Asian Journal of Water, Environment and Pollution, Vol. 16, No. 3 (2019), pp. 25-33.
DOI 10.3233/AJW190030

Nano-scale Iron Oxide as Heterogeneous Fenton Catalyst for Organic Pollution Degradation and Heavy Metal Remediation in Water Sample of Byramangala Lake, Karnataka

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Received December 8, 2018; revised and accepted May 14, 2019

Abstract: A nano-scale iron oxide (Fe_2O_3/Fe_3O_4) heterogeneous Fenton catalyst was synthesized for aqueous heavy metals remediation and was utilized to degrade organochlorine pesticides (OCPs), namely, alpha-, and delta-hexachlorocyclohexane (α-HCH and δ-HCH) as well as heptachlor epoxide (HE), in Byramangala lake water sample. Nano-scale Fe_2O_3/Fe_3O_4 was synthesized via Solution Combustion Synthesis (SCS), with glycine as fuel and iron oxide separated from raw laterite soil sample (RL) as oxidizer for the reaction. Based on detailed characterization, it was found that iron oxide constituted 34.31% of the RL sample. Later, detection of hematite (Fe_2O_3), hypersthenes ($MgSiO_4$) and lepidocrocite (FeOOH) in the separated fraction confirmed extraction of Fe_2O_3 fraction. Next, collection of Byramangala lake water samples and analyses of physico-chemical parameters revealed nil dissolved oxygen (DO) and high chemical oxygen demand (COD) levels. Oxidation of detected pesticides and susceptible heavy metals using the nano-scale Fe_2O_3/Fe_3O_4 was investigated under various experimental conditions: pH (3 and ~7), dosages of H_2O_2 (1-9 mM L^{-1}) and nano catalyst (0.05-0.4 g L^{-1}), as well as contact time (5-120 min) in batch experiments. Optimum values were found to be contact time of 50 mins at near-neutral pH at 5 mM L^{-1} H_2O_2 and 0.25g L^{-1} Fe_2O_3/Fe_3O_4 dosages, respectively. Under optimized conditions, 100% removal of both α-HCH and δ-HCH, and 99% removal of HE were observed. Furthermore, appreciable reduction in arsenic (As) and manganese (Mn) concentrations (95% and 76.6%) was also observed. Post-treatment, Fe_2O_3/Fe_3O_4 could be efficiently separated by an external magnetic field because of its ferromagnetic behaviour.

Key words: Laterite, hexachlorocyclohexane (HCH), arsenic, heterogeneous fenton catalyst, nano-scale iron oxide.

Introduction

Rapid urbanization in Indian cities and increase in industrial activities in and around them has given rise to critical issues of water pollution. Inadequate infrastructure to treat various municipal wastes and improper treatment of industrial waste had led to deterioration of the surrounding environment. An example of such urban centres is Bengaluru—a main commercial centre for some major industrial establishments. Looking back, Bengaluru has historically witnessed three phases of globalization: the colonial phase, the garment phase and the information technology phase (Pani, 2009). Currently, a start-up culture is being fostered in the city. Throughout all these phases, a shift in the nature of human interaction with

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the surroundings has led to the city obtaining notoriety with respect to environmental issues. With an increase in the percentage of urban area from 1.87% in 1973 to 31% in 2014 (Ramachandra and Majumdar, 2009; Singh, Unknown), the cityscape and land use patterns show dramatic changes. In totality, all of these activities have had a significant impact on the nature in the city – once characterized by a functional cascading lake system and ample greenery.

Water pollutants of concern such as persistent organic pollutant (POPs) and heavy metals have been detected in various environmental matrices in the city. For example, in a study conducted by Chakraborty et al. (2015), a trend was observed in OCPs residue in surface soil along the urban-suburban and rural transect of Bengaluru—rural > suburban > urban. This trend was attributed to the region specific usage of OCPs. It was also noted that rural Bengaluru soil had the highest concentration of hexachlorocyclohexane (HCHs) and hexachloro benzene (HCB), and second highest concentration of endosulfan.

For this study, Byramangala Lake was selected. Located on the outskirts of Bengaluru, significant accumulation of nutrients and surfactants from domestic sewage, heavy metals from industrial effluents and agro-chemicals from farming activities has been observed in previous lake water samples. Byramangala Lake has been widely reported in several print and electronic media as well (http://www.thehindu.com/todays-paper/tp-national/tp-karnataka/team-assesses-impact-of-pollution-around-byramangala-reservoir/article19388903.ece).

Advanced oxidation processes (AOPs) utilizing heterogeneous catalysts in Fenton reaction have garnered interest due to their potential to completely mineralize organic pollutants into harmless, final products. In addition, recent studies have shown their efficiency in the treatment of heavy metal contamination, such as nickel (Ni) (Fu et al., 2012), chromium (Cr) (Shahriari et al., 2014), arsenic (As), chromium (Cr) (Sharma and Kennedy, 2018). Simultaneous removal of organic pollutants and heavy metals has also emerged as a topic of interest worldwide. The process is preferred owing to its low energy consumption and cost, high treatment efficiency, minimized toxic sludge (as compared to classic homogeneous Fenton reactions), regeneration of solid catalyst and possible metal recovery.

In the present study, the treatability of Byramangala lake water sample by heterogeneous Fenton reaction was investigated. Operating parameters like pH, H_2O_2 concentration, dosage of nano catalyst and contact time were optimized. On optimization, pesticide and heavy metal analyses were carried out in order to assess the effect of the advanced oxidation process. In this complex system, effect of the treatment technology on individual sub-systems and the interaction between each other was studied.

Study Site

Byramangala tank is approximately 40 km from Bengaluru (GPS location: 12°45′N and 77°25′E), located in Byramangala, Bidadi, Ramnagara district, Bengaluru Rural in Karnataka. Constructed across Vrishabhavathi river in 1943, the tank covers a water spread area of about 430.25 ha at FRL and is fed by the Vrishabhavathi River (catchment area 561 km^2). Vrishabhavathi catchment is a part of Arkavathi sub-basin, itself a part of the larger Cauvery basin. It carries domestic sewage (partially treated), industrial effluents and storm water from urban, peri-urban and rural area. The catchment area includes urban areas in South Bengaluru as well as villages in rural areas outside the Bruhat Bengaluru Maharangara Palike's (BBMP) administration. Industrial areas located in Peenya and Bidadi are situated in the tank's catchment area, whose wastes are discharged into the river. In addition, the lake is a major source of water to the local population for various purposes like agriculture, fisheries, etc. Farmers downstream use the water for irrigation through a canal system. Crops such as ragi, paddy, and sugarcane were grown prior to deterioration of the water quality. After substantial changes in the water quality, commercial crops like baby corn, mulberry, tomato, cauliflower, as well as for fodder for milk-producing cattle. As a result of this, agricultural run-off also makes its way into the reservoir.

Experimental

Materials

All reagents used in this study were of analytical grade and were used directly without any further purification. Raw laterite soil was collected from National Institute of Technology, Karnataka (NITK) campus, Surathkal, Karnataka, India (GPS location: 13°01′N and 74°79′E). It was crushed, dried and sieved to get clean laterite particles passing through 75 μm sieve. Water samples were collected from Byramangala lake for analysis and experimentation. All glassware was purchased from Borosil. Double distilled water was used throughout the study.

Oxide Composition of Laterite Soil Sample and Separation of Iron Fraction

Analysis of relative proportions of oxides present in the collected laterite soil sample was carried out in order to characterize the sample, as well as determine the possible quantity of iron that could be separated. Silica (SiO_2), aluminum oxide (Al_2O_3) and iron oxide (Fe_2O_3) were determined according to IS: 2720 (Part XXV) – 1982 (Part XXV – Determination of Silica Sesquioxide Ratio, 1982). Titanium dioxide (TiO_2) by hydrogen peroxide (colorimetric) method, as prescribed in IS: 1493-1959 ((IS), 1959). Calcium and magnesium oxides (CaO and MgO) were estimated by complexometric titration method. A flame photometer (Model 112-Systronics) was used to measure sodium and potassium oxides (Na_2O and K_2O). Loss on ignition (LoI) was measured gravimetrically, the results of which are tabulated in Table 1. Furthermore, iron fraction was extracted as per IS 2728: Part (XXV).

Table 1: Oxide composition of laterite soil sample

Sl. No.	*Constituent*	*Percentage (%)*
1	Silica as SiO_2	19.58
2	Alumina as Al_2O_3	25.39
3	Ferric oxide as Fe_2O_3	34.31
4	Titanium oxide as TiO_2	1.80
5	Calcium oxide as CaO	0.89
6	Magnesium oxide as MgO	0.48
7	Sodium oxide as Na_2O	0.57
8	Potassium oxide as K_2O	0.48
9	Loss on Ignition (LoI)	16.35

Characterization

The raw laterite and extracted iron fraction were characterized for mineralogical composition and morphological properties with XRD and SEM techniques. Powder X-ray diffraction (XRD) patterns of the samples were recorded on a PANalytical X'Pert Pro Powder diffractometer operated at 40 kV and 30 mA using Ni-filtered Cu Kα radiation (λ = 1.5418 Å). Physical properties, namely, size and shape of raw laterite (RL) particles as well as the extracted iron fraction was captured using SEM with accelerating voltages of 5 and 10 kV.

Synthesis of Nano-scale Catalyst

Nano-scale iron oxide was synthesized by Solution Combustion Synthesis (SCS), with glycine as fuel. This complex system was investigated under stoichiometric and fuel rich conditions ($\varphi \geq 1$) so as to synthesize powders with high specific surface area. Iron fraction extracted from the laterite soil sample was dissolved in conc. nitric acid (HNO_3) and mixed thoroughly to generate ferric nitrate ($Fe(NO_3)_3$) (Eq (1)). Then, required amounts of glycine for stoichiometric and fuel-rich conditions were added (Eq. (2)).

$$Fe_2O_3 + 6HNO_3 \rightarrow 2Fe(NO_3)_3 + 3H_2O \qquad (1)$$

$$2Fe(NO_3)_3.9H_2O + \left(\frac{10}{3}\right)\phi\, CH_5NO_2 + \left(\frac{3}{10}\right)(\phi-1)O_2 \rightarrow Fe_2O_3 + \left(\frac{25}{3}\phi+18\right)H_2O \left(\frac{20}{3}\phi\right)CO_2 + \left(\frac{5}{3}\phi+3\right)N_2 \qquad (2)$$

The container was then placed on a hot plate in order to heat the mixture uniformly till the boiling point at a rate of ~5°C min^{-1}. This was followed by a relatively long (~5 min) constant temperature during which a pseudo-gelatinous mass was formed. Then, it was transferred to a muffle furnace and the temperature was gradually raised to 450°C. This stage involved combustion reaction with release of gases, either started by sudden uniform temperature rise (volume combustion synthesis) or reaction initiated by a specific hot spot followed by steady wave propagation through the mixture (self-propagating high-temperature synthesis). After cooling, the synthesized products were fine solid powders. The obtained powders were characterized using XRD and SEM with specifications as mentioned earlier.

Collection of Byramangala Lake Water Sample

The water samples were collected in the middle of the lake, at a point before the water reached an overflow weir for two months – March and April, 2018. The sampling was done in the morning hours at the centre so as to get a general water quality of the lake. Grab samples were collected at different depths in one-litre pre-washed polythene cans, and various physico-chemical parameters were analyzed using standard methods (AWWA, APHA). Since dissolved oxygen (DO) is an important water quality parameter it was fixed on site in glass (BOD) bottles. Analysis of pH, Electrical Conductivity, Dissolved Oxygen, Chemical Oxygen Demand, Biochemical Oxygen Demand, nitrate (NO_3-N), Total Hardness, calcium as $CaCO_3$, magnesium as $CaCO_3$, sodium as Na, potassium as K, Total Alkalinity, Total Suspended Solids (TSS), Total Dissolved Solids (TDS) and Total Phosphate as P was carried out. The pesticides concentration in the collected water sample was analyzed using gas chromatography-mass spectrometry (GC-MS, Agilent). Heavy metals were examined using inductively coupled plasma mass spectrometry (ICP-MS, Agilant 7900). The composition of the sample is shown in Table 2.

Batch Studies

To evaluate the removal of the detected OCPs and remediation of heavy metals found in the water sample, batch experiments were carried out in 500 mL conical flasks which could be sealed. To ensure uniform mixing, a rotary mixer was employed (150 rpm). All experiments were conducted at room temperatures (29±3°C). pH adjustment was carried out by the addition of appropriate amount of 0.1M HCl. Amount of H_2O_2 were maintained above stoichiometric concentrations so as to avoid the possible formation of highly toxic by-products. Various operating conditions were optimized, namely, pH (3 and ~7), H_2O_2 (1-9mM L^{-1}), nano-scale Fe_2O_3/Fe_3O_4 (0.1-0.4 g L^{-1}) and contact time (5-120 min). At corresponding intervals, 10 mL of the solution were collected and its COD was analyzed. Correction for hydrogen peroxide interference on COD test was performed by the correlation equation reported by Kang et al. (1999). All experiments were duplicate. Optimization of these conditions was carried out and then a final run under such conditions was conducted. The levels of pesticides and heavy metals post-treatment in optimized conditions were measured using GC-MS and ICP-MS.

Results and Discussion

Characteristics of Laterite Soil Sample

Chemical analyses of the collected raw laterite revealed it primarily constituted iron and aluminum oxides, with significant silica oxide content. More mobile elements such as Na, Ca, K, etc were detected in trace amounts.

The oxide composition of the laterite soil sample is mentioned in detail in Table 1. XRD analysis of the sample showed that it was composed of gibbsite [$Al(OH_3)$], anatase (TiO_2), kaolinite [$Al_4(OH)_8(Si_4O_{10})$] and hematite (Fe_2O_3).

Characteristics of Separated Iron Fraction

Oxide and hydroxide phases of iron in the separated sample included hematite (Fe_2O_3) and lepidocrocite

Table 2: Physico-chemical parameters of Byramangala lake water sample

Sl. No.	*Parameter*	*Unit*	*Measured value*		
			At surface	*0.5 m below surface*	*1 m below surface*
1	Colour	-	Colourless	Colourless	Colourless
2	Odour	-	Unpleasant	Unpleasant	Unpleasant
3	pH	-	7.07	6.92	6.89
4	Electrical Conductivity	µS/cm	1587	1591	1593
5	Dissolved Oxygen	mg L^{-1}	Nil	Nil	Nil
6	Chemical Oxygen Demand	mg L^{-1}	202	208	198
7	Biochemical Oxygen Demand	mg L^{-1}	27	25	23
8	Nitrate (NO_3-N)	mg L^{-1}	19	20	19
9	Total Hardness	mg L^{-1}	312	303	307
10	Calcium as $CaCO_3$	mg L^{-1}	175	175	175
11	Magnesium as $CaCO_3$	mg L^{-1}	138	128	132
12	Sodium as Na	mg L^{-1}	161	169	153
13	Potassium as K	mg L^{-1}	27	26	27
14	Total Alkalinity	mg L^{-1}	460	462	467
15	Total Suspended Solids (TSS)	mg L^{-1}	16	18	24
16	Total Dissolved Solids (TDS)	mg L^{-1}	998	1002	988
17	Total Phosphate as P	mg L^{-1}	1.2	0.88	0.69

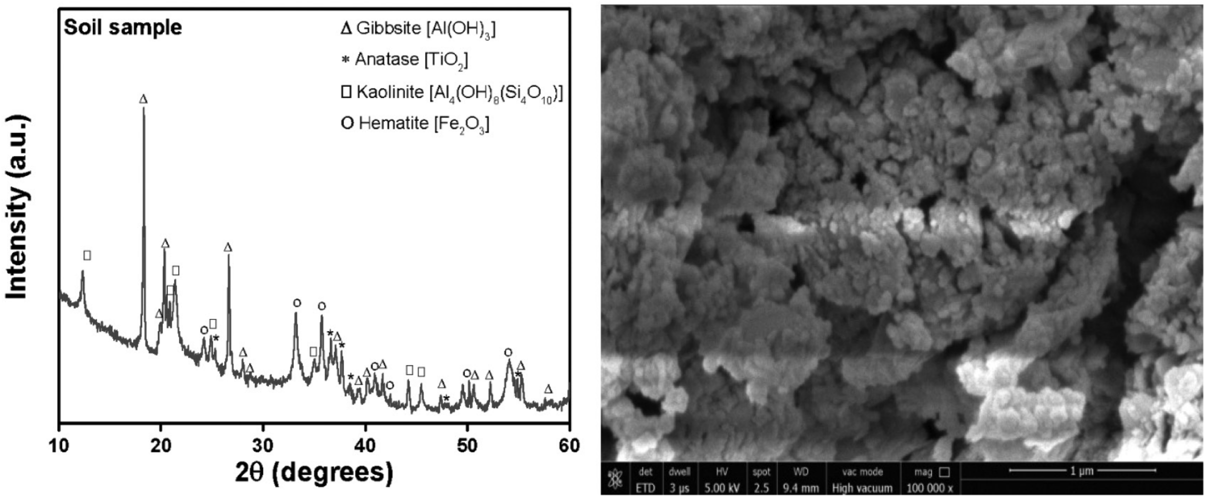

Figure 1: Characterization of collected laterite soil sample: (a) XRD pattern, and (b) SEM micrograph.

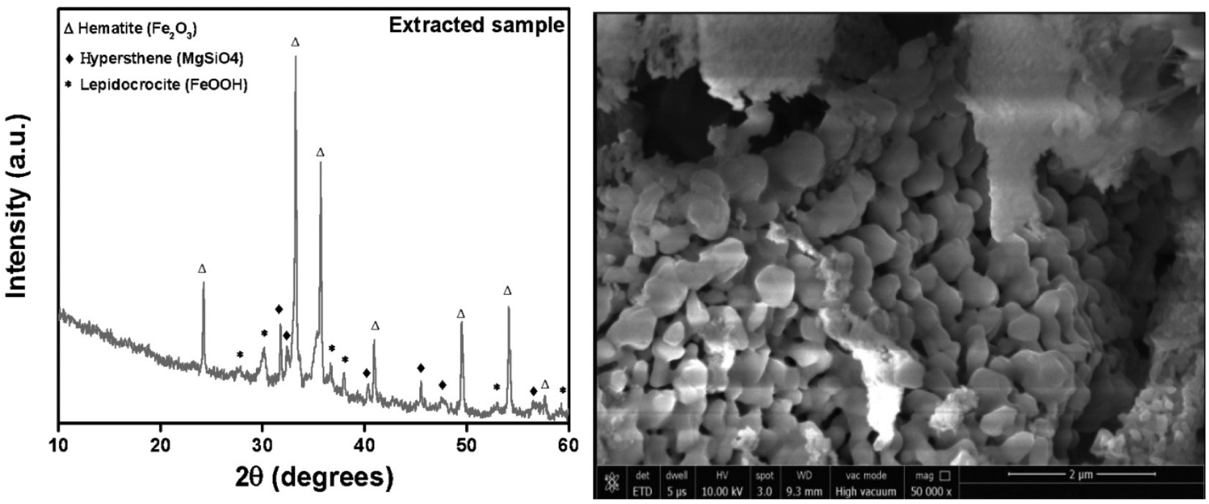

Figure 2: Characterization of separated iron fraction: (a) XRD pattern and (b) SEM micrograph.

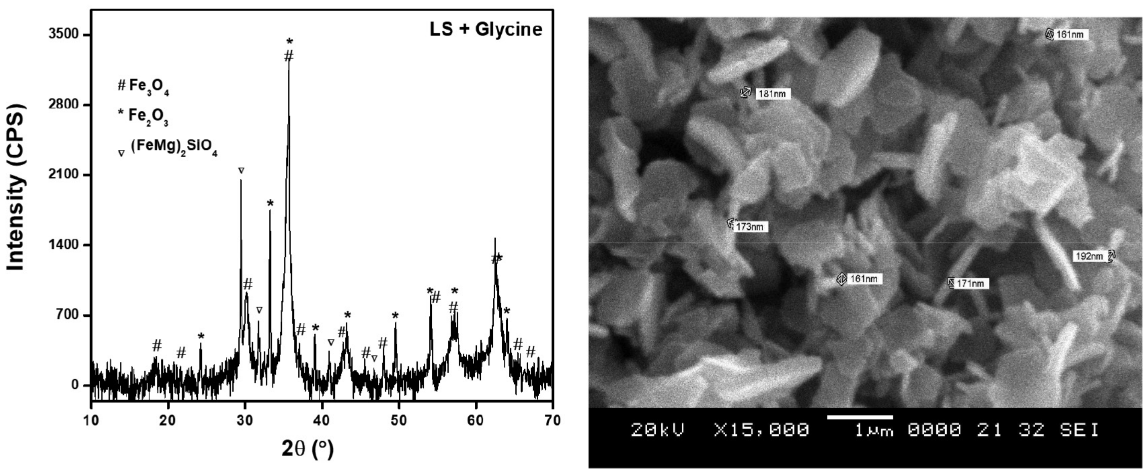

Figure 3: Characterization of nano-scale iron oxide: (a) XRD pattern and (b) SEM micrograph.

(γ-FeOOH). Peaks of hypersthene ($MgSiO_4$) were also detected. Almost spherical shape of the particles confirms the presence of hematite.

Characteristics of Synthesized Nano Catalyst

XRD results of the synthesized nano-scale iron oxide showed the presence of iron(III)oxide (Fe_2O_3) and iron (II,III) oxide (Fe_3O_4), with traces of iron magnesium silicate $(FeMg)_2SiO_4$. $(FeMg)_2SiO_4$ contains magnesium silicate (Mg_2SiO_4) and iron silicate (Fe_2SiO_4). SEM micrograph (Figure 3) showed the presence of flakes of thickness ranging from 161-192 nm.

Water Sample Analysis

Table 2 illustrates various physico-chemical properties of the raw water sample collected at Byramangala lake. With no dissolved oxygen detected, it was categorized as Class E – best designated to be used for irrigation, industrial cooling and controlled waste disposal. Subsequent analysis of pesticides and heavy metals brought forth the deteriorated condition of the lake water.

Table 3: (a) Heavy metal (in ppm) and (b) pesticides (in ppb) analyses of Byramangala lake water sample

Sl. No.	*Heavy metal*	*Measured value*		
		At surface	*0.5 m below surface*	*1 m below surface*
1	Cadmium	0.0001	0.0001	0.0001
2	Chromium	0.0078	0.0078	0.0082
3	Copper	0.0017	<0.001	<0.001
4	Iron	0.2600	0.2329	0.2593
5	Manganese	0.5207	0.4480	0.4518
6	Nickel	0.0117	0.0116	0.0114
7	Zinc	0.0047	0.0063	0.0011
8	Lead	0.0011	0.0036	0.0006
9	Arsenic	0.0005	0.0005	0.0005

Sl. No.	*Pesticide*	*Value*
1	Alpha – HCH	229.5
2	Delta – HCH	5.25
3	Heptachlor Epoxide	62.1

Pesticides analysis: GC analysis of the collected water sampled revealed the presence of three OCPs—α-, and δ-hexachlorocyclohexane ($C_6H_6Cl_6$) as well as Heptachlor epoxide ($C_{10}H_5Cl_7O$) (Table 3(b)). All three of them are listed as organic priority substances in the water policy of the 2013/39/EU Directive (Ribeiro et al., 2015).

Degradation of these OCPs is influenced by their physico-chemical properties – higher the water solubility and lower the octanol-water coefficient, faster is the degradation (Shoiful et al., 2016). Water solubility of α-, and δ-HCH as well as heptachlor is 100 mg L^{-1}, and 0.18 mg L^{-1}, with octanol-water coefficients (LogKow) of 3.8, 4.14 and 5.44, respectively. Therefore, the degradation rates of these OCPs are expected to be as follows (in descending order): α-HCH > δ-HCH > Heptachlor epoxide.

Optimization of Heterogeneous Fenton Process

Effect of pH: pH level of the reaction solution significantly influenced the COD removal efficiency. It was noted that pH 3 did not favour high COD removal. Furthermore, measurement of iron in water sample treated at pH 3 indicated a two-fold increase in iron content. Thus, enhanced leaching of the nano-catalyst in acidic conditions was considered as a possible explanation to the reduced efficiency. Increasing the pH to near neutral condition (~7) facilitated higher removal. Optimal removal was found to be 75% under near-neutral pH.

Effect of H_2O_2 concentration: COD removal efficiency was determined at a wide range of H_2O_2 concentration (1-9 mmol L^{-1}). It is well known that the degradation of organic contaminants is dependent on the concentration of H_2O_2 and radical formation. Removal efficiency was enhanced when the H_2O_2 concentration increased from 1 to 5 mmol L^{-1}. However, a subsequent decrease in efficiency was noted at higher concentrations, which suggests scavenging effects by H_2O_2 radicals (Figure 4.). The optimal concentration was found to be around 5 mmol L^{-1}.

Effect of nano-scale catalyst dosage: COD removal efficiency was determined for various nano-scale iron oxide loadings (0.1-0.4 g L^{-1}). As shown in Figure 4 (a), increase in the nano-catalyst dosage led to increased efficiency, upto 0.25 g L^{-1}. After this point, there was little variation in the removal efficiency noted. It was posited that after 0.25 g L^{-1}, though more active sites were added to the solution, a majority of the reaction had taken place. Considering other operation parameters (especially H_2O_2 concentration, which is considered as the limiting reagent), the driving force to facilitate oxidation reaction on the nano-catalyst surface reduces, thereby showing no appreciable increase in removal efficiency. Hence, the optimum dosage was determined to be 0.25 g L^{-1}.

Effect of contact time: Figure 4(b) shows the effect of contact time on COD removal efficiency. It is evident that the initial rate of percent removal increases rapidly up to 30 min, after which there is a gradual increase and finally equilibrium is reached. The optimum contact time was calculated to be 50 min.

Heterogeneous Fenton Reaction – Effect on Heavy Metal Ion Concentration

Initially, manganese (Mn), with a concentration of 0.5207 ppm, exceeded the prescribed limits (BIS/WHO). Treatment under optimized conditions brought down its concentration to 0.1217 ppm, thereby reducing it by 76.6%. Arsenic (As) as well was reduced by 50%. Figure 5 illustrates the variation in concentration of different heavy metals in raw sample (C_o) and post-treatment (C_e). Additionally, Figure 3(c) depicts the change in morphology of nano-catalyst post-treatment. Here, the nano-scale catalyst sample was found to be poly-dispersed (i.e., from nano-scale particles to micron scale aggregates). In near-neutral conditions, less than 1% leaching of iron was detected in each run. The increase in heavy metals could possibly be explained by their conversion from organic forms to inorganic form of the heavy metal. Though the inorganic form is relatively less toxic, further examinations are required to accurately attribute the reason for such an increase in concentration.

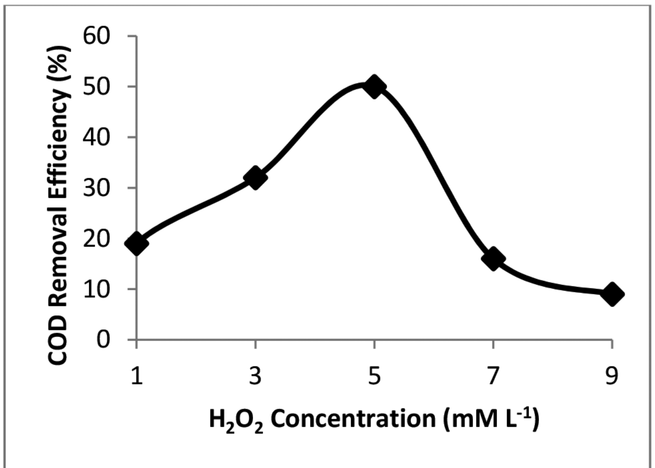

Figure 4: COD removal efficiency against various H_2O_2 concentrations (1-9 mmol L^{-1}).

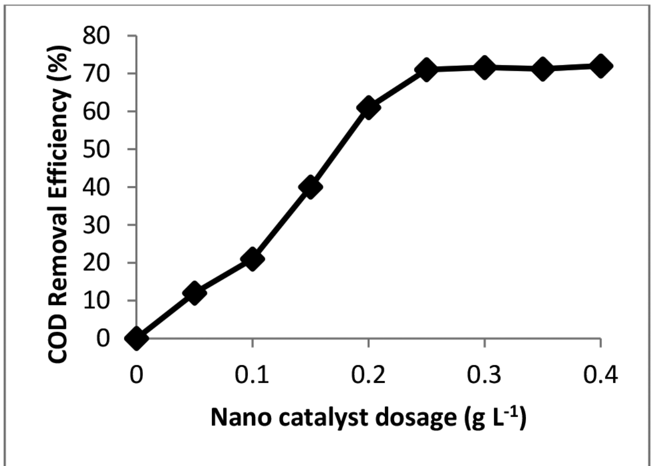

Figure 5: COD removal efficiency against nano catalyst dosages (0.1-0.4 g L^{-1}).

Conclusion

In this study, nano-scale iron oxide was employed to treat Byramangala lake water sample. It is unique that low cost materials were used in near-neutral reaction conditions at room temperature to treat a complex water matrix. Under optimized conditions, overall COD removal achieved was 78.5%; α-, and δ-HCH as well as heptachlor epoxide were removed 100% and 99%, respectively. Significant reduction in some heavy metals, namely, Mn and As by 76.6% and 95%, respectively was observed. Others, like Cd have remained constant at 0.0001 mg L^{-1}. However, other metals like Cr, Cu, Fe, Ni, Zn and Pb have been detected in higher concentrations. Further studies utilizing Eh-pH diagrams could be undertaken in order to provide a possible explanation for such behaviour of heavy metal ions. The increase in iron concentration can be associated with the leaching of catalyst. However, overall, the observed increased concentrates still met the prescribed standards.

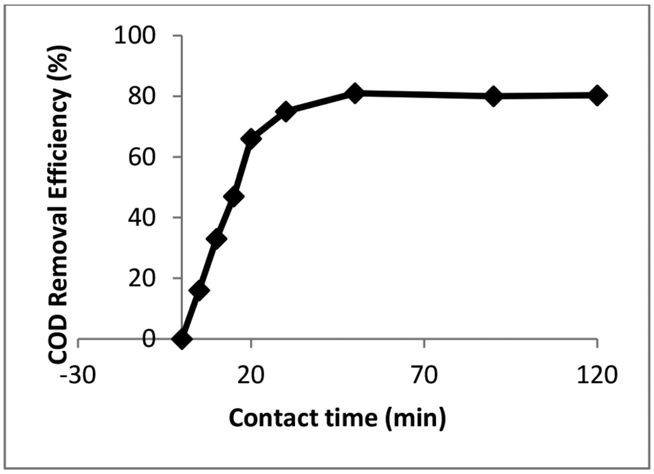

Figure 6: COD removal efficiency against contact times (5-120 min).

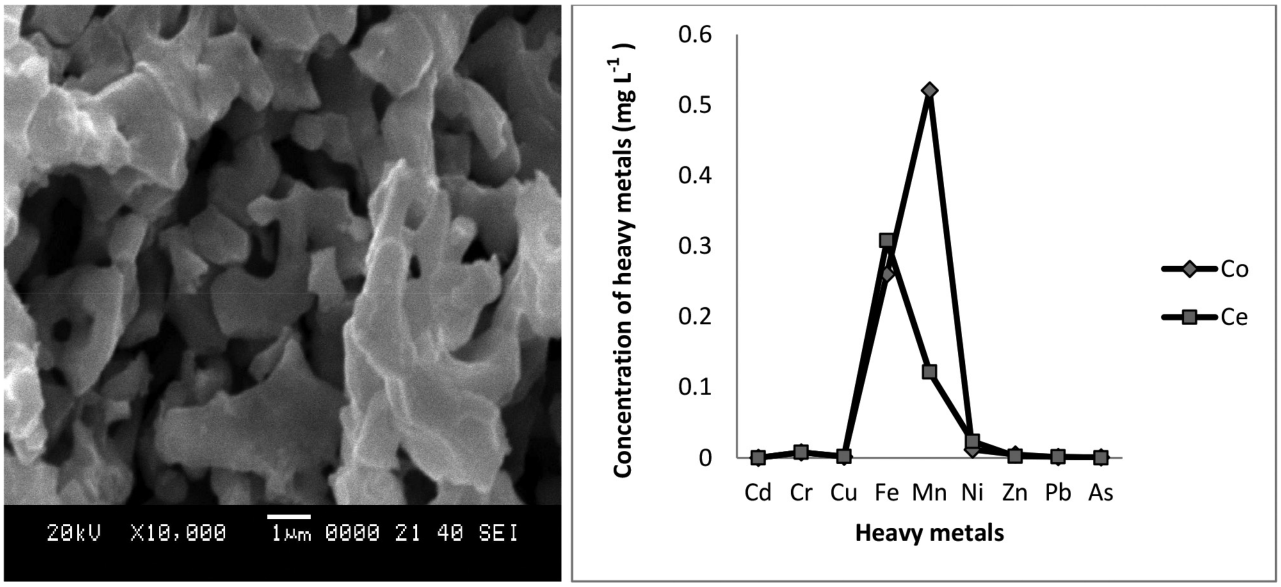

Figure 7: Post treatment effects on: (a) surface morphology of the nano catalyst, and (b) concentration of heavy metals in sample.

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Asian Journal of Water, Environment and Pollution

Volume 15 Number 3 **July – September, 2018**

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