer and reduce the chances for charge recombinations.

Doped metal nanoparticles are believed to suppress the recombination of photo-induced electron-hole pairs, when they migrate from the interior of the photocatalyst to the metal surface resulting in increased photo quantum efficiency (Xin & Quingquan, 2008, as cited in Choi, 1994; Lin & Yu, 1998). Thus, metal nanoparticles can be considered as an electron sink, which can promote interfacial charge transfer and consequently less charge recombinations occur.

Thus in the present work, we are looking for a synergistic effect from both dopings. That is the anion doping can provide the narrowing of the band gap and the noble metal nanoparticles are suppose to suppress recombinations. Our objective is the preparation of unsurpassed photocatalysts which are sun light active. Nano gold loaded nitrogen doped TiO₂ photocatalyst was prepared through the sol-gel route using titanium isopropoxide as titanium source, ammonium nitrate as nitrogen precursor and chloroauric acid was used as the precursor for gold nanoparticles. The combined doping of nano gold and nitrogen leads to the high activity of the prepared system for the photodegradation of MB under visible light irradiation. Here, nanogold is observed to provide substantial progress in the photocatalytic activity compared to that of simple nitrogen doped titania for the MB degradation under sunlight.

2. Experimental

2.1 Preparation of photocatalysts

Au-loaded N doped TiO₂ nano powders were prepared by sol-gel process. The sol was prepared following a reported procedure (Sunajadevi & Sugunan, 2004). Titanium isopropoxide (98%, Aldrich) was used as the precursor of TiO₂. 50 ml of Titanium (IV) isopropoxide was hydrolyzed in 600 ml water containing 5 ml nitric acid. Precipitation occurred immediately and the mixture was stirred continuously at room temperature to

form a highly dispersed sol. To this, 4.39g ammonium nitrate was added to get the N-doped titania and stirring was continued for another 4h. The sol was then aged for two days and dried at 70°C. The powdered sample was then calcined at 400 °C for 5h to get the yellow coloured nitrogen doped titania.

Nano gold loading: Different percentage of nanogold are loaded on loaded on N- doped TiO₂ photocatalysts by deposition- precipitation method, which can provide nano-sized gold particles with strong contact of Au particles with the support. Chloro auric acid was used as the gold precursor. The aqueous Chloroauric acid solution(2.1x10⁻³M) is heated to 70 °C. Then the pH of the solution was adjusted to 8 by dropwise addition of 0.5M NaOH solution. The required amount of the support was added in to it with vigorous stirring. The stirring was continued at 70°C for 2h. The pH was maintained as 8 throughout the preparation. It was then cooled to room temperature, filtered and washed with distilled water to make it free from chloride ions. Subsequently it was dried at 80°C for overnight followed by calcination at 200 °C for 5 h. The catalytic systems thus produced are designated as NTiO₂, 1AuNTiO₂ and 2AuNTiO₂ for nitrogen doped TiO₂ with no gold loading, 1% and 2% Au loading respectively.

2.2 Catalyst characterization

XRD patterns of the samples were recorded for 2θ between 10 and 80° on a Bruker AXS D8 Advance diffractometer employing a scanning rate of 0.02°/S with Cu Kα radiation (λ=1.5418). The FTIR spectra were recorded in Thermo Nicolet, Avatar 370 spectrometer in the region 400–4,000 cm⁻¹. TEM photographs of the prepared systems were taken in JEOL JEM 2100 Electromicroscope. SEM pictures are collected using a JEOL Model JSM - 6390LV.

2.3 Photocatalytic degradation studies

Photocatalytic degradation of Methyleneblue (MB) was done by the use of solar energy. All outdoor experiments were carried out in closed Pyrex flasks at room temperature with stirring. The irradiation was performed on sunny days, from 11.00 to 14.00 h when solar intensity fluctuations were minimum. The samples were immediately centrifuged and the quantitative determination of dye was determined using colorimeter (CL 157 - ELICO) before and after the irradiation. Experiments were repeated to get better results. The MB concentration of 5 mg/L mmol at 2h of exposure to sunlight are used for all measurements except for time study.

3. Results and discussion

With an objective to develop a visible light or solar energy responsive photocatalyst, nitrogen doping is done on titania where ammonium nitrate is used as the N precursor. N dopant is added to the stable sol and with gelation the development of a yellow colour is observed whereas introduction of gold changes the colour to violetish ash. With the increase in gold loading, the colour was found to deepen. The different catalytic systems prepared are characterized using various techniques.

3.1 Photocatalyst characterization

It is well established that the electron and hole recombination can be suppressed by increasing the crystallinity of the Semiconductor, minimizing the crystal defects which act as

recombination centers (Tsugio, 2006, as cited in Sclafani, 1990). Our objective here is the preparation of fine particles of small band gap semiconductor with high degree of Crystallinity, which can make photocatalysts that are capable of showing visible light activity. The crystalline nature of the present samples were analyzed using XRD analysis. Both anatase as well rutile phase were visible in the samples where anatase phase was the predominant one. The foremost peak corresponding to (1 0 1) reflections of the anatase phase of TiO₂ was well evident at the angle of 25.28°, as well as the minor peaks were appeared around 37.8°, 48.0°, 53.8° and 55.1°. The major peak of the (1 1 0) diffraction of rutile was observed at the angle of 27.50°, whereas the minor peaks appeared at 36.15°, 41.33°, 54.44°, 56.76°, 62.89° and 69.17°. Weight ratios of each phase were calculated using the following equation:

$$W_r = \frac{A_R}{0.884A_A + A_R}$$

Here A_A represents the integrated intensity of the anatase (1 0 1) peak and A_R the integrated intensity of the rutile (1 1 0) peak (jirapat, 2009, as cited in Gribb, 1997). Increase in gold loading increases the anatase to rutile ratio. This observation is apparently is surprising since we are adding gold precursor to the calcined gel. Thus it is expected that the 2AuTiO₂ can show maximum photoactivity, since anatase is considered as the most photocatalytically active form of TiO₂. TiO₂ obtained by following the same procedure without any N doping is also showing the existence of both anatase and rutile as the crystalline phases (XRD is not shown) (Sunajadevi & Sugunan, 2004). The crystallite size of different systems were calculated using Scherrer equation and the results are provided in table 1.

Catalyst	Weight fractions of phase (%)		Crystal size (nm)	
	Anatase	Rutile	Anatase	Rutile
1% AuNTiO ₂	65.74	34.26	6.17	11.62
2% AuNTiO ₂	86.78	13.22	6.98	7.71

Table 1. Anatase to rutile ratio and crystallite size of gold loaded catalysts

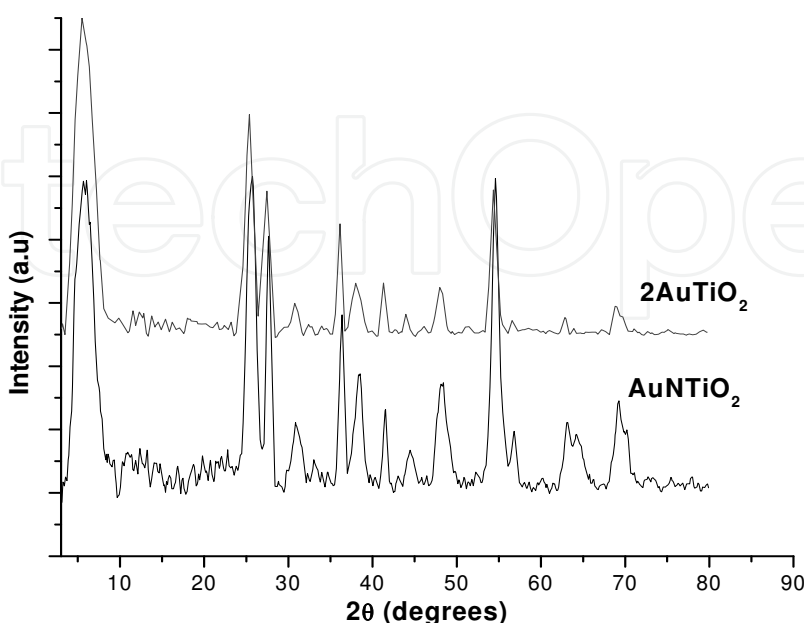


Fig. 6. XRD patterns of different systems

The surface morphology of different systems was analyzed using SEM. In Fig. 7, SEM micrographs of 1AuNTiO₂ and 2AuNTiO₂ are given. All samples appeared as agglomerations of smaller particles with a high tendency for crystallization.

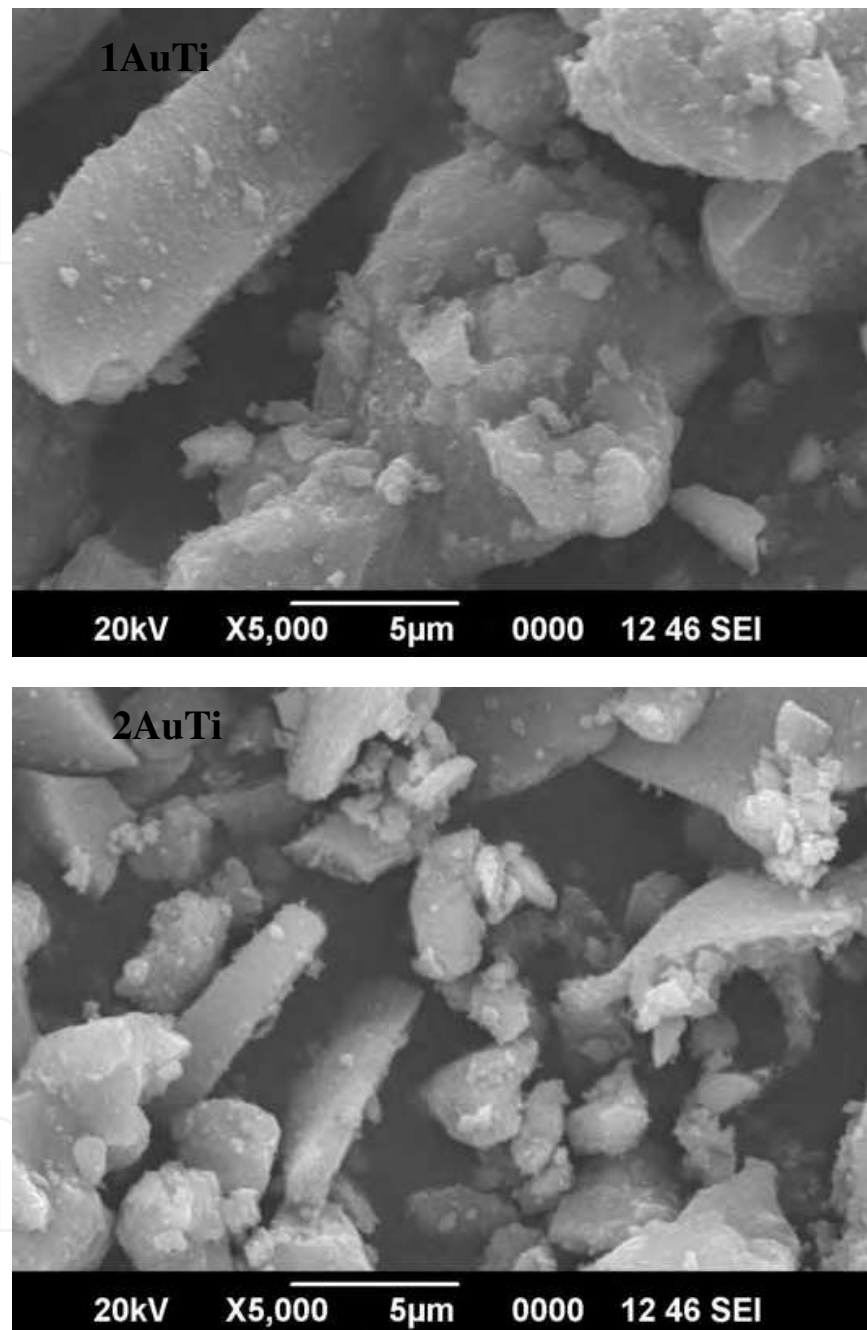


Fig. 7. SEM images of the prepared systems

The TEM micrographs of the photocatalysts show that the gold particles are highly dispersed on the surface of N doped TiO₂ and the mean diameter of gold particles estimated from the TEM images are less than 5nm. In the case of 1AuNTiO₂, only few gold particles are visible and its size is also found to be smaller when compared to that in 2AuNTiO₂. At higher gold loading of 2%, there is competent dispersion for the nanoparticles and the gold particles are visible as sharp dark spots over the gray coloured support. Furthermore, the

lattice fringes of the crystallographic planes of anatase and rutiles are found to be visible, consistent with the XRD patterns. The visible TiO₂ particles are more or less spherical in shape and are found to be of lower size of around 10 – 20 nm diameter

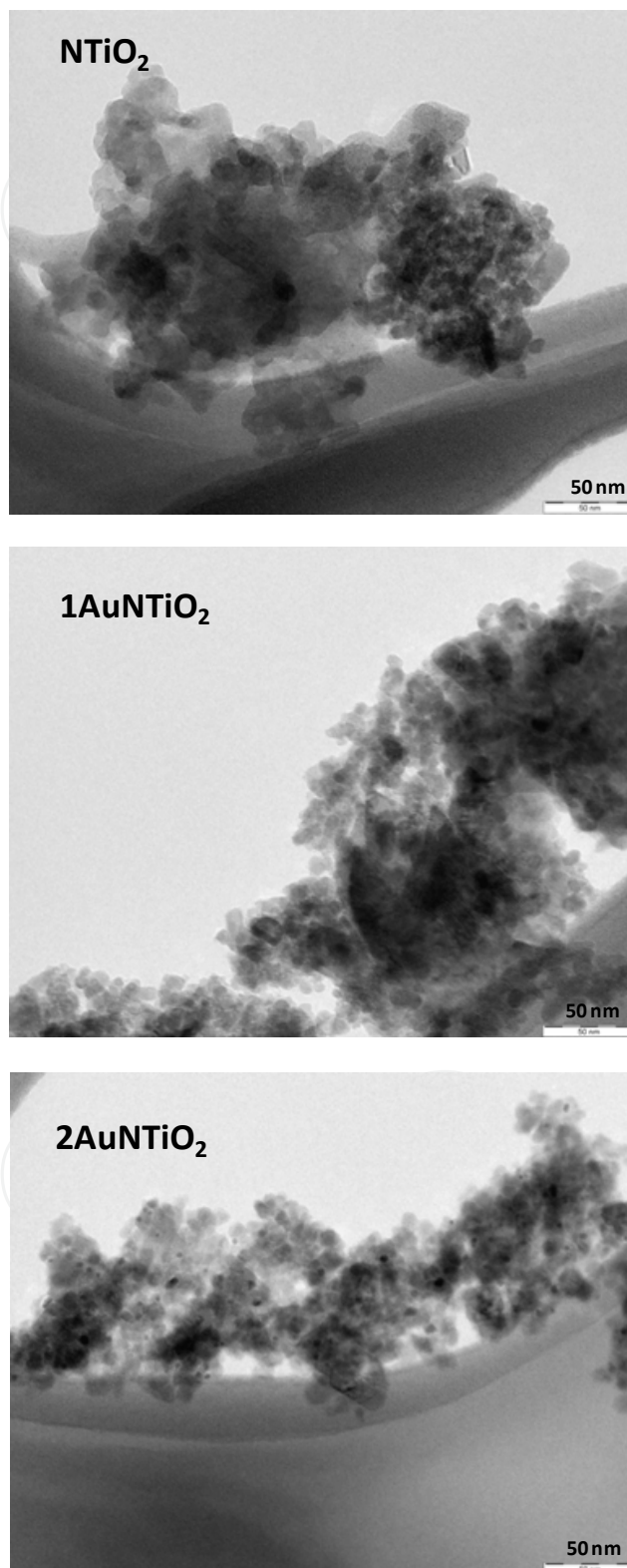


Fig. 8. TEM images of the prepared systems

In the FTIR spectra of the systems shown in Fig. 9, a broad peak seen around 3400 cm^{-1} can be assigned to stretching vibration mode of the OH groups within the TiO_2 sol-gel. The corresponding bending vibration band was observed at 1637 cm^{-1} . The TiO_2 -OH bonds arise from the hydrolysis reactions occurring during the gelling of the titanium alkoxide. In the low energy region of the spectrum, the bands around 500 cm^{-1} can be assigned to bending vibrations of Ti-O bonds.

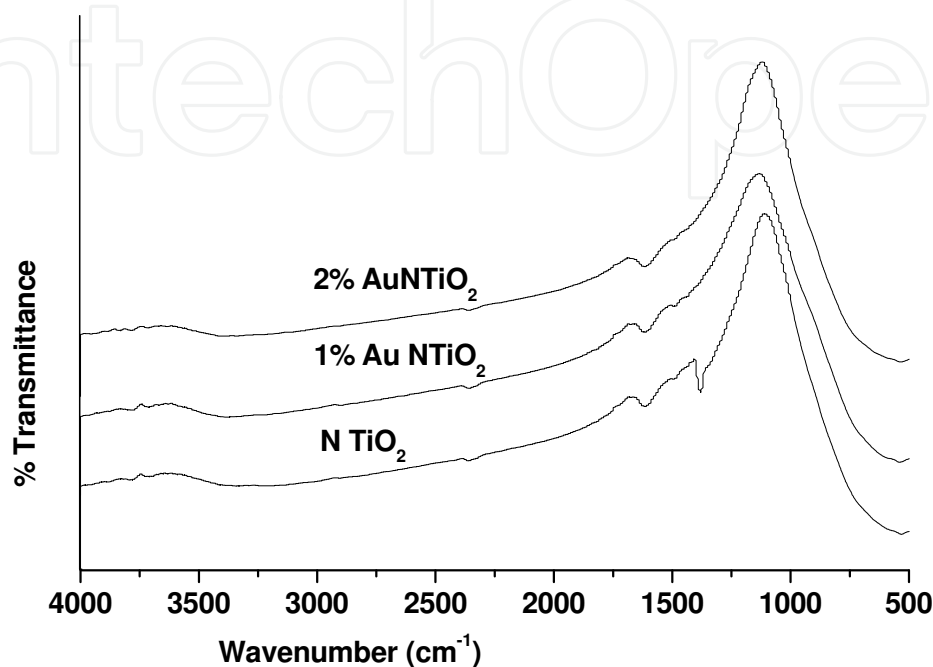


Fig. 9. FTIR spectra of the prepared systems

3.2 Photoactivity results

Photodegradation of MB was done to get the information on the pollutant degrading capability of the present systems. The reaction variables were optimized using 2AuNTiO_2 to achieve the conditions for maximum degradation.

3.2.1 Effect of catalyst dosage

A series of experiments were carried out to optimize the catalyst loading by varying the amount catalyst from $0.05\text{g} - 0.20\text{g}/50\text{ml}$ MB solution of concentration $5\text{ mg}/\text{l}$. The degradation results for 2 h irradiation are shown in Fig. 10. The rate of degradation increased linearly with increase in catalyst weight from 0.05g to 0.10g which then decreases with further increase in the amount of catalyst used. The initial enhancement in photoactivity with catalyst weight may be due to the increase in number of photons absorbed and the number of dye molecules adsorbed on the catalyst molecules. Also the density of the catalyst particles in the area of illumination increases with the catalyst dosage. When the amount of catalyst is exceeding certain limit, the dye molecules available are not sufficient for adsorption and hence the additional catalyst powder is not taking part in the photocatalytic activity and consequently the rate becomes independent of the amount of catalyst beyond certain limit. It is reported that the increase in opacity of the solution at high catalyst dosage decreases the penetration of light inside the solution with a consequent decrease in the photoreduction of the

dye (Binitha, 2011, as cited in Maruthamuthu, 1989). Thus, the optimum amount of catalyst needed to get maximum degradation of pollutant in the present case is 0.1 g.

3.2.2 Effect of volume of MB

The influence of volume of MB on degradation rate was studied by varying the volume of the dye from 25 to 100 ml at a constant TiO₂ loading of 2 g/L for 2 h exposure to sunlight. It was observed from Fig. 11, that the degradation rate increased from 25 ml to 50 ml and then decreased with further increase in the amount of dye. The absorption of light by the pollutant may be dominated at higher volumes which in turn decrease the absorbance of light by the catalyst causing a reduction in the photocatalytic activity.

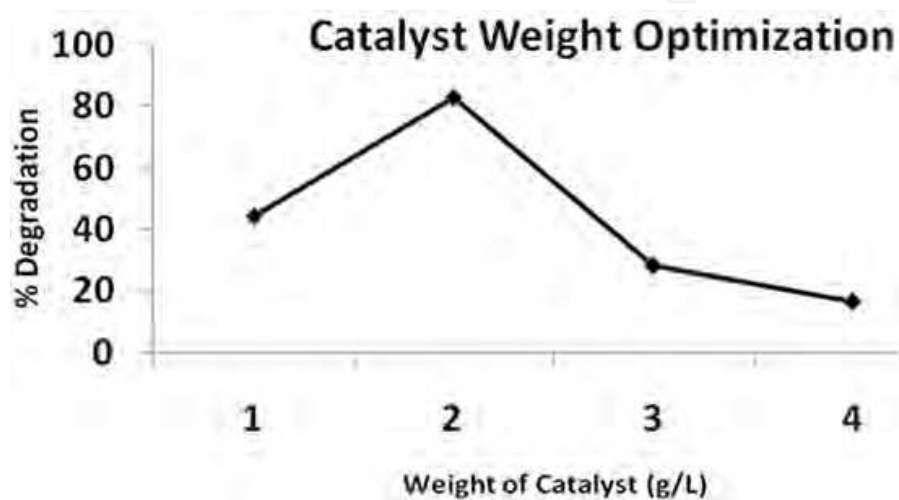


Fig. 10. Effect of catalyst dosage on the degradation of 50 ml of 5 mg/L MB for 2h irradiation

3.2.3 Effect of time

The degradation of 50 ml of 5mg/L MB was investigated by changing the irradiation time from 1 h to 3 h and the activity over the three catalytic systems is plotted in Fig. 12. It is

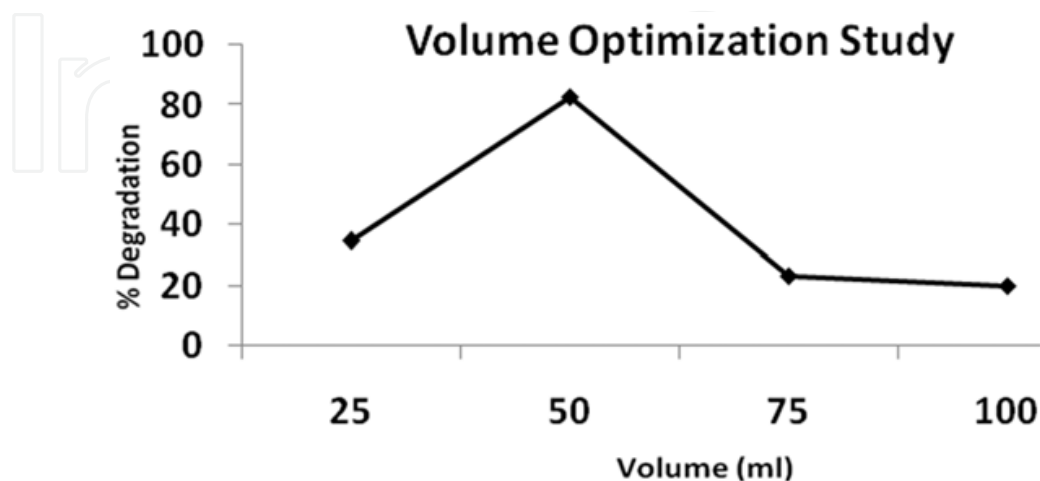


Fig. 11. Effect of volume of MB solution on the degradation of 5 mg/L MB for 2h irradiation using 2g/L catalyst

observed that the degradation reaches 100 % within 3 h of exposure to sunlight in the case of 2AuNTiO₂. This system is thus found to be capable for complete degradation of dye pollutants within this short duration of 3 h solar irradiation. Lower loading of gold (1%) also showed improvement in the photocatalytic activity when compared to N doped TiO₂ without metal nanoparticles. All the three catalytic systems studied showed far better activity than undoped titania which showed very low photodegradation of MB (not included in the figure) , less that 10% even after keeping for 3 h under sunlight.

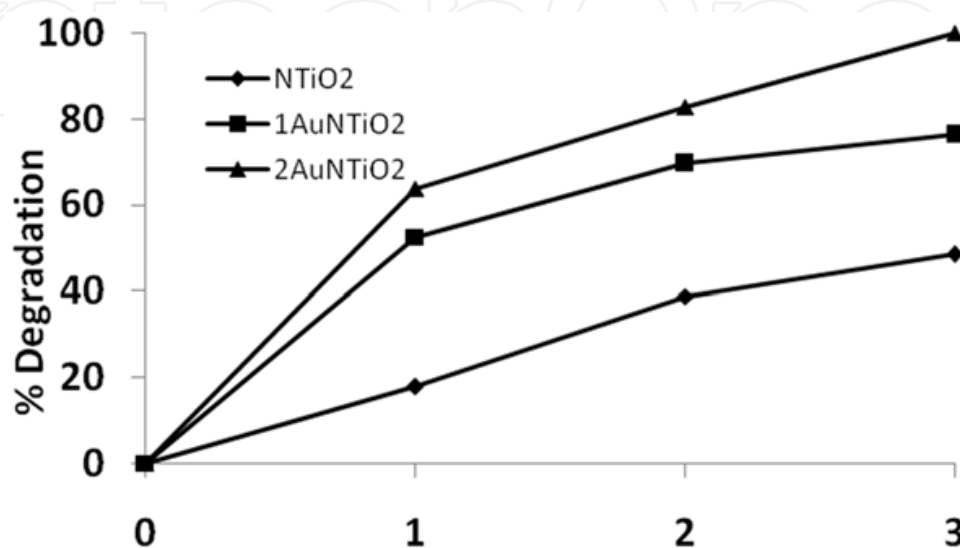


Fig. 12. Effect of Time on the degradation of 50 ml of 5 mg/L MB over a catalyst dosage of 2g/L

4. Conclusion

In conclusion, we are reporting the successful preparation of highly efficient photocatalytic systems, N-doped TiO₂ and Au nanoparticle incorporated N doped TiO₂. The photodegradation of aquatic pollutant methylene blue over N-doped TiO₂ and Au nanoparticle incorporated N doped TiO₂ catalysts were investigated and the catalytic performances were compared. Visible light activity was achieved for TiO₂ upon anion doping. It was found that Au nanoparticle loading over TiO₂ is capable of improving the photocatalytic activity of N doped TiO₂ to a greater extent and the inserted metal nanoparticles are believed to act as electron sinks to prevent the recombination of electron-hole pairs. In addition, X-ray diffraction patterns reveal the suppression of rutile phase with increase in the percentage of gold loading which also can be considered as a favorable factor to obtain superior photoactivity. The inserted gold particles were found to have spherical morphology of less than 5 nm dimension as evident from TEM micrographs. The 2AuNTiO₂ catalytic system was found to be the best among the three showing complete removal of the pollutant within 3 h exposure to sunlight.

5. Acknowledgment

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