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COMPARISON OF EFFICIENCY OF APM AND MNO_2 BY PHOTOCATALYTICAL DEGRADATION OF SARANINE-O BASED ON QUALITY PARAMETER MODIFICATION

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Abstract

A semiconductor mediated photocatalytic treatment is preferred due to low operating cost and effective treatment to dyes polluted effluent. But sometimes it can generate even more toxic dye intermediate than parent dye molecule. Before the treated water is discharged into ecosystem, it should be mandatory to analyse it according to quality parameters. Present investigation analyses the use of Ammonium phosphomolybdate and MnO_2 with sunlight irradiation for photo catalytic degradation of Safranin O dye contaminated water. Safranin O dye has wide application in counter staining nuclei red and applied to Cotton, fibres, leather, wool, silk and paper. Surplus and used dye is discharged in sink drain which cause hazardous and toxicity concern to ecosystem due to stability of dye and negative modification in quality parameters. When Safranin-dye solution (polluted water) was treated by APM and MnO_2 , significant changes were observed in pH, Alkalinity, Hardness, COD, BOD, DO, Conductivity, TDS and concentration of Ca^{+2}, Mg^{+2}, Cl^-, F^-, NO_3^-, SO_4^{-2}, Turbidity and Colour disappearance. Present paper analyse the comparison of photocatalytic activity of APM and MnO_2 based on quality parameters modification which occur in photocatalytic treatment of Safranin O dye.

Keywords- Safranin O, Photocatalysis, APM, MnO_2, Quality parameters

Introduction

Water is the prime natural resources and basic need of human and all other living being. It is valuable national asset and required in all aspects of life like agriculture, industrial activities, energy conservation and substance of life and development. In recent years, many natural and synthetic pollutants have deteriorated quality of water and they are not usually monitored and controlled, although they are known or suspected to destroy the ecological health. Modern synthetic dyes are essential in our routine life but due to its stability, toxicity and hazardous nature. These corrupted the valuable water sources. Much

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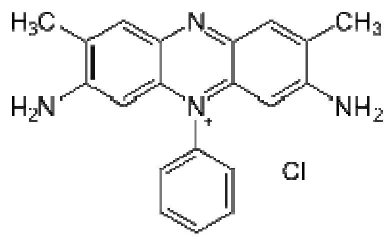
hyped commercial dyes are drawing attention of scientist across the world for pollution, degradation and toxicity investigation[1]. Dyes, used in research and medical sector have been paid poor attention in this context, as compared to industrial dyes. Net consumption of dyes used in medical and research sector is comparatively lesser than textile, paper and leather industry. But these dyes also have risk in disposal without checking bio-safety and water quality parameter. Presently photo-catalysis is a promising technology with the use of unlimited sunlight sources and little amount of semi-conductor. Therefore it is a part of green chemistry where catalyst used visible light and converts harmful dyes into lesser harmful products. In recent years, a lot of work has been carried out on semi-conductor mediated photo-catalytic degradation of different dyes under different values of pH, amount of particular semi-conductor, dye concentration and hence optimum condition will choose for optimum photo degradation. Many studies have been focused on this, like Qu et.al[2], Chen et.al[3], Abo farah et.al [4], Gullard et.al.[5] used TiO_2 and poulios et.al[6], Movahedi et.al[7] used ZnO, both are extensively used semi-conductor. Expect both Fe_2O_3 [Ameta et.al[8], Bhardwaj *et.al*[9], CdS [Devis *et.al*[10]and Reutergardh *et.al*[11], MnO_2 [Ameta *et.al*[12], Mittal *et.al*[13], Chaturvedi *et.al*[14], Pastor *et.al*.[15]and Ammonium phosphomolybdate (Bansal *et.al*[16], Sharma *et.al*[17, 18] Sachdeva *et. al*.[19, 20]) are also generally used semi-conductor to degrade different dyes like Azure-B, fast green, Rhodamine 6-G, Janus green B, Rose-Bangal, Methylene Blue, Erichrome black tea etc. Few researches have been carried out on the comparative study of dye degradation through different semi-conductor. Gandhi *et.al*.[21] worked on comparative study of ZnS and CoS-ZnS(1:2) in photobleaching of Azure-B. Ameta *et.al*[22] compared the efficiency of PbO_2 and HgO to degrade Rose Bengal. Safranin O is heterocyclic azine group of dye, which is derivative of phenazine. It is a basic dye which is applied to wool, silk or tannin mordant, cotton, fibres, leather and paper. Safranin O is most commonly used as a biological and medical stain used in cytology and histology. It shows acute toxicity, irritation in eyes, respiratory system and skin. This dye was degraded by Chaturvedi et.al. Using NiO[23] and MnO2[14] semiconductor for photodegradation and Sharma *et.al*.[24] also degraded it by APM.



Structure of Safranin O

Molecular formula: $C_{20}H_{19}ClN_4$
Molar mass: 350.84 g/mol
Solubility: Water

Most of such semiconductor mediated photocatalytic treatments remain silent over water quality parameter, eco-friendly and toxicity concern. So before this degraded water is released into ecosystem, it must be confirmed whether water quality parameter lies in permissible limit. According to these parameters treated water can be used for irrigation, wild life or drinking purpose. Even though Bharadwaj et.al. Used TiO_2[25] and Fe_2O_3[9] as hetrocatalyst to degrade Giemsa dye and Congo red dye respectively and compared the quality parameter of dye contaminated water and treated water. They analyzed toxicity concern by seed germination and executed bioassay of treated and untreated water. Tiwari *et.al*.[26] also compared polluted water (Cresol red dye contaminated water) and treated water (by ZnO) on the basis of quality parameter modification. Significant changes describe whole analysis of water which declared where it can be used, in irrigation, household use, drinking or in animal and aquatic system. Proposed investigation will provide a pathway to choose better semiconductor based on quality modification. It will also indicate how photocatalyzed dye product affect ecosystem and whether photocatalytic water treatment methodologies may be appropriate up to an extent. We have made a first ever attempt to compare the efficiency of semiconductors (APM and MnO_2) which worked on better photocatalytic treatment of Safranin O dye contaminated water based on quality parameter analysis. Quality of APM mediated photocatalytically treated water and MnO_2 mediated photocatalytically treated water was compared with own parent SO dye solutions by analysing pH ,Turbidity, TDS, Conductivity, Salinity, Alkalinity, Hardness, Concentration of Ca^{+2}, Mg^{+2}, F^-,Cl^-, NO_3^-, $SO4^{-2}$, DO, BOD, COD and this quality factor was compared with WHO, IS standards for drinking and other uses.

Experimental approach

Chemicals

Safranin O(Sigma), Ammonium phosphomolybdate (APM) (Himedia) , Mangnese (IV) oxide (MnO_2), Sodium Fluoride, Zirconium Reagent, Sodium Arsenite, Sodium Azide, Sodium Iodide, Hypo (Na2S2O3), Erichrome Black-T Indicator, Methyl Orange Purchased From Qualigens. Murexide Indicator, Ferrous, Ammonium Sulphate from Fisher Scientific. $AgNO_3$, AgCl, Silver Sulphate, Mercuric Sulphate, EDTA, $K_2Cr_2O_7$, Magnesium Sulphate, HCl, NaOH were Purchased from CDH.

Apparatus

Systronics Spectrophotometer 106, Water Analyser 371, Digital pH Meter 335, PC Based Double Beam Spectrophotometer 2202, Citizen Balance

Method

Water samples were collected from the pond of the Keoladeo National Park situated in Bharatpur District. The entire physio – chemical quality parameters of water samples were determined for comparative Reference.

0.0350 gm of Safranin O was dissolved 1000 ml in KNP pond water samples to prepare stock solution (1x10-4 M). This stock solution was diluted to 1000 ml using water samples to prepare 3.2x10-5 M dye solution which was considered as polluted water. This water was further divided into two equal parts. One part of dye solution of 3.2x10-5 M was safely placed in borax glass beaker and exposed to sunlight for 4 hours with 1 gm APM at 10.5 pH in control condition for optimum PCD according to Sharma *et al.*[25]. After 4 hours this treated water was centrifuged to sediment the APM with using a G-3 sintered glass crucible. The remaining solution was considered as treated water.

Photo catalytically treated and untreated water samples were analysed for-

pH - Digital pH Meter 335

Turbidity, TDS, Conductivity – Water Analyser 371

Alkalinity, Hardness, Concentration of Ca^{+2}, Mg^{+2}, F^-, Cl^-, NO_3^-, SO_4^{-2} , DO, BOD, COD as per method assessment of water, sewage and industrial effluent[27].

Same procedure is repeated by using MnO_2. [30] ml of stock solution was diluted to 1000 ml to prepare 3.0x10-6 M dye solution. This solution photocatalytically treated with 5 gm MnO_2 at 11 pH exposed under sunlight for optimum degradation according to Chaturvedi et al.14 After 4 hours this treated water was centrifuged to sediment the MnO_2 with using a G-3 sintered glass crucible. Now remaining solution considered as treated water. Photo catalytically treated and untreated water samples were analysed for already discussed all quality parameters.

Result and discussion

For maximum photocatalytic degradation of Safranin O by two different semiconductor means APM and MnO_2, all optimum condition such as pH, Concentration of dye, amount of both semiconductor APM and MnO_2 used in present research according to Sharma et. al. and Chaturvedi *et. al.* In presence of sunlight and semiconductor Ammonium phosphomolybdate and Magnese (IV) oxide photo catalytic treatment affect the Quality parameters.

The results are in table 1.

Table -1 Comparative Analysis of quality parameters Parameter

Parameter	Safranin-O dye solution I	Safranin-O dye solution I treated by APM	Safranin -O dye solution II	Safranin-O dye solution II treated by MnO_2	WHO Standard
pH	9.5	8.3	8.45	7.2	6.5-8.5
Alkalinity (mg/L)	440	304	290	270	200
Hardness (mg/L)	330	280	280	220	300
Calcium (mg/L)	210	140	150	120	75-200
Magnesium(mg/L)	120	140	130	100	30-100
Chloride (mg/L)	780	850	830	710	250
Fluoride (mg/L)	1.5	2.2	1.5	1.8	1-1.5
Sulphate (mg/L)	138	148	160	128	200
Nitrate (mg/L)	4.43	15.505	8.86	2.215	45
DO (ppm)	11.2	6.7	9.7	10.5	5
BOD(ppm)	1.2	0.7	2.7	5.3	6
COD (ppm)	34.84	18.25	37.44	11.52	10
Conductivity(µS/cm)	778	1120	786	842	1000
TDS(ppm)	403	600	418	447	500-2000
Turbidity(NTU)	1.9	3.9	0.61	0.26	5

Effect on pH

pH of water samples denotes the extent of its pollution by acidic and alkaline wastes. All chemical and biological reaction directly depends upon the pH of medium. Safranin O dye is basic azo dye in nature and it increases pH 7.81 to 9.5 (Safranin O dye I 3.2x10-5 M) and 8.45 (Safranin O dye II 3.0x10-6 M). After treatment pH was reduced to 8.3 and 7.2 respectively. It is clear that photocatalytic treatment affects the pH and makes it more desirable. Both dye contaminated solution are not in permissible limit which is not suitable for irrigation and drinking purpose according to WHO standard, whereas pH of photocataltically treated water by APM and MnO_2 are suitable for drinking, irrigation, animal and aquatic biota. From them MnO_2 mediated treatment more effectively reduced pH and takes it within range.

Effect on Alkalinity

Alkalinity is important as an indicator of water body's ability to resist pH change with the addition of acid from an accidental spill or acid precipitation[28]. Ability to neutralize acid is generally due to carbonate, bicarbonate, hydroxide ions; sometimes it includes Borates, Phosphate, Silicate or other bases. Alkalinity of Safranin O dye solution I (3.2x10-5 M) noted at 440 ppm which reduced to 304 ppm after photocatalytic treatment by APM. On other side, Alkalinity was found approximately same in polluted water (3.0 x10-6 M) and treated water by MnO_2, means value change 290 ppm to 270 ppm. All water samples have high alkalinity value than WHO standard for drinking purpose.

Effect on Hardness

Hardness is deemed to be the capacity of water for reducing and destroying the lather of soap. Temporary hardness is caused by calcium bicarbonate and boiling converts it into insoluble carbonate. Hardness from other salts is called permanent hardness. Ca and Mg are principle cations causing hardness. In Safranin O dye solution I and II hardness was measured at 330 ppm and 280 ppm respectively. After photocatalytic treatment by APM, Safranin O dye I hardness value reduced to 280 ppm and MnO2 reduced Safranin O dye II value to 220 ppm. Total hardness is lower than alkalinity which shows that neutral salt of Ca^{+2}, Mg^{+2} ion are carbonate not sulphate.

Effect on Calcium

The presence of calcium in water is mainly due to its passage through or over deposits of lime stone, dolomite, gypsum and gypsiferous materials. High levels of calcium ion promote scale formation in water system. Even though, the human body requires approximately 0.7 to 2.0 g of calcium per day as a food element29. Calcium was found to 210 ppm and 150 ppm in Safranin O dye solution I and II respectively. APM mediated photocatalytic treatment largely reduced calcium ion concentration of Safranin O dye I (3.2x10-5 M) to 140 ppm. However, MnO2 mediated photocatalytic treatment was not so much effective which also reduced calcium level of Safranin O dye II (3.0 x10-6 M) to 120 ppm.

Effect on Magnesium

Magnesium is an essential element for human being and normal plant growth. It is relatively non-toxic to man but at higher concentration causes unpleasant taste to water, laxative effect, and act as cathartics and diuretics among animals as well as human beings. Magnesium level in Safranin dye solution I was found at 120 ppm which increased to 150 ppm when it is photocatalytically treated by APM. Whereas MnO_2 mediated treatment reduced magnesium level from 130 ppm of Safranin dye solution II 3.0 x10-6 M to 100 ppm. Except treated water by MnO_2 all water samples are above magnesium permissible limit.

Effect on Chloride

Reasonable amount of chloride is generally not harmful to human beings, however higher concentration with cations likes Ca, Mg, Na, K produces harmful effect to human, agriculture sector and accelerates the rate of corrosion of steel, stainless steel alloys and aluminum. All water samples are alkaline in nature and hence, chloride was found in excess. One side APM mediated treatment increase chloride level in Safranin dye solution I, where chloride concentration increases 780 ppm to 850 ppm. On other side MnO_2 mediated treatment decreased it, where chloride concentration reduced 830 ppm to 710 ppm.

Effect on Nitrate

Nitrogen as in form of Nitrate plays a vital role in plant growth, hence effect food chain. Nitrates are found in trace quantity in surface water and soil which are enhanced by fertilized farm soil, industrial effluents and animal wastes. Where below the standard limit rise in NO_3^- concentration stimulate plant growth, on other side high level in water causes methemoglobinemia, headache, dizziness, weakness and difficulty in breathing. It forms Nitrosamine in stomach which causes gastric cancer.

Safranin- O dye solution I has 4.43 ppm nitrate concentration which increased in treated water by APM to 15.505 ppm. Whereas Safranin - O dye solution II has more nitrate value than I i.e. 8.86 ppm which reduced in treated water by MnO_2 to 2.215 ppm. All water samples are in the range of standard limit (WHO). Due to higher nitrate level in APM treated water, it is more useful for irrigation.

Effect on Fluoride

Fluoride is very high reactive element so it cannot occur in elemental state in nature. It is found in all natural water resources especially in ground water. In the range of 1 – 1.5 ppm, it is effective preventive of dental carries. But above this range it may causes dental fluorosis and skeletal fluorosis. Safranin O dye solution I and II have similar value 1.5 ppm. After photocatalytic treatment by APM and MnO2, fluoride concentration increases to 2.2 and 1.8 ppm respectively. Both treated water were not in the safe zone.

Effect on Sulphate

Sulphates occur naturally in water as result of leaching from gypsum and other common minerals. In addition, sulphates increased into water system during clarification by alum. Sulphates appear to increase the corrosiveness of water towards concrete. Regarding irrigation concentration above 200 mg/l are undesirable. It also reduced under anaerobic conditions to H_2S

which cause odour and is oxidized to H_2SO_4 and corrode sewers.

APM mediated treatment increased sulphate concentration of Safranin O dye solution I 138 ppm to 148 ppm. On other side MnO2 mediated treatment reduced sulphate concentration of Safranin O dye solution II 160 ppm to 128 ppm.

Effect on DO

Dissolved oxygen analysis declared physical and biological activity in water. The minimum standard limit is 5 ppm. DO[30] and stirring affect the photocatalytical efficiency. Oxygen can serve as electron sink to trap the excited conduction band electron from reactive oxygen species. DO is proposed to cleave aromatic ring of dye molecule[31]. Safranin O dye solution I and II has DO value 11.2 and 9.7 respectively. Dye solution I on treatment by APM DO value falls to 6.7 ppm whereas treatment by MnO_2 brings rise in DO value of dye solution II to 10.5 respectively. Increase in DO value after treatment is a good sign whereas depletion in DO due to time consumed in treatment. Therefore APM photocatalysis reported better treatment than MnO_2 in case of DO value.

Effect on BOD

Biological oxygen demand is a chemical procedure for determining the amount of DO required by aerobic biological organism in a body of water to break organic material present in a given water sample at certain temperature over a specific time period. Water samples with BOD level exceeding 8 ppm is considered to be polluted.

BOD of KNP water sample at 4.4 ppm which is reduced to 1.2 and 2.7 ppm in safranin O dye solution I and II due to prevention of biological activity by dye molecule. APM mediated treatment of safranin O dye solution I reduced BOD to 0.7 ppm. Lower BOD value shows the lower consumption of oxygen and minute population load in the water. On the contrary MnO2 treatment of safranin O dye solution II increased BOD to 5.3 ppm which shows improvement in the quality of water.

Effect on COD

COD test is an artificial oxidation induced by chemical reagent which is effective on both biodegradable and bio inert material. It is main determinant used to access organic pollution in aqueous system and vital parameter in water monitoring[32]. COD data are helpful in knowing toxic condition and the presence of biologically resistant organic substances. COD of safranin O dye solution I and II are 34.84 ppm and 37.44 ppm respectively. Both APM and MnO2 mediated treatment reduced COD of safranin O dye solution I and II to 18.25 and 11.52 respectively. All the samples were in the range of COD hazard but MnO2 treated water was safer than other samples to use.

Effect on Conductivity

Conductivity as a summation parameter is measure of the level of ion concentration of a solution. It is directly proportional to its dissolved mineral matter content. Consequently it is an index of the salt load in waste water or the purity of potable water. Conductivity value 250-2000 µs/cm is permissible for irrigation. Lower the value is more suitable for this purpose. In APM mediated photocatalytic treated water conductivity value extremely increases from 778µs/cm to 1120 µs/cm. it shows dissociation of dye particle into small ions. Whereas MnO_2 mediated treatment has not so much effect on conductivity of Safranin-O dye II solution where it slightly increases from 786 µs/cm to 842 µs/cm. MnO_2 treated water is comparatively more suitable for irrigation then APM treated water.

Effect on TDS

Dissolved solids refer to any minerals, salts, metals, cations or anions dissolved in water. This includes anything present in water other than pure water (H_2O) molecules and suspended solids. In general, the TDS concentration is the sum of the cations and anions ions in the water. High TDS results in an undesirable taste, which could be salty, bitter or metallic. It could also cause gastrointestinal irrigation. TDS value of safranin O dye solution I extensively increased from 403 ppm to 600 ppm when this solution was treated by APM in optimum degradation.

On other hand MnO_2 mediated optimum degradation has no significant impact on safranin O dye solution II. All the samples are in the range (WHO) for drinking water.

Effect on Turbidity

Turbidity is a measure of the degree to which the water losses its transparency due to the presence of suspended particles. Turbidity is considered as a good measure of the quality of water. Turbidity was found to increase in polluted water (safranin O dye I) and treated water by APM to 1.9 NTU and 3.9 NTU. Whereas turbidity was found to be 0.69 NTU in safranin O dye II treated which reduce in water by MnO2 to 0.26 NTU. According to WHO standards turbidity of drinking water should not be more than 5 NTU and should be ideally below 1 NTU. High turbidity can cause aphotic zone in aquatic ecosystem. Inhibitions of photosynthesis can severely disturbed food chain.

Conclusion

Present paper supports the view that photocatalytic degradation affects many quality parameters and helps to degrade various pollutants like dyes and increase biodegradability of polluted water. Since photocatalytic treatment is driven by sunlight and semi-conductor. It makes this method eco-friendly green chemistry enabled and low cost method. Main object of this paper is to compare the efficiency of APM and MnO_2 to degrade safranin–O dye in optimum condition the basis of quality parameter changes. Photocatalytic treatment was found effective to through APM and MnO_2 reduce pH, alkalinity, hardness, Ca^{+2}, COD, along with the increase of F^- ,Conductivity, TDS. Whereas Mg^{+2}, Cl-, SO_4^{-2}, NO_3^- quality parameter increase in APM treated water and reduce in MnO_2, treated water compared to its own parent dye solution. On other hand DO and BOD decreased in APM treated water and increase in MnO_2 treated water which shows MnO_2 treated water more biodegradable than APM treated water. Increase in F^-, Cl^- concentration is a serious issue. Except F^-, Cl^-, Alkalinity. All the quality parameters were found to match with WHO standards. Although treated was safer to use but still it needs secondary treatment before it is discharged into the environment. So it can be used after secondary biological treatment for washing, irrigation and bathing.

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