

Comparative Studies of Photochemical and Electrochemical Methods on Mineralization of Picric Acid

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ABSTRACT

The effluents generated from synthesis and processing plant of Picric acid is reported to be carcinogenic and hence needs to be treated to reduce its concentration below safe discharge limit. Present paper deals with studies on mineralization of Picric acid by adopting photochemical and electrochemical processes and the efficacy of these processes. In both these processes, effect of Fenton reagent is also studied. In photo chemical process, further effect of TiO₂ photo catalyst with different particle size has also been studied. The treated solution was characterized by using UV-Visible spectrophotometer, High Performance Liquid Chromatography (HPLC) and Total Organic Carbon (TOC) analyzer. The observed results have been interpreted and reported in the present paper. Crucial process parameters in all these processes viz. pH, concentration of oxidizer and catalyst are optimized to achieve picric acid mineralization in reasonable time. It was observed that the time required to bring down the concentration of Picric acid from 100 ppm to less than 1 ppm using photo chemical method was 2 hours whereas this time was reduced to 1 hour using Fenton reagent in photo chemical process. In photo catalysis process carried out using TiO₂ catalyst, it was observed that TiO₂ nano-particles are more efficient compared to that of commercially available micron size TiO₂ powder. To prevent the problem of difficulty in recovery of TiO₂ powder after treatment, nano particle TiO₂ beads blended in Polysulfone were also used. These beads were advantageous in terms of recovery and its performance was comparable to commercial TiO₂ powder. In case of electrochemical treatments at optimised process parameters, picric acid was found completely mineralised to bring its concentration less than 1 ppm using Electro-Fenton reaction picric where as under Electro-peroxide reaction picric acid is mineralised up to 3 ppm after 3 hrs. After studying these processes, it is observed that electro-chemical process is more users friendly and economical; however, photo-Fenton process is more effective in achieving complete mineralization of Picric acid in reasonable time.

Keywords: Effluent; Toxic; Degradation; Photo-catalyst, Electrochemical process; Photo-fenton

1. INTRODUCTION

Picric acid is an important energetic material being used in specialized application. It comes under the class of nitroaromatic compounds. Nitroaromatic compounds are known to be the most extensively used energetic compound in military and other applications because of its thermal and chemical stability & its sensitivity toward impact, temperature and friction¹⁻⁵. It is also reported to be toxic and has mutagenic impacts on different organisms including human beings⁶⁻⁹ and thus designated as a possible human carcinogenic substance by United States Environmental Protection Agency (USEPA). Occupational and incidental exposures to trinitroaromatic compounds result in many types of disorders in living things¹⁰⁻¹¹. Toxic effects of nitroaromatic compounds including disturbance of liver function and anemia in workers have been reported in the large scale manufacturing plants¹²⁻¹³. In view of the above, treatment of effluent contaminated with Picric acid is very important and its recommended safe discharge limit in land water is 1 ppm¹⁴.

Recent developments in the domain of effluent treatment have led to considerable improvement in oxidative degradation procedures for organic compounds dissolved or dispersed in aquatic medium. These methods are generally referred to as advanced oxidation processes (AOP) which is defined as the oxidation process mediated by most reactive free radicals like hydroxyl (OH) radical which is known as one of the strongest oxidants¹⁵. It degrades variety of organic compounds oxidatively leading to innocuous reaction products like carbon dioxide, water etc. Photo-Fenton process¹⁶ and photo-oxidation methods¹⁷ are currently gaining much interest to treat effluents contaminated with nitroaromatic compounds. It involves use of UV light which consumes high electrical energy. In case of photo catalyst the preparation of nanosized photo catalyst is tedious and time consuming works and commercial photo catalyst is not used since it settles down at the bottom of reactor, due to which the efficiency of the process decreases. To resolve the above said problems electrochemical process is adopted to degrade the Picric acid; in this process there is no use of UV, excess chemicals and also preparation of nanomaterial is not required. This process also takes less time to degrade the

nitroaromatic compounds from its initial concentration to its drain discharge limit. There are different means and methods to separate trinitroaromatic compounds from their source one of them is carbon column used for the removal of toxic chemicals from the waste water¹⁸ and carboxymethyl chitosan intercalated montmorillonite (CMCTS-MMT) clay is also used as adsorbent for the removal of cationic dye from the waste water¹⁹.

Another method which is being emerged for treating such effluent is the electrolysis process involving redox reactions. Some reports are available on using this process for oxidation of nitroaromatic compounds but with limited efficiency up to 10 to 15 %²⁰. Gilbert *et al*²¹ have performed experiments with underground water contaminated with trinitroaromatic compounds and succeeded to reduce its concentration from 1 mg/L to 0.03 mg/L in 8 hours. However, the process effluents released from process plants may have much higher contaminant concentration of the order of their solubility limits. Efforts are still continued to find out the methods to achieve maximum degradation of trinitroaromatic compounds within reasonable time. In view of above we have studied the photochemical and electrochemical methods to mineralize Picric acid from 100 ppm to ≤ 1 ppm. In present study under photochemical method i.e. Photo-Fenton and photo catalyst (TiO_2) methods are discussed and findings are reported based on experimental works. We have also studied electrochemical process and optimized the process parameters so as to reduce the treatment time and enhance the efficiency of this process.

2. EXPERIMENTAL DESCRIPTION

2.1 Materials and Methods

Picric acid used in these experiments was from Sigma-Aldrich and of analytical reagent grade. Commercially available anatase TiO_2 (98 %, Aldrich) is used in photo catalysis. This is referred further as Com- TiO_2 . The nano sized TiO_2 has been synthesized by sol gel process using titanium isopropoxide (TIP) as a precursor for TiO_2 . The Titanium (IV) isopropoxide (97 % Aldrich) diluted in propanol (AR grade, Thomas and Bakers) was hydrolyzed under acidic condition to form a gel. The solvent from gel pores was extracted at ambient pressure to obtain nano sized TiO_2 powder. It is referred hereafter as nano- TiO_2 . The beads of TiO_2 have been made using polysulfone (PS). The PS was dissolved in N-Methyl Pyrrolidone (NMP) and to this solution, nano sized TiO_2 was added. The resulting viscous solution was injected to water using 1 mm diameter syringe needle to obtain TiO_2 -PS beads.

The photochemical reactions were carried out in a SS cylindrical reactor equipped with 253.7 nm UV lamp (16 watt) enveloped in quartz tubes axially. Active volume available in the reactor is 1 litre and it has two 2.5 cm diameter openings for liquid circulation. Oxidizer and Fenton reagent are used in stoichiometric ratio. Photo-Fenton reaction has been conducted at $\text{pH} \leq 3.5$ in presence of ozone, hydrochloric acid (AR grade, Thomas and Bekers) and ferrous sulfate (AR grade, Merck) as catalyst. Same setup that is used for Photo-Fenton experiment is also used for photo catalyst (TiO_2 -Polysulphone beads) reaction. Laboratory scale experiments were conducted using this reactor for which more details are reported elsewhere²².

The electrolysis experimental setup consists of Platinum (Pt) electrodes fitted in Perspex which is mounted on top of a glass reactor of 1 litre volume containing Picric acid solution (100 ppm) and Aplab make regulated DC power supply source. Hydrogen peroxide (30 wt % assay, AR grade, Merck) is used for generation of OH radicals and 0.10 M NaCl is added as a strong electrolyte to improve conductivity and the ionic strength; which is optimised by doing a series of experiments and details are mentioned in one of our earlier publication²³. The glass reactor has provision for stirring and purging oxygen and the details can be found in our earlier publication²⁴.

2.2 Analysis

pH is measured by pH meter (Accu pH – 3). Total organic carbon concentration in the treated solution was measured using the ANATOC II SERIES analyzer. The instrument makes use of NDIR as detector. Benzoic acid was used to calibrate the instrument. The treated solution was analyzed by photo absorption measurement in UV-Vis region using UV 3000+ spectrometer, LABINDIA, India. Picric acid concentration in the treated solution was measured by using HPLC (YL-3000) instrument equipped with a HIQ Sil C18W column of size 4.6 mm ϕ x 250 mm and UV detector. For the elution methanol: water (50:50 % v/v) was used as the mobile phase with a flow rate of 0.5 ml/min at $\lambda=254$ nm for the UV detector.

3. RESULTS AND DISCUSSIONS

3.1 Photochemical Reaction Carried out Using UV+ H_2O_2 + FeSO_4

The UV-Visible spectral analysis of the aqueous Picric acid solutions, UV photolysed for different extent, indicates gradual degradation of the Picric acid followed by the formation of intermediate byproducts having absorption in the UV region. The final spectrum after 60 minutes of irradiation indicates in degradation of Picric acid and observed formation of byproducts. UV-Visible spectrum of aqueous 100 ppm Picric acid solution photolysed for different time intervals is shown in Fig. 1.

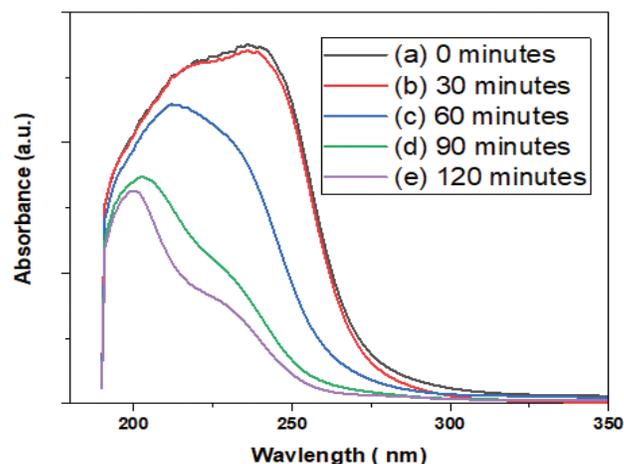


Figure 1. UV-Visible Spectrophotometer analysis of UV photolysed aqueous Picric acid solution at different time interval of: (a) 0 minutes, (b) 30 minutes, (c) 60 minutes, (d) 90 minutes, and (e) 120 minutes.

Photo-Fenton process is also carried out in the presence of FeSO_4 with H_2O_2 . It is an eco-friendly process mediated by the strong OH radical intermediates. For this purpose the pH of the water should be adjusted in acidic region with the help hydrochloric acid. This is necessary to keep the ferrous salt catalyst of the reaction in soluble form. Hydrogen peroxide is the powerful oxidant of the reaction and should be used in stoichiometric ratio.

The treated samples are qualitatively and quantitatively characterized for Picric acid degradation using HPLC. In the present case ODS is used as stationary phase and 50:50 (MeOH: Water) is used as mobile phase. The retention time for Picric acid is 8.95 minutes by keeping the flow rate of mobile phase at 0.5 ml/min. HPLC chromatograms of the aqueous 100 ppm Picric acid solution treated at pH of 3.5 on UV irradiation for different interval of times from 30 minutes to 2 hours as shown in Fig. 2, which indicates that the UV exposure in presence of oxidizer (H_2O_2) and Fenton reagent (FeSO_4) of 30 minutes results in the oxidation of Picric acid and formation of certain byproducts while at 2 hrs of irradiation almost complete degradation of Picric acid and without any byproducts are observed.

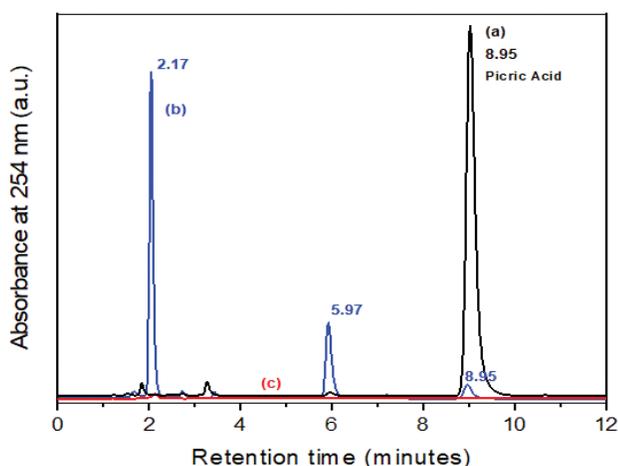


Figure 2. HPLC analysis of UV photolysed in presence of (oxidizer and Fenton reagent) Picric acid solution at different time: (a) 0 minutes, (b) 30 minutes, and (c) 120 minutes.

3.2 Photochemical Reaction carried out using Photo Catalyst

Using TiO_2 as a photo-catalyst is well known to enhance the photolytic degradation of organic pollutants. Therefore, the photo catalysis degradation experiments have been attempted in the presence of TiO_2 in different size and forms. A known quantity (1 gram) of Com- TiO_2 powder (size 20 to 30 micron) was dispersed in one litre of 100 ppm Picric acid solution and solution was exposed to UV light. The dispersion of this Com- TiO_2 was not found to be homogeneous due to settling of some powder. In case of nano- TiO_2 synthesised from Titanium Isopropoxide (TIP) by sol-gel method, the settling of catalyst powder was reduced to some extent due to colloidal range particle size. Results of the degradation studies are shown in Fig.3. Efficiency of nano- TiO_2 is found to be better than that of

the Com- TiO_2 . In both the cases almost complete degradation of Picric acid was observed while the extent of degradation of the byproducts was found to be more in the case of nano- TiO_2 . It is obvious that nano- TiO_2 prepared by sol-gel method has high specific surface area that is found effective in catalysis. It is also known that band gap energy is also more in case of nano particles which might have also helped to avoid electron-hole pair recombination in TiO_2 photo catalysis and effective reaction of these species with organic molecules.

For efficient utilization of this method, the TiO_2 particles need be separated from the reaction mixture so that it can be recycled and can be used in multiple experiments. Separation of the Com- TiO_2 and nano- TiO_2 from the treated solution is difficult due to fine particle size. For easy separation of the TiO_2 beads of TiO_2 with polysulfone (1-2 mm in size) were prepared and tested for photolytic degradation. The efficiency of these beads is found less than nano- TiO_2 . It was expected as the surface area of nano- TiO_2 could have reduced due to polymer blending. However, TiO_2 -polysulfone (PS) beads can easily be separated from the reaction mixture and can be used several times.

To assess the degree of degradation of Picric acid solution after photo catalysis treatment, experiments were carried out using Com- TiO_2 , nano- TiO_2 and TiO_2 -PS beads. Total Organic Carbon measurements of treated sample are shown in Fig. 3. The TOC value for untreated sample was recorded as 30 ppm which was reduced to less than detectable limit within 1 hour for nano- TiO_2 . The TOC value for sample treated with Com- TiO_2 for one hour was 4 ppm. These experiments were repeated for 5 times and on statistical average, it is found that the TOC values measured at different time interval and the deviation from mean observed value was found to be within $\pm 2\%$.

It is observed that nano- TiO_2 reduced the TOC concentration of picric acid more than TiO_2 -PS beads and Com- TiO_2 . By comparing the degradation efficiency it is imperative that photo catalyst nano- TiO_2 is superior over TiO_2 -PS beads.

It is also observed that nano- TiO_2 and TiO_2 -PS beads are more efficient compared to Com- TiO_2 in terms of fast mineralization of picric acid. The TiO_2 -PS beads show less efficiency than nano- TiO_2 . However, the Picric acid oxidation efficiency for TiO_2 -PS beads is comparable with Com- TiO_2 and

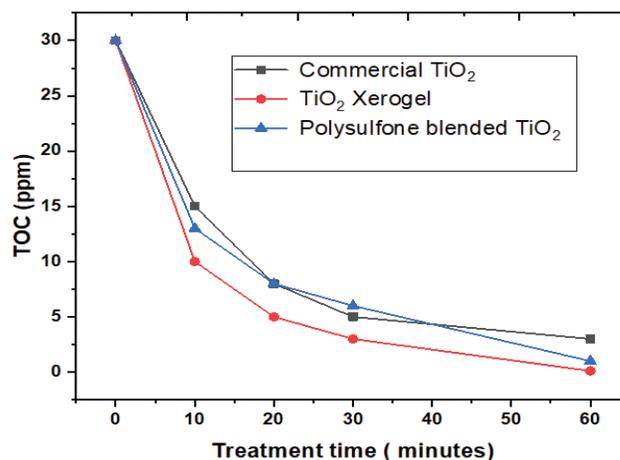


Figure 3. TOC of photocatalysed aqueous Picric acid as a function of treatment time.

advantage of TiO₂-PS beads is that it can easily be recovered from the treated solution. Therefore, the beads made of nano-TiO₂ in polysulfone are promising candidate for treating the Picric acid effluents.

3.3. Electrochemical Treatment of Picric Acid

Under electrochemical treatment of Picric acid from 100 ppm to its drain discharge limit, electro-peroxide and electro-Fenton methods are explored. In case of electro-peroxide reaction strong electrolyte and hydrogen peroxide are used; whereas in case of electro-Fenton reaction strong electrolyte, hydrogen peroxide and ferrous sulphate are used to oxidize the picric acid from 100 ppm to less than 1 ppm. Complete mineralization of Picric acid by the oxidation of organics produces carbon dioxide and water or other oxides. Therefore, no generation of secondary pollutants takes place during electrochemical treatment of picric acid. Electro-oxidation involves chlorine species (Cl₂, HOCl and ClO⁻) generated during electrochemical process which are mainly responsible to mineralize Picric acid during electrochemical treatment.

A series of experiments have been carried out to optimize the process parameters to mineralize the Picric acid from 100 ppm to its drain discharge limit within 3 hrs. It was observed in the present study that catalyst concentration of 50 ppm, oxidizer of 30 mM and pH of 3 are optimal parameters to mineralize the picric acid. With optimized value of process parameters, these experiments were recreated for 5 times and the averaged results are presented in Fig. 4. The concentration values averaged over 5 experiments are within range of $\pm 3\%$ from mean value. From Fig. 4 it is observed that Electro-Fenton reaction seems comparatively much faster than Electro-peroxide reaction during the mineralization of picric acid. It was also observed during the Electro-peroxide reaction, the solution became turbid in the initial phase of the reaction due to formation of some acids during mineralization of picric acid. Formation of turbidity and the absence of iron salt are the main reasons for the slower rate of mineralization for picric acid under Electro-peroxide reaction. In case of Electro-Fenton reaction

the regeneration of ferrous ion results in higher concentration of OH radicals which accelerates the rate of mineralization of picric acid. Decarboxylation and higher concentration of OH radicals in presence of ferrous ion are two crucial conditions under Electro-Fenton reaction which are responsible for the fast mineralization of picric acid within 3 hrs. It is also observed from Fig. 4 that under Electro-Fenton reaction picric acid is completely mineralised below its drain discharge limit where as under Electro-peroxide reaction, picric acid is mineralized upto 3 ppm within 3 hrs.

4. CONCLUSIONS

Photochemical and electrochemical methods have been adopted to mineralize Picric acid from 100 ppm to its drain discharge limit (≤ 1 ppm). Under photochemical method, Picric acid is treated using UV+H₂O₂+FeSO₄ (Photo-Fenton) and UV+TiO₂ (Photo-catalyst) at optimized process parameters. It is observed that Picric acid is treated from 100 ppm to ≤ 1 ppm by using photo-Fenton and photo-catalytic processes in 2 hrs and 1 hr, respectively. It is also observed that TiO₂ xerogel and beads of nano-TiO₂ are more efficient than commercially available TiO₂ and also TiO₂-PS beads show less efficiency than TiO₂ xerogel. Electrochemical treatment is carried out with Pt electrode at optimised process parameters viz. catalyst concentration of 50 ppm, oxidizer of 30 mM and pH of 3; it is observed that under Electro-Fenton reaction picric acid is completely mineralized where as under Electro-peroxide reaction picric acid is mineralized upto 3 ppm within 3 hrs. Electrochemical is user's friendly and Nobel method to mineralize Picric acid from 100 ppm to its drain discharge limit with in 3 hrs. Lab scale optimised process parameters will be used for scaling up the process for energetic industries. We have demonstrated a simple, convenient and cost effective method for complete degradation of Picric acid effluent.

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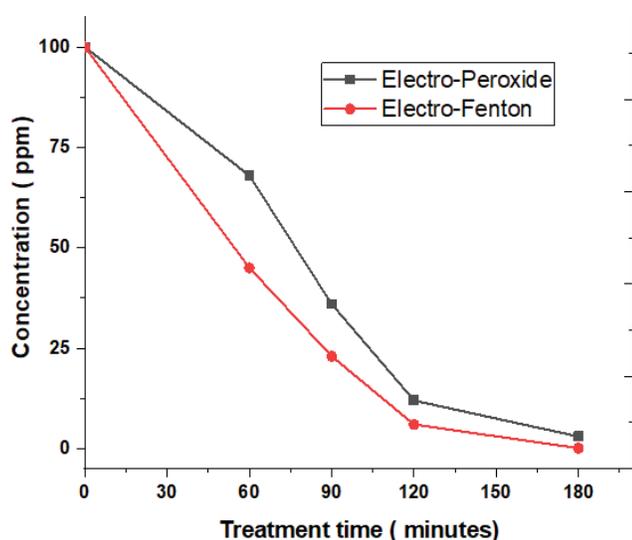


Figure 4. Concentration of Picric acid as a function of treatment time.

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Dr Ratanesh Kumar obtained his MSc in Chemistry from MKU in 2011 and PhD in Chemical Sciences from Homi Bhabha National Institute, Mumbai in 2022. Since 2005, he is working in Applied Physics Division, BARC, Mumbai. Dr Ratanesh Kumar is Technical Officer 'D' in BARC and is involved in development of electrolytic technique for treatment of non-biodegradable toxic effluents. In the present study, he carried out experimental works to optimise the process parameters for efficient and effective degradation of picric acid. He analysed the treated samples by various analytical techniques to support his observations.

Dr P.B. Wagh obtained his MSc in 1993 and was awarded PhD (Physics) degree in 1999 from Shivaji University, Kolhapur. He joined BARC in 2002. Presently, he is working as Scientific Officer 'G' in Applied Physics Division, BARC. His main areas of research include 'Aerogel materials' and 'Energetic materials'. He has to his credit more than 100 research publications in cited international and national journals. He is life member of High Energy Materials Society of India. In the current study, he proposed idea of electrochemical and photochemical treatment and guided the experimental works.

Dr Sanjay V. Ingale has completed his MSc from Shivaji University, Kolhapur and PhD from HBNI, Mumbai. Since 2002, he is working in the Applied Physics Division, BARC. He is involved in development and processing of nano structured energetic materials. In the current study, he participated in characterization of treated samples by various techniques.

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