Removal of Colour from Aqueous Solutions by using Zero Valent Iron Nanoparticles

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Abstract: *Nanoscale studies are gaining importance for the removal of chemical substances, dyes or other pollutants from the wastewater to reduce the future environmental impacts. Among them iron and its oxide nanoparticles have been successfully utilized for the elimination of hazardous and toxic wastes due to their catalytic, super-magnetic property and greater efficiency. The present study evaluated the removal of various dyes from water solutions by using ZVI (Zero Valent Iron nanoparticles). Synthesis of nanoparticles was carried out by borohydride chemical reduction method. Its characterisation was done by using X-Ray diffraction (XRD), Scanning Electron Microscope (SEM) and Brunauer- Emmett-Teller method (BET). SEM images showed that the size of synthesized nanoparticles were mostly in the range of 30-100μm in diameter. Some particles were less than 30 μm in size. Batch experiments were carried out to investigate the adsorption capacity of zero valent iron nanoparticles for various dyes from aqueous solutions. The ZVI particles showed maximum adsorption capacity (5339.3mg/g) for Drimarene dye S-RB (Reactive Red 198). The effects of pH, contact time, electrolyte, initial dye concentration and detergent were also investigated. The nano particle works effectively at acidic pH (3-6) but may also be used at basic or neutral pH. ZVI particles may be used more effectively for the removal of dye in the presence of salts.*

Keywords: adsorption capacity, colour removal, nanoparticles, reactive dyes, wastewater, zero valent iron.

I. Introduction

Dyeing industries are considered as one of the biggest water polluters in the world because the effluent released from these industries consists of toxic compounds and variety of dyes which are carcinogenic in nature, highly coloured and unmanageable to degradation. As a result, colored wastewater generated from the different industries is the major concern for the environment [1].

Different type of dyes such as reactive, acid, basic, disperse, sulphur and vat depending upon the requirement are used by the textile industries. In India, about 80% of textile industry use pure cotton fibre and its blend with other fibres [2] which mainly uses reactive dyes for the dyeing process because it gives a high degree of fixation.

Reactive dyes exhibit the presence of potential chromophoric system i.e. the reactive group or sulphonate groups for water solubility and the bridging group that attaches the reactive group either directly to the chromophore or to some other part of the dye molecule to increase the probability of reaction with the fibre [3]. Reactive dyes are characterised by nitrogen to nitrogen double bonds (N.N azo bonds) and the presence of coloured molecules (chromogens). The dyes are specifically used for cellulose, get easily absorbed onto it and then react with the fibre. The reaction forms the covalent bond between the dye molecule and the fibre [4].

Sulphur, disperse and vat dyes are water – insoluble. Sulphur dyes are synthetic organic substantive dyes, produced by the thionation or sulphurisation of organic intermediates containing nitro or amino group. Vat dyes are composed of indigo and the anthraquinone derivatives that are used particularly on cellulosic fibres. Disperse dyes are often substituted azo, anthraquinone or diphenylamine compounds which are non-ionic and contain no water solubilising groups. The dyes particle are thus held in dispersion by the surface-active agent and the dyes themselves are called disperse dyes.

The major environmental impact of dyes and other products are water pollution, mutagenic effects, degradation of soil quality etc. Studies have outlined the applicability of several inorganic and biological adsorbents and a number of technologies such as electrochemical precipitation, bio-adsorption/ bio-oxidation, ozonation, membrane separation processes, physicochemical treatment and chemical oxidation [5] for the removal of dyes. Adsorbents such as Activated carbon, fuller's earth and silica, synthetic clay [6,7] have shown potential in achieving high adsorption capacity; however, most of the biological adsorbents suffer from low adsorption capacity such as maize cob, wood and rice hull [8,9].

To combat this problem, Iron is emerging as an important industrial metal used in various removal processes. Zero valent iron nanoparticles are intensively used as a reactive medium for wastewater treatment (containing chlorinated organic compounds [10], toxic metals [11] and inorganic Compounds [12,13] because the iron metal is of low cost, act as strong reducer, possess higher efficiency and contaminant degradation ability. In the destruction of toxic dyes, Fe^0 nanoparticles gets oxidised and dye molecule undergoes reduction process. This particle exhibits an enhanced reactivity due to their higher surface area [14] for the degradation of halogenated compounds. Textile effluents containing dyes and other organic compounds require fast treatment or decolourisation due to their toxic health and environmental effects. Zero-valent iron (ZVI) particles are inexpensive, environmentally friendly, exhibits fast reaction and can donate two electrons to many environmental contaminants [15, 16]. Recently many studies have been reported the increased reducing effect of Fe^0 with different catalytic metal and supported nanoparticles [17]. Over the years, laboratory studies have reported their growth in experiments and modification to have better surface properties, efficiency enhancement for reactions [18].

In the present paper, studies have been carried out on the removal of reactive, sulphur, disperse and vat dyes with the synthesized zero valent iron nanoparticles. The synthesis of ZVI particles has been optimized and the material obtained has been characterized by various instrumental techniques such as XRD (X-Ray Diffractometer), SEM (Scanning Electron Microscopy) and BET (Brunauer–Emmett–Teller) isotherm studies. Its adsorption capacity for various dyes has been evaluated. The experimental conditions such as pH, contact time, concentration of dye have been optimized for a reactive dye. Effect of salts and auxiliaries on dye adsorption has also been studied for its application in removal of colour from dyeing effluents.

## II.   Materials And Methods
2.1 Dyes and Chemicals Used
Commercially available Drimarene Reactive dyes (Yellow HE6GB, Orange F2RI, Black SNN, Blue HEGN and RED-SRB) and Disperse dye (Foron Brill Red ST) were obtained from Clariant Colorants, Mumbai. Other dyes such as Vat dye (Jade Green FFB) and Sulphur Dye (Navy Blue) were obtained from Monomer Chemical India Pvt. Ltd, Mumbai.

Reducing agent Sodium borohydride ($NaBH_4$) and chemical compound ferrous sulphate heptahydrate ($FeSO_4.7H_2O$) were procured from Merck chemicals to prepare nanoparticles. All other reagents used were of analytical grade. Fe^0 nanoparticles used for the study were synthesized in the laboratory by the reduction of $FeSO_4.7H_2O$ with $NaBH_4$.

2.2 Preparation of Zero Valent Iron Nanoparticles
Synthesis of ZVI nanoparticles was done by chemical reduction method. Sodium borohydride was used for reduction of $FeSO_4.7H_2O$ aqueous mixture [19]. Molar solution of $FeSO_4.7H_2O$ (0.1M) was reduced by using $NaBH_4$ (0.4M) which was added slowly into it under continuous stirring by magnetic stirrer at room temperature (25 ± 2^0C). The mixing volume was taken in 4:1 ratio for faster reduction of particles. The process involves the following reaction [20].

$$4Fe^{3+} (aq) + 3BH_4^- + 9H_2O \rightarrow 4Fe^0(s) + 3H_2BO_3 + 12H^+ (aq) + 6H_2 (g)$$
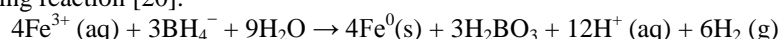
During the reduction reaction colour changes immediately into black with the formation of colloidal precipitate. Solution containing magnetic iron nanoparticle was stirred continuously for another 5 minutes and the particles were separated by centrifugation at 3800rpm for 3minutes. After separation, the precipitate was washed 3-4 time with distilled water to remove excess sodium borohydride and then by ethanol for at least 2 times. The washed precipitate was then oven dried at 60^0C for 2hours.

2.3 Characterisation
The synthesised nanoparticle was characterised with the help of various instrumental techniques such as XRD (X-Ray Diffraction), SEM (Scanning Electron Microscopy) and BET (Brunauer–Emmett–Teller) isotherm studies

2.3.1   XRD
XRD method was used to analyse the structure of iron nanoparticles. Patterns were recorded over a 2-theta (2θ) configuration by using Rikagu Bench top Mini Flex diffractometer operated at a voltage of 40Kv with X-ray source Cu-kα radiation (1.546A) and graphite monochromator to produce X-rays. Iron nanoparticles were placed in a glass holder and scanned from 3^0-50^0.

2.3.2 SEM

SEM observations were performed by mounting the samples onto a carbon tape coated with gold sputter thin film under high vacuum mode and observed at 15kv-20kV using a FEI-QUANTA 200F, Netherlands. The magnification was adjusted above 1, 00,000 .i.e.120, 000x-160,000x (Fig. 2a, 2b).

2.3.3 BET

The surface area of nanoparticles was analysed by BET adsorption isotherm. It is based on determining the extent of nitrogen adsorption on a given surface. The particles were dried at different temperatures i.e. room temperature, 90^0C and 200^0C and then exposed to nitrogen gas (analysis adsorptive) to determine the surface area by using surface area analyzer (Micromeritics ASAP 2010, USA) at 77.3 K.

2.4 Chemical and Thermal Stability of Nanoparticle

Chemical stability of the nanoparticle was evaluated by equilibrating it with solutions of different pH values at 25^0C for 2 hours. Distilled water (100ml) was taken in several conical flasks and their pH was adjusted in the range 1 to 13 with HCl and NaOH. Powdered nanoparticles (0.1g) was added in each conical flask containing pH adjusted distilled water. The mixtures was shaken on an electrical shaker for 2 hours at 25 ± 2 ^{0}C. The samples were then filtered using previously dried filter papers. The filter papers were dried in a hot air oven at 103^0C for 1-2 hours and weighed after cooling in a desiccator. During filtration sample residues were rinsed with distilled water 2-3 times to remove the excess of acids or alkalis and then placed in an oven at 103^0C for 2 hours. The residue along with filter paper was weighed after cooling in a desiccator.

For determination of thermal stability weighed amount (0.1 g) of adsorbent in a beaker was kept at different temperatures (30, 45, 60, 75 ^{0}C) and reweighed after cooling in a dessicator.

Percent loss of adsorbent was then calculated using the following equation-

%Wt. Loss of adsorbent= (Wt. of adsorbent taken - Wt. of residual adsorbent/Wt. of adsorbent taken)*100
Where, Wt. of residual adsorbent= ((Wt. of residue + Wt of filter paper/beaker) - Wt of filter paper/beaker

2.5 Evaluation of dye concentration

The dyestuff concentration in each solution was determined by using UV-Vis spectrophotometer of Shimadzu (U.V-160A), Japan based on Beer-lambert law. The wavelength for the maximum absorption of dye was evaluated and all measurements were taken at (λmax). A calibration curve was prepared by plotting dye concentration against the measured absorbance using a set of seven standards of dye concentration i.e. 5,10,20,30,40,50,60 mg/l for each dye solution. Distilled water was used as zero blank during these measurements. The equations obtained from the calibration curves were used for the estimation of dye concentrations in the solution.

2.6 Equilibrium Studies

Equilibrium studies were carried out by using batch technique to determine dye adsorption capacity of the nano particle and effect of various process parameters like pH, time, dye concentration, presence of salts, nanoparticle dosing on the adsorption capacity.

The adsorption capacity was determined by adding 0.01 g of ZVI to solutions (100 ml) containing the dye in a 250 ml conical flask. After equilibrating for 2 hours at room temperature (25 ^{0}C) the nanoparticle was removed from the dye solution by filtering through a filter paper and absorbance of filtrate was measured at λmax . The experiments were carried out by using reactive, disperse, vat and sulphur dyes. Control tests were also carried out in the absence of nanoparticles under the same experimental conditions.

The concentration of residual dye in the solution was calculated from the equations obtained from the calibration graph. The adsorption capacity of the adsorbent was evaluated by determination of quantity of dye uptake from concentration of dye before and after equilibration using the following equation [21].

$$Q = C_i - C_f \; V/W$$

Where Q = Adsorption capacity/ Quantity of dye uptake (mg/g)
C_i = Initial concentration of the dye in the solution (mg/l)
C_f = Final concentration of the dye in the solution (mg/l)
V = Volume of the dye solution (ml)
W = Weight of the adsorbent (g)

Drimarene Red-SRB (Reactive Red 198, C.I.18221) was taken as a model to carry out the detailed study on adsorption behaviour of the nano-particle because maximum adsorption capacity was observed for this particular dye.
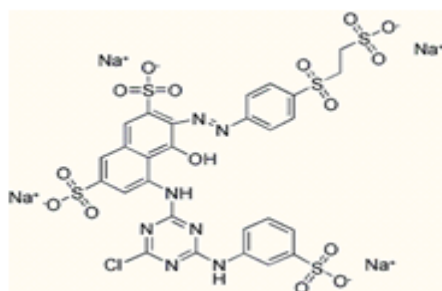

Figure 1 Chemical structure of Reactive Red S-RB (Reactive Red198)

The effect of process variables such as contact time, pH, salinity, dye concentration, detergent concentration and nanoparticle dosing were studied on adsorption capacity (uptake of dye) by equilibrating 0.01 g of the nanoparticle with 100 ml dye solution. The effect of contact time was studied by equilibrating the nanoparticle (0.01 g) and dye solution (3000 mg/l)) mixture for different time intervals (5-240 minutes) and determining the dye concentration left in the solution. Similarly, Effect of dye concentration was studied by adding 100 ml of dye (1000-5000 mg/l) to the conical flask containing the nanoparticle (0.01 g). Effect of pH was studied by adjusting pH of the solution (3-10) by adding HCl or NaOH as per the requirement. The effect of nanoparticle dosage was studied by adding different amount of nanoparticle (0.01-0.05 g) to the dye solution (3000 mg/l). The effects of salt and detergent concentrations were studied by adding different quantities of NaCl (10-60 g/l), Na_2CO_3 (5-30 g/L) and Extran detergent (0.1-0.3 g/L) to the mixture containing nanoparticle (0.01 g) and dye solution (3000 mg/l).

III.   Results And Discussions
3.1 Structure of nanoparticles

SEM micrograph (Fig. 2 a, b) clearly depict that iron nanoparticles are present as nanospheres and reported as to form chain or linear structures due to magnetic forces between the particles [23]. The laboratory prepared nanoparticles were largely spherical and showed single particle size around 50-80nm. Some particles were having size of less than 30nm. This variation in nanoparticles size and linear orientation may be related to strong magnetic properties of iron itself.
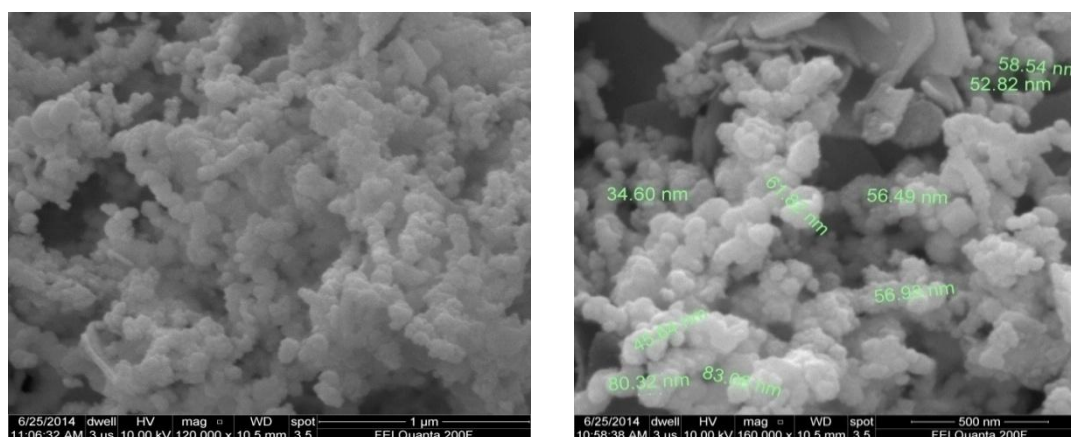

Figure 2 (a & b): SEM images showing aggregates of iron nanospheres and their size

XRD pattern of the synthesised particle is shown in Fig. 3. The broad peak at 2θ value of 45.6^0 gives the presence of zero valent iron nanoparticles. The crystalline size measurement was calculated using Scherer equation [22].

$$D = K\lambda/\beta Cos\theta$$

Where   λ= wavelength of incident ray (1.5418Å)
         K= shape factor (0.9)

θ is the Bragg angle
β = is the line broadening at half the maximum intensity (FWHM), it is also denoted as 2θ
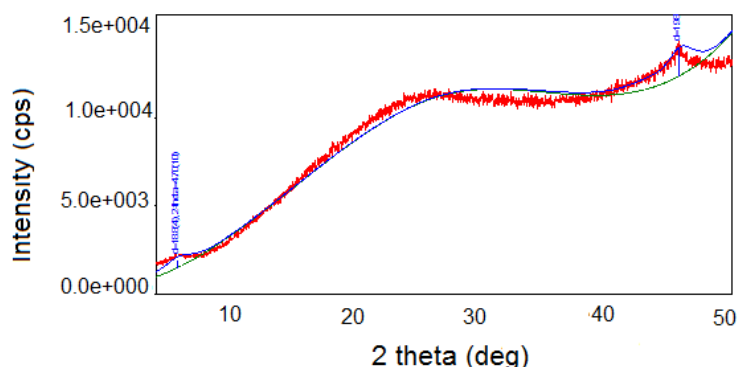D = Mean size of the ordered (crystalline) domains

Figure 3: XRD pattern of nanoscale zero valent iron

The mean crystalline size, D comes around 1.98497nm and size of one crystal particle is near about 29.6nm from the XRD pattern.

From BET isotherm studies it was found that single point surface area at relative pressure (P/P_O) 0.30042107 is 24.6173 m²/g. BET surface area was found to be 24.8080 m²/g. Langmuir Surface Area was calculated as 37.8706 m²/g. The adsorption average pore diameter and total pore volume were found to be 105.8837 Å and 0.065669 cm³/g respectively.

3.2 Chemical stability
Chemical stability of the nanoparticle was evaluated by equilibrating the nanoparticle with solutions having different pH values ranging from 1-13 and determining the loss in its weight. Fig. 4 shows that the nanoparticle is stable over a wide range of pH of the solution. It is more stable in pH range 3-13 because there is negligible weight loss in the nanoparticle in this pH range. It was highly unstable at pH 1 and had a considerable weight loss at pH 2. This suggests that the nanoparticle can be used in slightly acidic, neutral and basic medium without any significant weight loss in the material.
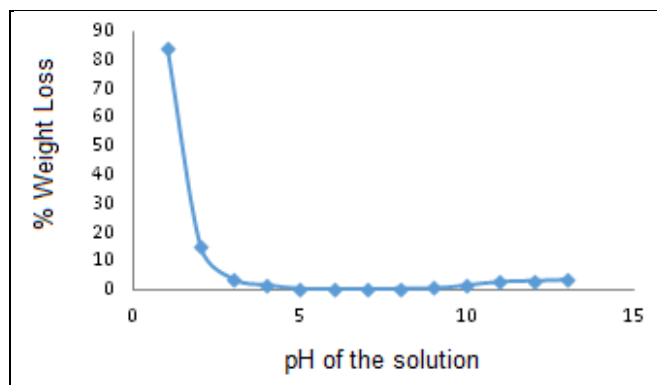

Figure 4: % weight loss of the adsorbent at different pH of the solution

3.3 Thermal Stability
Thermal stability of the nanoparticle was evaluated by keeping it at different temperature ranging from 30-75 ^{0}C and determining the loss in its weight. Fig. 5 shows that there was negligible weight loss in the nanoparticle when the temperature was increased from 30 to 60 ^{0}C but there was considerable weight loss on further increasing the temperature from 60-75 ^{0}C. The slight weight loss from 30-60 ^{0}C may be attributed to removal of moisture content from the nanoparticle and weight loss from 60-75 ^{0}C may be due to decomposition of the material at higher temperature. This suggests that nanoparticle can be used safely in the temperature range 30-60 ^{0}C.
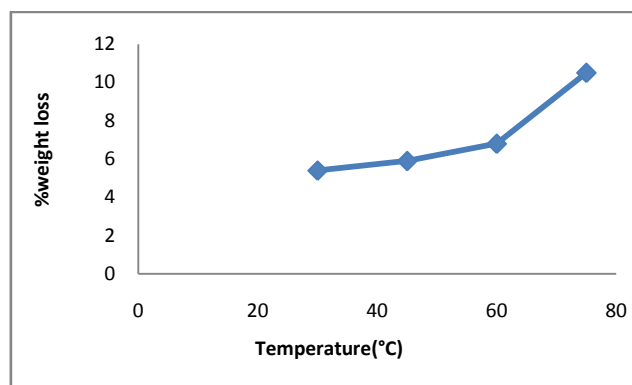
Figure 5: % weight loss of the adsorbent at different temperature

3.4 Adsorption Capacity

Zero valent iron nanoparticles have magnetic and electrostatic properties due to which dyes get attached to the nanoparticle. The decolorization of reactive dyes in aqueous solution by ZVI particles mostly occurs via the reductive degradation of dye molecules. ZVI can donate two electrons to the dyes and hence act as a strong reducer. The reductive cleavage of the azo bonds of reactive dyes is accompanied by the oxidation of Fe^0, resulting in the decolorization of the dye. In the destruction of dyes, Fe^0 nanoparticles get oxidised and dye molecules undergo reduction process.

$$Fe^0 + 2H^+ \rightarrow Fe^{2+} + H_2$$
$$-N\ N- + 2H^+ + 2e^- \rightarrow -NH-NH-$$
$$-NH-NH- + 2H^+ + 2e^- \rightarrow -NH_2 + H_2N-$$
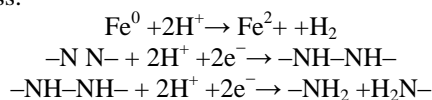
The heterogeneous reaction using ZVI involves five steps [25]
   i.   Mass transfer of reactive dye to the ZVI surface from the bulk solution,
   ii. Sorption of dye on the ZVI surface
   iii. Reductive degradation of reactive dye at the ZVI surface
   iv. Desorption of the reaction product from the ZVI surface
   v.   Mass transfer of the product into the bulk solution.

Adsorption capacities of the ZVI nanoparticle for various dyes with their commercial and generic name as reported in the colour index [19] are summarized in Table 1. The nanoparticle showed maximum adsorption capacity for Reactive Red-198 dye. Therefore, this dye was chosen for further studies.

Table 1 Adsorption capacity of various dyes

Type of Dye	Commercial name	Colour Index number(C.I.)	Adsorption capacity(mg/g)
Reactive	Yellow HE6GB	Reactive Yellow 135	2086.1
Reactive	Orange F2RI	Reactive Orange 122	4702.5
Reactive	Black SNN	_	3647.4
Reactive	Blue HEGN	Reactive Blue 198	964.6
Reactive	RED-SRB	Reactive Red 198	5339.3
Disperse	Foron Brill Red ST	Disperse Red 152	3780.4
Vat	Vat Jade Green FFB	Vat Green 1	2764.4
Sulphur	Navy blue	Sulphur blue 5	2587.4

3.5 Effect of Contact Time

Fig. 6 shows that there was a gradual increase in dye uptake by the nanoparticle with increase in contact time up to 120 minutes in the case of all the three dye concentrations (3000, 4000, 5000 mg/l) chosen for equilibration with the nanoparticle. After that it became almost constant (up to 240 minute). This suggests that rate of adsorption of dye on the nanoparticle is quite fast. Therefore, 120 minutes (2 hours) equilibration time was taken as standard contact time for further studies.
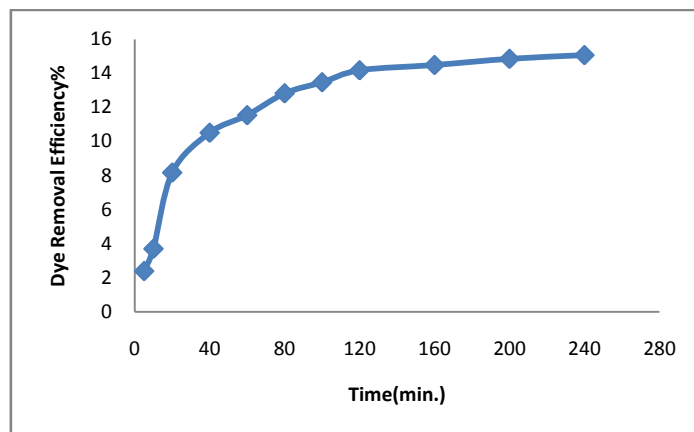
Figure 6: Effect of contact time on Dye removal efficiency

3.6 Effect of pH

The pH of textile waste water varies widely from acidic to basic range. Variation in pH is one of the most significant factors which affect the extent of decolourisation by physicochemical treatment. Therefore the effect of pH of dye solution on adsorption capacity of the nanoparticle was investigated. Fig.7 shows that the nanoparticle has high adsorption capacity (3750-4131mg/g) and remained almost constant from pH 3-10. This suggests that the nanoparticle can be used for de-colorization of textile waste water having a pH in this range. Above pH 10 there was a sharp decrease in adsorption capacity and it dropped to less than 2000 mg/g at pH 13. The low adsorption capacity at alkaline pH may be attributed to a decrease in reducing power of nanoparticles due to covering of nanoparticle with corrosion products; there is also a change in the charge of ZVI surface from positive to negative at alkaline pH which causes a repulsion force between the dye and nanoparticles [24].
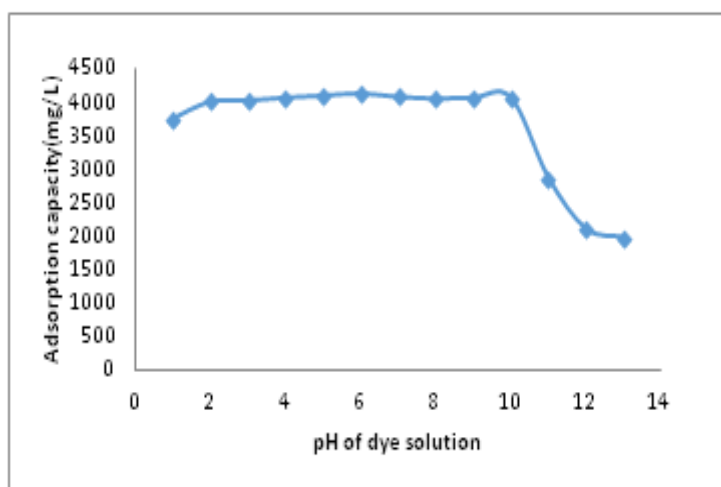

Figure 7: Effect of pH on the adsorption capacity

3.7 Effect of adsorbent quantity

The effect of ZVI dosages on the removal of red dye was carried out by using different quantities of the nanoparticle ranging from 0.01g to 0.05g. The experiment was done at room temperature and monitored for 2hours. Percent adsorption of the dye increases with the increase in nanoparticle dosage as shown in Fig. 8. It may be due to more surface sites available for reaction with reactive dye to accelerate its removal. However, a decrease in overall adsorption capacity was observed with the increase in adsorbent dosage because adsorption of the dye does not increase in the proportion of increase in nanoparticle dosage (Fig. 9). The maximum adsorption capacity was observed when 0.01 g adsorbent was added to 100 ml of 3000 mg/l dye solution. Therefore, this nanoparticle to dye liquor ratio was taken for all other studies.
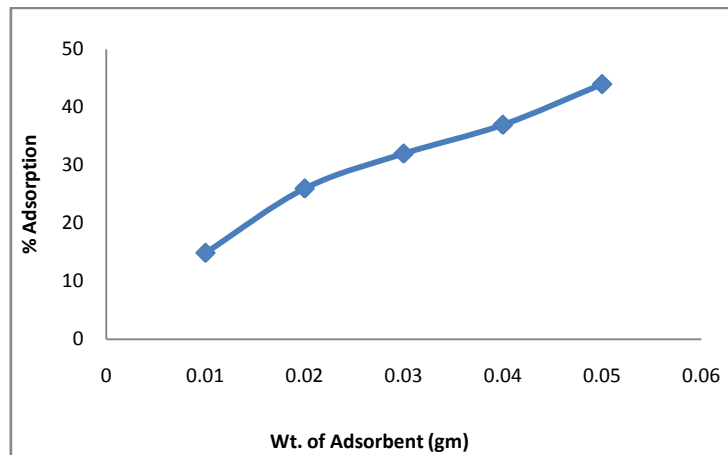
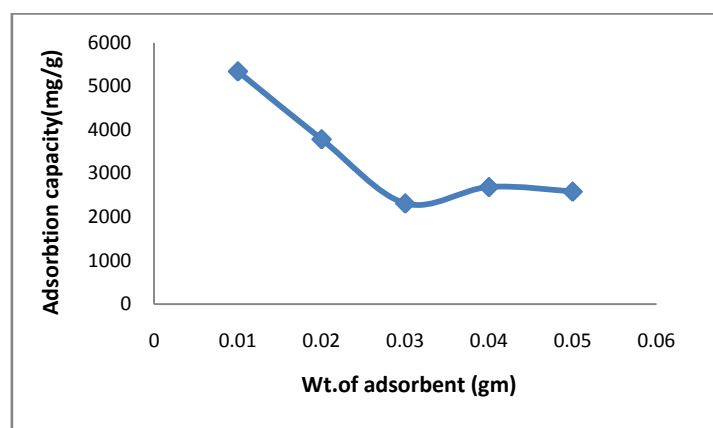Figure 8: Effect of adsorbent dosage on % adsorption of dye

Figure 9: Effect of adsorbent dosage on adsorption capacity of the nanoparticle

3.8 Effect of dye concentration

The effect of dye concentration on colour removal efficiency of ZVI nanoparticles was studied in the range of 1000 to 5000 mg/L dye solution and was monitored for 2hours. The adsorption capacity of the nanoparticle gradually increases with an increase in dye concentration as shown in Fig. 10. This may be attributed to more dye molecules available for the equilibrium. However, there was a gradual decrease in % adsorption of the dye as the concentration of dye liquor was increased (Fig. 11). It is probably because the increase in dye uptake was not in the proportion of increase in dye concentration.
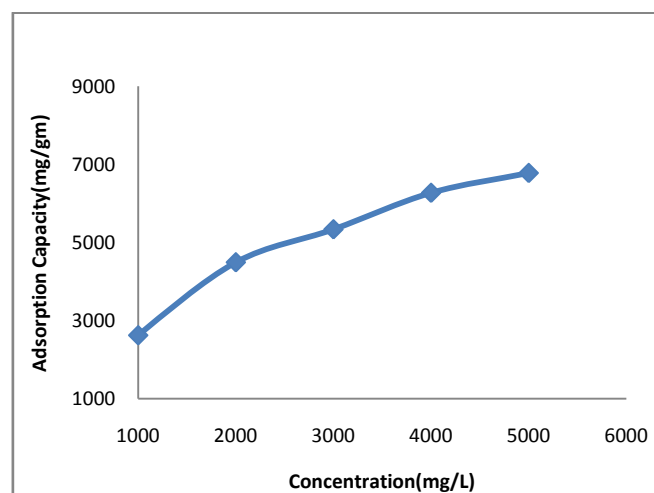

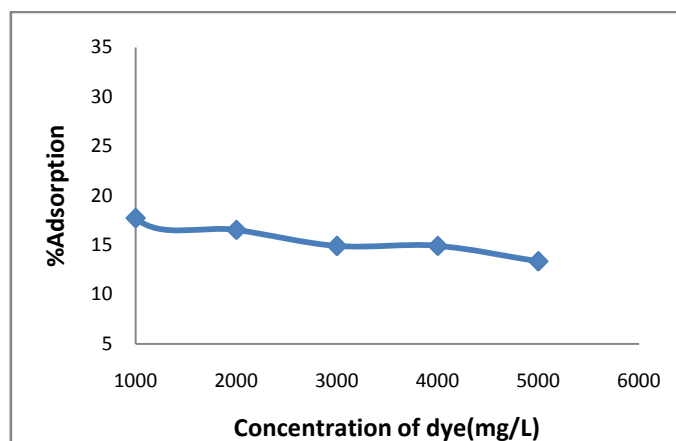Figure 10: Effect of dye concentration on adsorption capacity

Figure 11: Effect of dye concentration on % adsorption

3.9 Effect of salinity on dye adsorption

Textile effluent is characterised by high salinity due to use of high amount of sodium salts in the dyeing process. The concentration of salt is found to vary to a great extent depending upon the type of process adopted. Hence an investigation on the behavioural pattern of the adsorbent was done under varying range of salinity. The experiments were conducted using the dye solution (3000mg/l) in the presence as well absence of sodium carbonate and sodium chloride and the results are shown in Fig. 12 and 13 respectively.

Fig. 12 shows that dye adsorption capacity gradually increases when the concentration of sodium carbonate in dye solution is increased from 5-15 gpl after that it became almost constant from 15-30 gpl. There was a considerable increase in adsorption capacity when NaCl concentration was increased from 10-30 gpl after that it became almost constant from 30-60 gpl (Fig. 13). The increase in dye adsorption by the nanoparticle in the presence of inorganic salts may be due to the following reasons-

1. The inorganic salts neutralize the negative charge in the reactive dye molecule and hence increase the affinity of dye towards electron donating nanoparticle.
2. They also decrease the solubility of the dye in water thus inducing the dye to migrate from water towards the nanoparticle.
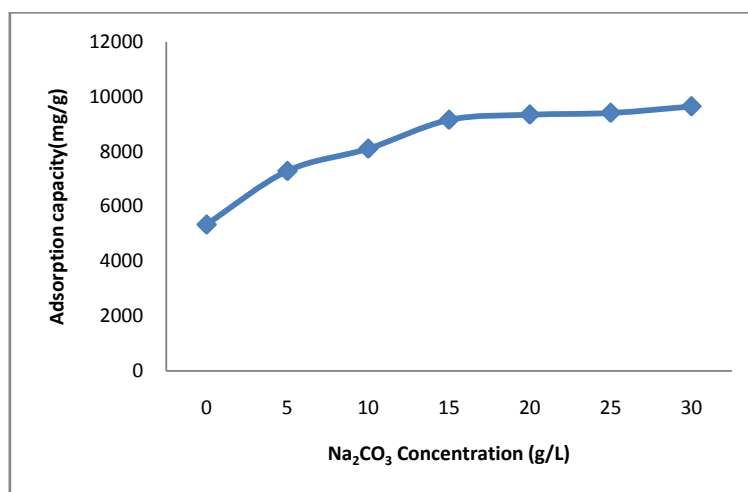

Figure 12: Adsorption capacity in the presence of Na_2CO_3 in the dye solution
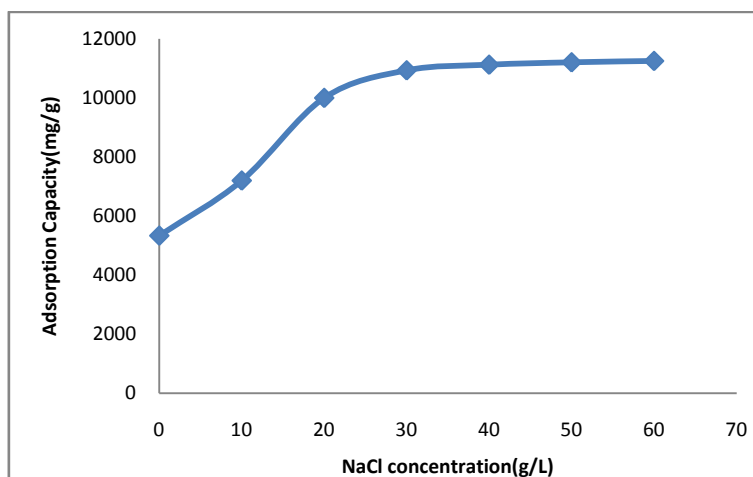
Figure 13: Adsorption capacity in the presence of NaCl in the dye solution

3.10 Effect of detergent on dye adsorption

The effect of detergent on dye adsorption behaviour was studied by carrying out experiments in the presence as well as absence of detergent (Extran MA02, Liquid, and Neutral). The nanoparticle (0.01g) was added to 100 ml of dye solution (3000mg/L) containing 0 to 15 gpl detergent. The results are plotted in Fig. 14, which shows that there was a sharp decrease in adsorption capacity when 5 gpl surfactant was added to the dye solution. Further addition of surfactant (5-15 gpl) had no significant effect and the adsorption capacity became almost constant.

The presence of anionic detergent may adversely affect the dye adsorption by the nanoparticle. ZVI showed less decolorization of the dye in the presence of detergent. This is probably because detergent molecules are preferentially adsorbed by the nanoparticle and the detergent blocks the reactive sites of the nanoparticle. There is electrostatic repulsion between sorbed detergent and reactive dye molecules. Moreover phosphate groups of the detergent passivate reactive sites of ZVI by forming secondary minerals or accelerate non-specific corrosion of ZVI particles.
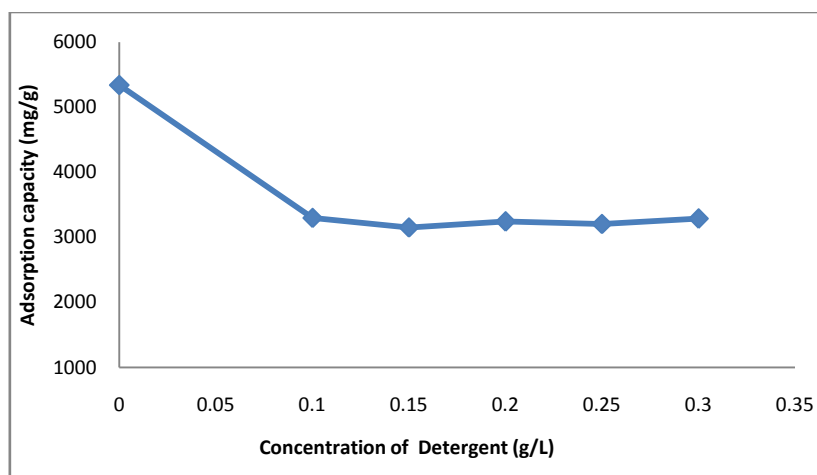

Figure 14: Effect of adsorption capacity in the presence of surfactant in the dye solution

IV.   Conclusion

The prepared ZVI nanoparticle is having a mean crystalline size of 1.98 nm and size of one crystal particle is about 29.6 nm. BET isotherm studies suggest that its surface area is in the range of 24.6-37.87 m^2/g. The nanoparticle is stable over a wide pH range of the solution and over the temperature range of 30-60 ^{0}C. It is capable of decolorizing various types of dyes such as disperse, reactive, sulphur and vat dyes which are used in textile processing. It showed maximum decolorization of reactive dyes particularly Reactive Red-198. Complete equilibrium attained in two hours contact time. It was capable of decolorizing the dye solutions efficiently having pH in the range 3-10. Hence it could be used for decolorization of slightly acidic, neutral and basic dye solutions. There was an increase in % adsorption of dye from the solution with the increase in adsorbent dosage. The study showed an increase in dye adsorption capacity but a slight decrease in % dye adsorption on increasing the dye concentration. Presence of salts resulted in gradual increase whereas presence of detergent showed a

decrease in the uptake of dye by the nanoparticles. This suggests that the nanoparticle is suitable for decolorization of textile effluents containing large quantity of salts.

Acknowledgement

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