



COMPARISON OF EFFICIENCY OF APM AND MnO_2 BY PHOTOCATALYTICAL DEGRADATION OF AZURE-B BASED ON QUALITY PARAMETER MODIFICATION

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ABSTRACT

This paper describes the comparison of photocatalytic activity of Ammonium Phosphomolybdate (APM) and Manganese(IV) Oxide for photocatalytic degradation of Azure-B. This study is based on modifications in quality parameters which take place during optimum photocatalytic degradation of Azure-B contaminated water using two heterocatalysts Ammonium Phosphomolybdate and Manganese(IV) Oxide separately. Various quality parameters like pH, Alkalinity, Hardness, COD, BOD, DO, Conductivity, Salinity, TDS, and Concentration of Ca^{+2} , Mg^{+2} , Cl^- , F^- , NO_3^- , SO_4^{-2} and turbidity were used for comparison.

KEYWORDS

Photo catalytic degradation; Azure-B; APM; MnO_2 ; Quality parameter

Academic Discipline and Sub-Discipline

Chemical Science

SUBJECT CLASSIFICATION

Environment Science

TYPE (METHOD AND APPROACH)

Experimental study

Council for Innovative Research

Peer Review Research Publishing System

Journal: Journal of Advances in Chemistry

Vol. 10, No. 1

editorjaconline@gmail.com

www.cirworld.org/journals

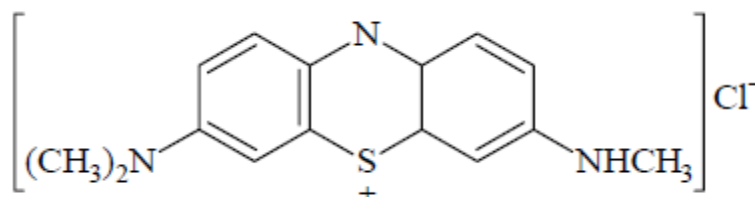


1.0 INTRODUCTION

In past decade, organic, Inorganic natural and synthetic pollutants suspected to cause harmful ecological effect and can be dangerous to terrestrial and aquatic biota including human, wild life, crops, aquatic plants etc. These hazardous pollutants include sewage containing organic load in residential wastewater and municipal waste water, agrochemical leach, industrial effluents containing dyestuff, heavy metal ions and many other toxic organic substances. Modern synthetic dyes have such a great contribution to human life that we can't imagine our routine life scenario without them. It is estimated that over 10,000 tons of dyes are per year produced globally[1]. If 10% of these is discharged into environment, it can be harmful and hazardous to ecosystem due to their stability, toxicity, fast reactivity and negative modification in quality parameters. Various chemical, physical and biological methods like adsorption by activated charcoal, flocculation, coagulation, precipitation, reverse osmosis, ultra filtration, chlorination, membrane filtration were traditionally used to remove pollutants from water bodies but these methods have some drawbacks. The removal of the non-biodegradable toxic chemicals is a crucial ecological problem. So conventional treatment methods are not impressive resulting often in an intensively colour discharge from the treatment facilities. Physicochemical processes only transfer pollutants from one phase to another phase. These methods have been diagnosed with high operational cost, toxic secondary pollutants, large amount of water and incomplete degradation[2]. However, photocatalysis plays a vital role to solve this problem to an extent. It is preferred over these methods due to low operating cost and effective treatment. A lot of studies have paid attention towards degradation of dyestuff and any other toxic compound present in waste water using various semiconductors like oxides and sulphides of metal ions. Previously we have reported that many semiconductors are commonly used for photocatalysis like TiO_2 , ZnO , Fe_2O_3 , MnO_2 , ZnS , NiO , CuO , WO_3 , CdS , Ammonium Phosphomolybdate. In order to achieve a more effective photocatalytic degradation, it is necessary to consider the surface charge property of semiconductor and nature of dye molecules. Each semiconductor has its specific degradation qualities on specific dye to decompose it.

Gullard et.al.[3] commonly used TiO_2 as photocatalyst to degrade various pollutant anionic [Aligin, Azo-Methyl Red, Congo Red, Orange G,] and Cationic [Methylene Blue]. Gary A. Epling [4] indicate that photobleaching rates differ from families of dyes and are dependent on light sources and crystalline form of TiO_2 used. Many researchers worked on different forms of TiO_2 e.g. Colloidal form [Ping Qu et.al.][5], Nanoparticle [C.C. Chen et.al.][6], Aqueous form [M.N. Rashed et.al., H. Kyung et.al.][7][8], Supported and powdered form [Gullard et. al.][3], Adsorbed TiO_2 [D. Chatterjee et. al.][9]. TiO_2 is broadly and successfully used as photocatalyst because of non-toxicity, photochemical stability and low cost [10]. Movahedi et.al.[11] used ZnO as alternative of TiO_2 and both were compared by photodegradation of Congo Red Dye. Use of semiconducting Iron(III) oxide in photocatalytic bleaching of some dyes were studied by Ameta et. Al.[12]. Sharma et.al.[13] and Bansal et.al.[14] used Ammonium Phosphomolybdate as photocatalyst to degrade Erichrome Black T and Rhodamine-6G respectively. Photocatalytic degradation of Rose Bengal using MnO_2 reported by Mittal et. al.[15]. Use of Manganese (IV) oxide particulate system as photocatalyst in photobleaching of some dyes (Methylene Blue, Toluidine Blue, Azure-B) are reported by Ameta et.al.[16]. Many researchers worked to find out optimum condition for photocatalytic degradation of dye by specific semiconductor. But only few researches are reported for comparative study of dye degradation by different semiconductor. Gandhi et.al.[17] worked on comparative study of ZnS and $\text{CoS-ZnS}(1:2)$ in photobleaching of Azure-B. Similarly Ameta et.al.[18] compared the efficiency of PbO_2 and HgO to degrade Rose Bengal.

Photocatalytic treatment of dyes can generate even more toxic intermediates and modify quality parameter in positive or negative way. Despite of all these experiment related to dye degradation, these studies remain silent over water quality parameters, eco-friendly issue 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 [19] and Fe_2O_3 [20] as photocatalyst to degrade Giemsa dye and Congo Red dye respectively and compared the quality parameter of dye contaminated water and treated water. They analysed toxicity concern by seed germination and executed bioassay of treated and untreated water. Tiwari et.al.[21] 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. In present investigation we worked on Azure-B dye degradation and quality comparison. Previously Azure-B dye degradation by APM [22], MnO_2 [16], Bismuth oxide [23], ZnS and $\text{CoS-ZnS}(1:2)$ [17] are reported.



Structure of Azure - B

IUPAC Name : 3H-Phenothiazine, 3-(dimethylamino)-7-(methylimino) hydrochloride



Molecular formula : $C_{15}H_{16}ClN_3S$

Molar mass : 305.83 g/mol

Solubility : Water

λ_{max} : 648-655 (650 nm)

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 Azure-B dye contaminated water based on quality parameter analysis and seed germination. Quality of APM mediated photocatalytically treated water and MnO_2 mediated photocatalytically treated water was compared with own parent AB dye solutions by analysing pH, Turbidity, TDS, Conductivity, Salinity, Alkalinity, Hardness, Concentration of Ca^{+2} , Mg^{+2} , F^- , Cl^- , NO_3^- , SO_4^{-2} , DO, BOD, COD and this quality factor was compared with WHO[24], IS[25] standards for drinking and other uses. Seeds of *Vignaradiata*(L.)R.Wilczek were sown germinated in treated and untreated water to assess the portable hazardous risks to agriculture sector.

2.0 EXPERIMENTAL APPROACH

2.1 Chemicals

JANUS Green-B, (Lobachemia) Ammonium Phosphomolybdate (APM) (Himedia), Manganese (IV) oxide (MnO_2), Sodium Fluoride, Zirconium Reagent, Sodium Arsenite, Sodium Azide, Sodium Iodide, Hypo ($Na_2S_2O_3$), 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.

2.2 Apparatus

Sytronics Spectrophotometer 106, Water Analyzer 371, Digital pH Meter 335, PC Based Double Beam Spectrophotometer 2202, Citizen Balance.

2.3 Method

Water samples were collected from the ponds of the KNP Bharatpur. The entire physio – chemical quality parameters of water samples were determined for comparative reference.

A 100ml stock solution (1×10^{-3} M) of AZURE- B dye was prepared in pond water samples. 17.5 ml of this stock solution was diluted to 1000 ml using water samples to prepare 1.75×10^{-5} M dye solution which is considered as polluted water. This water was further divided into two equal parts. One part of dye solution of 1.75×10^{-5} M was safely placed in borax glass beaker and exposed to sunlight for 4 hours with 0.7 gm APM at 7.5 pH in control condition for optimum PCD according to Sachdeva et al.[22]. 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, Salinity 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 [26].

Same procedure was repeated by using MNO_2 50ml of stock solution was diluted to 1000 ml to prepare 5×10^{-5} M dye solution. This solution was photocatalytically treated with 2 gm MNO_2 at 6.5pH and exposed to sunlight for optimum degradation according to Ameta et al.[16]. After 4 hours this treated water was centrifuged to sediment the MNO_2 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 all quality parameters.

3.0 RESULT AND DISCUSSION

For maximum photocatalytic degradation of Azure-B 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 Sachdeva et. al. and Ameta et. al. In presence of sunlight and semiconductor Ammonium Phosphomolybdate photo catalytic treatment affect the Quality parameters.

The results are in table 1.



Table -1 Comparative Analysis of quality parameters

Parameter	KNP water	AB dye solution I	AB dye solution I treated by APM	AB dye solution II	AB dye solution II treated by MnO ₂	WHO Standard
pH	7.81	8.40	7.05	8.03	7.96	6.5-8.5
Alkalinity (mg/L)	72	84	56	100	48	200
Hardness (mg/L)	370	280	260	260	200	300
Calcium (mg/L)	170	120	140	120	130	75-200
Magnesium(mg/L)	200	160	120	140	70	30-100
Chloride (mg/L)	310	290	220	370	250	250
Fluoride (mg/L)	1.2	0.9	1.2	0.75	1.25	1-1.5
Sulphate (mg/L)	45.6	31.8	61.26	49.7	100	200
Nitrate (mg/L)	2.215	8.86	6.645	13.29	4.43	45
DO (ppm)	6.4	5.8	4.5	6.1	4.7	5
BOD(ppm)	4.4	2.4	2.9	2.7	2.3	6
COD (ppm)	12.8	24.83	5.12	36.86	20.48	10
Conductivity(μS/cm)	904	858	1290	821	817	1000
TDS(ppm)	445	477	760	462	471	500-2000
Turbidity(NTU)	1.4	2.6	1.3	3.1	0.84	5

3.1 Effect on pH

It is measure of Hydrogen ion concentration or more precisely the Hydrogen ion activity. pH of Azure-B dye solution I (1.75×10^{-5} M) and Azure-B dye solution II (5×10^{-5} M) are 8.40 and 8.03 respectively. After photo catalytic treatment both semiconductor (APM and MnO₂) reduced pH value of dye solution I and II to 7.03 and 7.96 respectively. In between them APM has reduce pH value more efficiently. However MnO₂ has little effect on pH. All water samples were found to be within the IS permissible limit (6.5-8.5). Photocatalyzed intermediates seem to have slightly different functional group compared to parent dye. It seems to appear different groups in APM treated water.

3.2 Effect on Alkalinity

Alkalinity is not a pollutant is the quantitative capacity of aqueous system to react with hydrogen ions. Ability to neutralize acid is generally due to carbonate, bicarbonate and hydroxide ions; sometimes it includes borates, phosphate, silicate or other bases. Alkalinity of Azure-B contaminated water (1.75×10^{-5} M) reported at 84 mg/l, which reduced to 56 mg/l in treated water by APM. Whereas other Azure-B dye solution (5×10^{-5}) has 100 mg/l alkalinity and MnO₂ mediates photocatalytic treatment reduced it to 48 mg/l. Both APM and MnO₂ affected alkalinity in similar manner, but MnO₂ affected it more than APM.

3.3 Effect on Hardness

Hardness in water is due to the natural accumulation of salts from contact with soil and geological formation or it may enter from direct pollution by industrial effluents. Ca and Mg are the principal cations causing hardness. Iron, Aluminium, Manganese, Strontium and Zinc also cause hardness but in a negligible amount. So, total hardness indicates the concentration of Ca and Mg ions only.

Hardness was reduced from 280 mg/l to 260 mg/l when A-B Dye solution I (1.75×10^{-5} M) photochemically treated by APM. On other side MnO₂ reduced it from 260 mg/l (Azure-B Dye solution II (5×10^{-5})) to 200 mg/l (treated water). Comparative results showed that MnO₂ was more effective than APM. Total hardness higher than Alkalinity showed that neutral salt of Ca⁺², Mg⁺² ion are sulphate not carbonate.

3.4 Effect on Calcium

Calcium salts and Calcium ions are the most commonly occurring species in nature [27]. Calcium salts can be readily precipitated from water but high levels of Calcium hardness tend to promote scale formation in the water system.

Ca was noted at 120 mg/l for both Azure-B dye solution I and II. After photocatalytic treatment calcium ion concentration for both solutions increased to 140 mg/l and 130 mg/l by APM and MnO₂ respectively. All the water samples are in the maximum permissible limit (IS), 200 mg/l in drinking water.



3.5 Effect on Magnesium

Magnesium enters in the drinking water system from geological sources[28]. It is relatively non-toxic to human, however high concentration causes unpleasant taste to water.

In KNP water Mg^{+2} ion concentration was reported at 200 mg/l which reduced to 160 mg/l and 140 mg/l in Azure-B dye solution I (1.75×10^{-5} M) and Azure-B dye solution II (5×10^{-5} M) respectively. It seems that dye particle reacted with Mg^{+2} ions, So after photocatalytic treatment APM further reduced dye I solution Mg^{+2} concentration to 120 mg/l and MnO_2 reduced AB dye II solution Mg^{+2} concentration to large extent means 70 mg/l.

3.6 Effect on Chloride

Chloride is the common anion found in water and sewage. The toxicity of chloride salts depends on the cations present, but that of chloride itself is uncertain. Although excessive intake of drinking water containing NaCl above 2.5 g/l has been reported to produce hypertension.

Estimation of chloride ion in Azure-B dye solution I (1.75×10^{-5} M) is 290 mg/l which is reduced in treated water to 200 mg/l by APM mediated photocatalytic treatment. On other side chloride concentration in Azure-B dye solution II (5×10^{-5} M) concentration higher than previous one reported at 370 mg/l. After photocatalysis by MnO_2 it reduced to 250 mg/l. Although treated water by APM had lower value of Cl^- than treated water by MnO_2 , but MnO_2 created larger difference on treatment than APM.

3.7 Effect on Nitrate

Nitrates which occur in trace quantities in surface waters, wastes from chemical and fertilizer manufacturing plants are also important contributors of nitrate to water sources. The maximum permissible limit is 45 ppm. High Nitrate levels in water can cause blue baby syndrome, headache, dizziness, weakness and difficulty in breathing. In APM mediated treated water, nitrate decreased from 8.86 ppm to 6.645 ppm. MnO_2 mediated treatment also decreased nitrate value from 13.29 mg/l to 4.48 mg/l. Nitrate is an indicator of nutrient enrichment when it is in limit.

3.8 Effect on DO

Dissolved oxygen analysis measures the amount of gaseous oxygen dissolved in an aqueous solution. It is significant parameter which indicates the quality of water. Oxygen gets into water through diffusion from the surrounding air, by aeration and as a waste product of photosynthesis [29]. DO is an index of physical and biological process occurring in water.

Azure-B dye contaminated water (1.75×10^{-5} M) has 5.8 ppm DO value which was reduced to 4.5 ppm when it was treated by APM. Azure-B dye solution II has DO value 6.1 ppm which also reduced to 4.7 ppm when this dye solution treated by MnO_2 .

3.9 Effect on BOD

Despite of all the limitations, BOD still remains the single best test that gives a measure of the amount of biologically oxidizable organic matter presents [30]. It is a factor in the choice of treatment method and is used to determine the size of certain units, particularly trickling filters and activated sludge units. In present study BOD value of reference water was recorded 4.4 ppm which reduced to 2.4 ppm and 2.7 ppm in AB dye solution I and II respectively. After photocatalytic treatment of dye solution I and II by APM and MnO_2 respectively, BOD value increased in APM treatment case but to small extent ie, 2.9 ppm but reduced in MnO_2 treatment to 2.3 ppm. Lower value of BOD shows the lower consumption of oxygen and minute population load in the water.

3.10 Effect on Conductivity

It is useful to evaluate the purity of water. It is highly dependent on the dissolved solids in the water which affect the taste of water and suitability for various uses. However, it is only a quantitative measurement. In APM mediated photocatalytic treated water conductivity value increases significantly from 858 $\mu S/cm$ to 1290 $\mu S/cm$. It shows mineralization of dye contaminated water. On other hand, MnO_2 mediated treatment has no significant effect on dye contaminated water where conductivity value changes from 821 $\mu S/cm$ to 817 $\mu S/cm$. In APM treated water slightly decrease in pH and rise in conductivity confirms the mineralization of dye contaminated water samples into CO_2 and inorganic ions [31].

3.11 Effect on TDS

Total dissolved solids refer to any minerals, salts, metals, cations, or anions dissolved in water. The most important aspect of TDS with respect to drinking water quality is its effect on taste. The palatability of drinking water with a TDS level less than 600 mg/l is considered to be good (WHO standard). High TDS results in undesirable taste which could be salty, metallic or bitter.

445 ppm TDS found in reference KNP water which further enhanced in polluted dye solution I to 477 ppm. After APM photocatalytic treatment, it reduced to 460 ppm. Whereas TDS value of dye solution II noted at 462 ppm which increased to a small extent, 471 ppm. So where APM reduced TDS, MnO_2 increased it but both are effective with small limit.



3.12 Effect on Turbidity

Turbidity is a measure of the cloudiness of water. In natural water it arises due to presence of suspended matter. High turbidity can cause aphotic zone in aquatic system. Inhibition of photosynthesis can severely disturb food chain. Dye particle is main contributor for TDS in polluted water. Sometimes photocatalysed dye intermediate also can cause turbidity in reactor leading to reduce photocatalytic efficiency [32].

Turbidity of Azure-B dye solution I is 2.6 NTU (1.75×10^{-5} M) which increase with the rise of dye concentration which is found 3.1 NTU in 5×10^{-5} M. After treatment turbidity of AB-I and AB-II reduced to 1.3 and 0.83 NTU. When AB-I treated by APM and AB-II treated by MnO_2

3.13 Effect on Fluoride

Fluoride is a natural element found in rocks and soil everywhere in fresh water and in ocean water. Particularly, the soil at the foot of mountains is likely to be high in fluoride concentration from the weathering and leaching bed rock with fluoride content [33]. Fluoride in amount of 1 to 1.5 mg/l, it is effective preventive of dental caries. Above this amount, fluoride may cause dental and skeletal fluorosis.

AB dye solution I and II noted at 0.94 and 0.75 mg/l that is very low in concentration. Both semiconductor enhanced fluoride value of own sample. APM increased AB dye solution I value to 1.2 mg/l while MnO_2 also increased AB dye solution II value to approximately same value 1.25 mg/l. All the water sample are in the range of IS permissible limit. But effect of MnO_2 on increasing fluoride value is greater than APM.

3.14 Effect on Sulphate

Sulphate ion form salts with various element including Potassium, Magnesium, Sodium (more soluble) and Calcium, Barium (less soluble). Sulphates have a detoxifying effect on liver and stimulate the function of the gall bladder and the digestive function as well [34]. After photocatalytic treatment in both cases sulphate value increased to 31.8 mg/l and 49.78 mg/l sulphate value found in AB dye solution I and II respectively. After treatment by APM, value of dye solution I increased to 61.26 mg/l and by MnO_2 , value of dye solution II increased to 100 mg/l.

3.15 Effect on COD

Chemical oxygen demand is a vital test for assessing level of organic pollution in aqueous system and it is one of the most important parameter in water monitoring [35]. COD value of AB dye solution I reported at 24.832 ppm which is extremely reduced to 5.12 ppm in APM mediated photocatalytic treated water. However AB dye solution II (5×10^{-5} M) have COD value at 36.86 ppm which also reduced in MnO_2 mediated photocatalytic treated water to 20.48 ppm. Except APM treated water all water samples are found in hazardous category of COD values.

3.16 Effect on Seed germination

In present study seed germination and quality parameter takes place simultaneously. In Reference water 60% of seed germinated completely whereas 35% seeds germinated partially and left 5% seeds did not germinate. Dye particles prohibited seed germination capability of seeds. AB dye solution I (1.75×10^{-5} M) promote only 50% seeds are completely and 40% partially germinated. When dye concentration increased in AB dye solution II (5×10^{-5} M) seed germination reduced where only 30% seeds germinated completely, 30% germinated partially and 40% (large quantity) seeds did not germinate.

When AB dye solution I was treated by APM, seed germination percentage appreciably increased. All seeds germinated in this treated water. From these 75% seeds germinated completely and 25% germinated partially. On the other hand, MnO_2 also enhanced seed germination capability of AB dye solution II where 50% seeds germinated completely, 40% germinated partially, lesser efficient than APM. Above results are based on seed germination assay which was executed two days only. Here it is notable that AB may increase acclimatization time for seed germination and each seed may respond in fluctuating manner due to genetic variability within species.

Table-2 Seed germination percentage in different solution

Solution	Germination Complete %	Germination Partially %	Germination Inhibited %
Reference water	60	35	5
Azure-B dye solution I(1.75×10^{-5} M)	50	40	10
Azure-B Dye solution II(5×10^{-5} M)	30	30	40
AB I + APM	75	25	0
AB II + MnO_2	50	40	10





Figure1. (A) Seeds germination of Vignaradiata (L.) R.Wilczek in KNP water, (B) Seeds germination of Vignaradiata (L.)R.Wilczek in Azure-B dye solution I(1.75×10^{-5} M) (C)Seedsgermination of Vignaradiata (L.)R.Wilczek in Azure-B dye solution II(5×10^{-5} M), (D) Seeds germination of Vignaradiata (L.)R.Wilczek in photocatalyzed AB I dye by APM, (E) Seeds germination of Vignaradiata (L.) R.WilczekinphotocatalyzedAB II dye by MnO_2 .

4.0 CONCLUSIONS

Present work supports the view that photocatalytic treatment affects many quality parameters and helps to degrade pollutants like dyes and increases biodegradability of polluted water. Since photocatalytic treatment is driven by sunlight and semiconductor as catalyst (in small amount), it is an eco-friendly and low cost method.

Main object of present work is to compare APM and MnO_2 effect on AB dye. Investigation claims that sink drain of AB dye solution is hazardous to environment. APM and MnO_2 mediate treatment supports reduction in pH, Alkalinity, Hardness, Mg^{+2} , Cl^- , Turbidity, COD, DO, nitrate along with rise in Ca^{+2} , sulphate, fluoride, Conductivity, except BOD and TDS which reduce in APM and increase in MnO_2 . Rise in sulphate and Conductivity occur due to degradation of dye and ions formation. Almost all quality parameters found match with WHO standard for drinking water after APM and MnO_2 treatment. But rise in BOD and Conductivity value along with falls in pH, TDS and COD in treated water by APM made it more significant, non-hazardous water which proved by seed germination assay where almost all seeds are germinated. Although APM and MnO_2 mediated treatment of AB dye was found satisfactory but still it needs secondary treatment before it disposed off into environment. So it can be used after secondary biological treatment for washing, cooling, irrigation and bathing.

Abbreviation:

AB – AZURE-B dye

APM – Ammonium phosphomolybdate

WHO – World Health Organization

Acknowledgement: Authors are thankful for Dr. Sangeeta Chaturvedi, Lecturer in Zoology, M.S.J. College, Bharatpur for kind co-operation.

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