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Removal of dye from textile wastewater using zero valent iron nanoparticle
Eliminación del tinte del agua residual textil utilizando nanopartículas de hierro cero valente

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ABSTRACT

Water is a precious commodity which is indispensable and is absolutely necessary for sustenance for life. Although 71% of the earth surface is covered with water which consists of nearly 97.5% salty water and 2.5 % of the freshwater and out of which only 0,007 % is available for drinking. The industrial wastes are a major source of the water pollution which contains various kinds of dyes and other pollutants. Among industries, textile dyeing industries have a wide range of color dyes and bright hues. Over 70,000 tons of approximately 10,000 types of dyes and pigments are annually worldwide disposed of which 20 to 30% industrial effluents are those which are released during the textile curing and finishing process. Dyes have been extremely used in textile, leather tanning, cosmetics, pigments and many other industries. The presence of these dyes in the hydrosphere possesses a significant source of pollution because of their visibility at very low concentration and their nature of recalcitrance which can be lead to undesirable danger to the visibility of aquatic life such as sunlight penetration and resisting photochemical reaction. This waste water generated during the dyeing process increases chemical oxygen demand (COD), and Biology oxygen demand (BOD) levels of aquatic source. The discharge of wastewater without any treatment has an impact on the receiving water bodies crying out for an effluent treatment process. The objective of this project was to identify a nanoparticle that is capable of removing color / dye from textile wastewater.

Keywords: Water, Pollutants, Textile Dyes, Treatment and Nanoparticle.

RESUMEN

El agua es un bien preciado el cual es indispensable y absolutamente necesario para el sustento de la vida. A pesar de que el 71% de la superficie del planeta está cubierto en agua, que consiste en cerca del 97,5% de agua salada y 2,5% de agua dulce, solo un 0,007% del agua dulce es potable. Los residuos industriales son las principales fuentes de contaminación del agua, que contiene varios tipos de tintes y otros contaminantes. Entre las industrias, las industrias de teñido textil tienen una amplia gama de tintes de colores y tonos brillantes, más de 70.000

toneladas de aproximadamente 10.000 tipos de tintes y pigmentos son desechados anualmente en el mundo, de los cuales un 20 a 30% son efluentes industriales que se desechan durante el proceso de curado y acabado textil. Los tintes han sido usados exhaustivamente en textiles, curtido de cuero, cosméticos, pigmentos y en muchas otras industrias. La presencia de estos tintes en la hidrosfera representa una importante fuente de contaminación debido a la visibilidad a muy baja concentración y a su naturaleza recalcitrante, lo que puede llevar a un peligro indeseado en la visibilidad de la vida acuática, como la penetración de la luz solar y la resistencia a la reacción fotoquímica. Las aguas residuales generadas durante el proceso de teñidos incrementan la demanda química de oxígeno (DQO) y los niveles de demanda biológica de oxígeno (DBO) de las fuentes acuáticas. El vertido de aguas residuales sin ningún tratamiento tiene un enorme impacto en las masas de agua receptoras, que requieren un proceso de tratamientos de efluentes. El objetivo de este proyecto es identificar una nanopartícula capaz de eliminar el color / tinte de las aguas residuales textiles.

Palabras clave: agua, contaminantes, tinturas textiles, tratamiento, nanopartículas

INTRODUCTION

The industrial wastes are a major source of water pollution which contains various kinds of dyes and other pollutants. One of the most important serious environmental problem is the existence of harmful and toxic pollutants in the environment which are effluents from industrial or municipal source which contains thousands of chemicals out of which only a few are harmful for aquatic toxicity (V. Premkumar,M.R. Rajan and R.Ramesh, 2016). Textile industrial effluents are highly color dyes with large amount of organic solid which is harmful to aquatic life by their ability to enhance mutagentic and carcinogenic effects. The dyestuff wastewaters are treated before discharge to minimize the threat to the environments. The common dyes includes reactive, disperse, acid and direct dyes. The discharge of wastewater without any treatment has an impact on the receiving water bodies crying out for an effluent treatment process. The releasing of dye into the surrounding water bodies has toxic effect on the human health and also marine life. Therefore removal of these dyes is necessary to protect the environment. This waste water generated during the dyeing process increases chemical oxygen demand (COD), and Biology oxygen demand (BOD) levels of aquatic source. Therefore it is necessary to eliminate dye from wastewater effectively previously to their final discharge to the environment. Large quantities of dye are discharged into the environment from the various industrial processes (Anitha et. al., 2015).

Dyes: Dyes are basically organic compounds that can connect themselves to the surface of fabrics to impart a color. The most commonly used dyes are classified as azo dyes, anthraquinone dyes, phthalocyanine, indigoid dyes, nitroso dyes, nitro dyes and triaryl methance dyes. The azo, anthraquinone and triaryl methane dyes are most important groups among these dyes. The reactive dyes and azo dyes usually forms covalent bonds between the reactive group such as vinyl sulfone, chlorotraiazine, trichloro pyrimidine and difluro chloropyrimidline. In addition, dyes usually used in textile industry are cationic, anionic and non-ionic depending on their particle charge dissolution in aqueous solutions. The complex structure of dyes is ineffective in the presence of heat, light, microbes and even oxidizing agents which make it difficult to degrade. The presence of dyes in water affects sunlight penetration and oxygen solubility which decreases the water quality and thus creates difficulties for photosynthesis on aquatic flora and fauna (Maha A. Tony, Shehab A. and Mansour, 2019).

Dye removal techniques: A wide range of technology has been use for the removal of dyes. They can be divided into three main categories: Biological methods, Chemical methods and Physical methods. High capital costs, low efficiency and generation of excess sludge limit their practical application. Some of these methods have been found to be more versatile and superior to other techniques and are appropriate for removing of a wide range of dyes in wastewater. As a better alternative, adsorption offers the low initial cost, produces nontoxic by-products and completely removes dye even from dilution solution (P. Heera and S. Shanmugam, 2015).

Neem: Neem belongs to family of Meliacease and its role as a health promoter is attributed because of its rich source of antioxidant. It has been widely used in Chinese, Ayurvedic and Unani medicines worldwide and especially in Indian subcontinent in the treatment and prevention of various diseases (Asha Roshan and Navneet Kumar Verma, 2000). The natural products show important role in disease prevention and treatment through the enhancement of antioxidant activity, inhibition of bacterial growth and modulation of genetic pathways. Neem is used in Ayuveda, Unani, Homeopathy and Modern medicine for the treatment of many infections, metabolic and cancer disease (Sushree Priyanka Dash et.al., 2017) (Figure 1).

Figure 1: Neem Leaves

Synthesis of nanoparticle: Nanoparticles can be synthesized chemically or biologically. The chemically synthesized methods are due to the presence of some toxic chemical absorbance on the surface. They are various chemical methods, physical methods and biological methods which are used. The Green synthesis of nanoparticles has been proposed as a cost effective and environmentally friendly alternative to chemical and physical methods. The plant extracts are the best option for cost efficient and require low maintenance. It traditional methods, the green method are more effective for the generation of NPs. Thus, Green synthesis is more effective to reduce the toxicity of NP (P. Christian et.al., 2008).

Biosynthesis: Biosynthesis of nanoparticles using microorganisms is a green and eco-friendly technology. Microorganisms both prokaryotic and eukaryotic are used for the synthesis of metallic nanoparticles such as silver, gold, zirconium, palladium, iron, cadmium and metal oxide. The microorganisms include bacteria, fungi and Actinomycetes (Bhavika Turakhia et.al., 2018).

Zero valent iron nanoparticle: Zero valent iron is inexpensive, non - toxic and is a reducing reagent agent. The zero valent iron is capable of oxidizing organic pollutants. The ZVI have bulk iron and is of low cost, exhibits high reactivity and has good adsorption capacity (Pichsinee Somchaidee and Karaked Tedsree, 2018). The ZVI also possesses strong reducing power and is reactive towards a larger amount of organic and inorganic compound (Shahram Eslamic et.al., 2018). Thus, the Zero valent iron nanoparticle is very useful for environmental waste water pollution remediation.

MATERIALS AND METHODOLOGY

Collection of Sample: Leaves were collected from the local farm. They were washed under running tap water to remove the dirt and were sundried. 10 grams of this dried Neem leaves were taken and washed with distilled water. The leaves were ground in a motor pestle and were put into 100ml of distilled water. The extracts were filtered using filter paper and were used for Phytochemical analysis following standard protocols.

Preparation of Zero Valent Iron Nanoparticle:The Neem leaves were collected and were washed with distilled water. 6 grams of Neem leaves were taken and were cut into small pieces with sterile knife and was boiled in 100ml water for 10 minutes. The extracts were filtered with filter paper and 30ml of ferric chloride was used to wash the leaves and 10ml of plant extract was added to the nanoparticle synthesized. Determination of immediate appearance of color changes indicates the presence of synthesized nanoparticle. The nanoparticle was further characterized by SEM and FTIR.

Decolorization of Textile Waste Water: 50 ml of wastewater was taken and different concentration of ZVI nanoparticles (0.5ml,1 ml and 1.5ml) were added and suspended and was kept under stirring continuously at a required temperature for different reaction time 30 minutes, 60 minutes and 120 minutes. The pH of the samples was adjusting by adding HCL or NaOH solution. Different pH (3-7) was maintained for the reaction. Different temperatures (40, 50 and 60˚C) were also optimized. UV – Vis Spectrophotometer was used for absorbance measurements of the sample. The maximum wavelength used for determination of absorbance was 550nm and 700nm. The amount of absorbance was determined using the following decolorization formula.

Decolorization Formula

$$Decolorization\ (\%) = \frac{(I - F)}{I * 100}$$

Where,

I = initial absorbance

F = final absorbance

RESULT AND DISCUSSION

Qualitative Phytochemical Analysis of Neem Extract: Phytochemical analysis of the neem extract was carried out using aqueous solvent and ethanol solvent extracts. The presence of Alkaloids, Glycosides, Flavonoids, Terphenoids, Carbohydrates, Phenol, Saponins and Steroids were observed in aqueous extract and Tannins in the ethanol extract (Table 1).

Table 1: Qualitative Phytochemical Analysis of Neem Extract

S. No.	TESTS	AQUEOUS EXTRACT	ETHANOL EXTRACT
1	Alkaloids	+	-
2	Glycosides	+	-
3	Flavonoids	+	-
4	Tannins	-	+
5	Terphenoids	+	-
6	Phenol	+	-
7	Saponins	+	-
8	Carbohydrates	+	-
9	Steroids	+	-

Quantitative Phytochemical Analysis of Neem Extract

Quantitative analysis was carried out and the estimation of following compounds were observed (Table 2).

Table 2: Quantitative Phytochemical Analysis of Neem Extract

S. No.	TESTS	VALUE
1	Alkaloids	10.8±3.0308
2	Glycosides	76.6±4.7404
3	Steriods	13.7±8.2398
4	Flavanoids	8.6±2398
5	Carbohydrates	7.1±4.9947
6	Phenols	70.0±1186

Synthesis of Zero Valent Iron Nanoparticle

Neem leaf extract was used to produce zero valent iron nanoparticle. The neem extract was mixed with the FeCl3 solution in the ration 1:2. The reduction of Fe^{3+} into Fe^{0} indicates the immediate change in the pH of the solution and color change to black or brown color. The ferric chloride solution was yellow in color in the distilled water but on mixing with plant extract it immediately changed its color indicating the synthesis of zero valent iron nanoparticle (Figure 2).

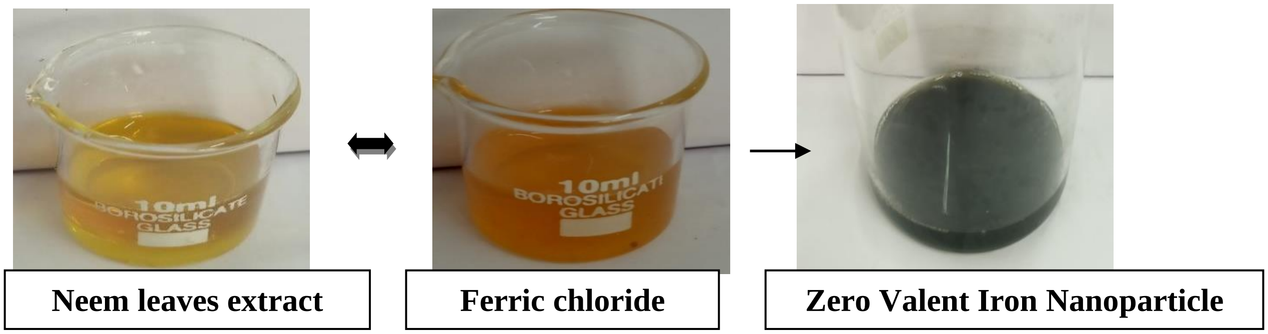


Figure 2: Synthesis of Zero Valent Iron Nanoparticle

Characterization of Zero Valent Iron Nanoparticle By Scanning Electron Microscopy

SEM analysis was carried out to observe the topology, shape and size of the synthesized ZVI nanoparticle. The SEM image of green synthesized zero valent iron revealed sphere shape nanoparticle with an average diameter of 50nm (Figure 3).

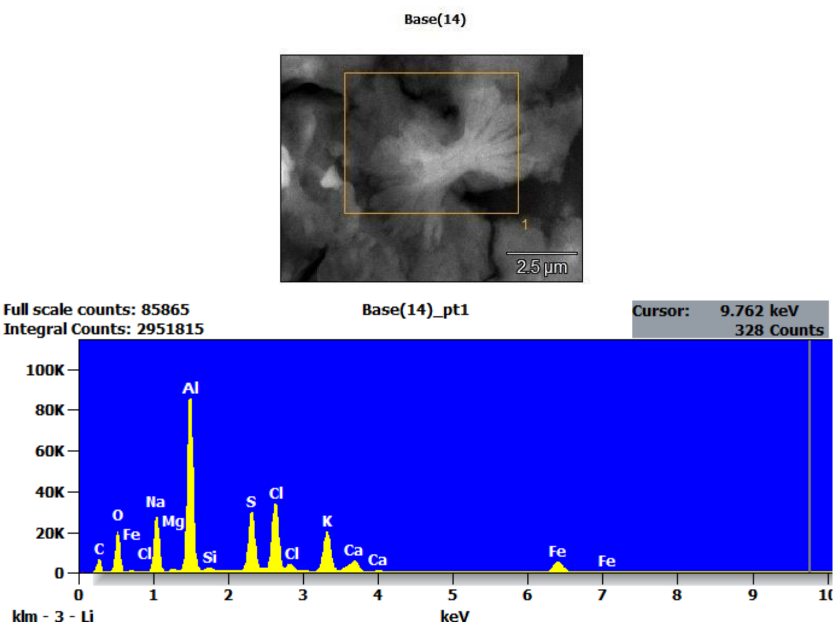


Figure 3: SEM analysis of Zero Valent Iron Nanoparticle

Fourier Transform Infrared Spectrometer (FTIR) Analysis

FTIR analysis was carried out for Neem leaf extract and the synthesized ZVI nanoparticle. The strong bond was observed at 3336.18 and function group to be stretching in O-H of compound carboxylic acid or phenol. The bands of 2139.82 function group is stretching in N=N=N of compound carbodiamide. The function group of 637 is C=C stretching of compound alkenes. The band of 619.47 and 570. 60 both are C-Br stretching function group of compound is halo compound (Figure 4).

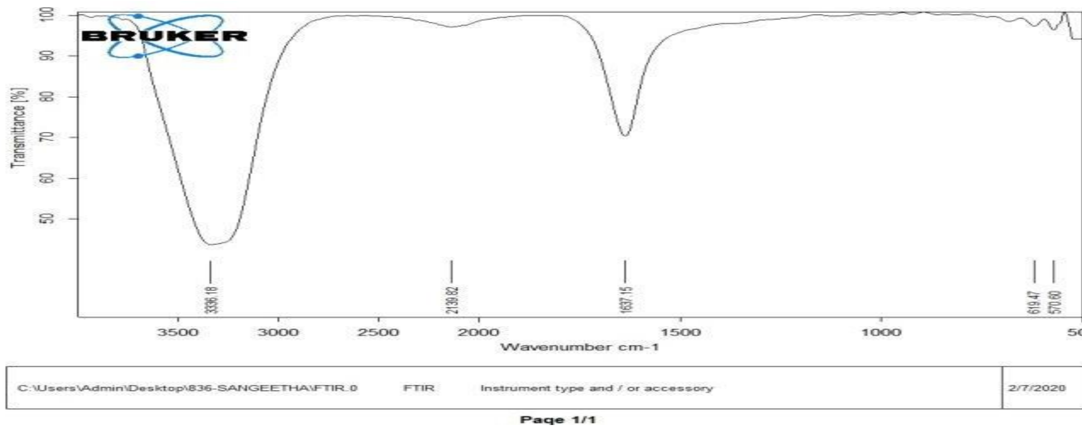


Figure 4: FTIR spectrum of Zero Valent Iron Nanoparticle

Characterization of Textile Waste Water

Textile waste water revealed high concentration of TSS, TOD, COD, BOD, Acidity and Alkalinity (Table 3).

Table 3: Characterization of Textile Wastewater

S. No.	PARAMETERS	SAMPLE – 1	SAMPLE - 2
1	TSS	1.5mg/L	0.7mg/L
2	TDS	5.052 mg	0.894mg
3	DISSOLVED OXYGEN	89mg/L	413mg/L
4	COD	89mg/L	4.313mg/L
5	BOD	919mg/L	2553mg/L
6	ACIDITY	109 N	41 N
7	ALKALINITY	Nil	Nil
8	Ph	8	10.5

Decolorization

Sample 1 was treated with different concentrations of the nanoparticle and pH. The sample was taken in a 50 ml beaker and was treated with different concentrations of nanoparticle such as 0.5ml, 1ml and 1.5ml. The time was noted at 30 minutes, 60 minutes and 120 minutes and pH 4, 6 and 7. 1ml of nanoparticle reported the highest removal of dye in Sample 1. The high decolorization efficiency was at 73.3%, the percentage of decolorization 66.6%, 73.3% and 33.3% respectively (Figure 5 and Graph 1).

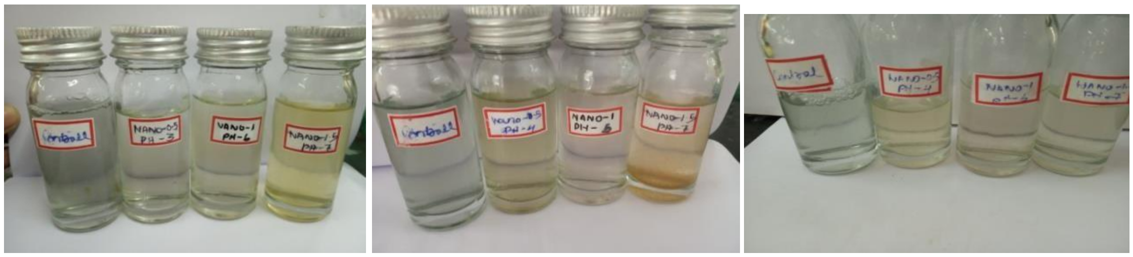
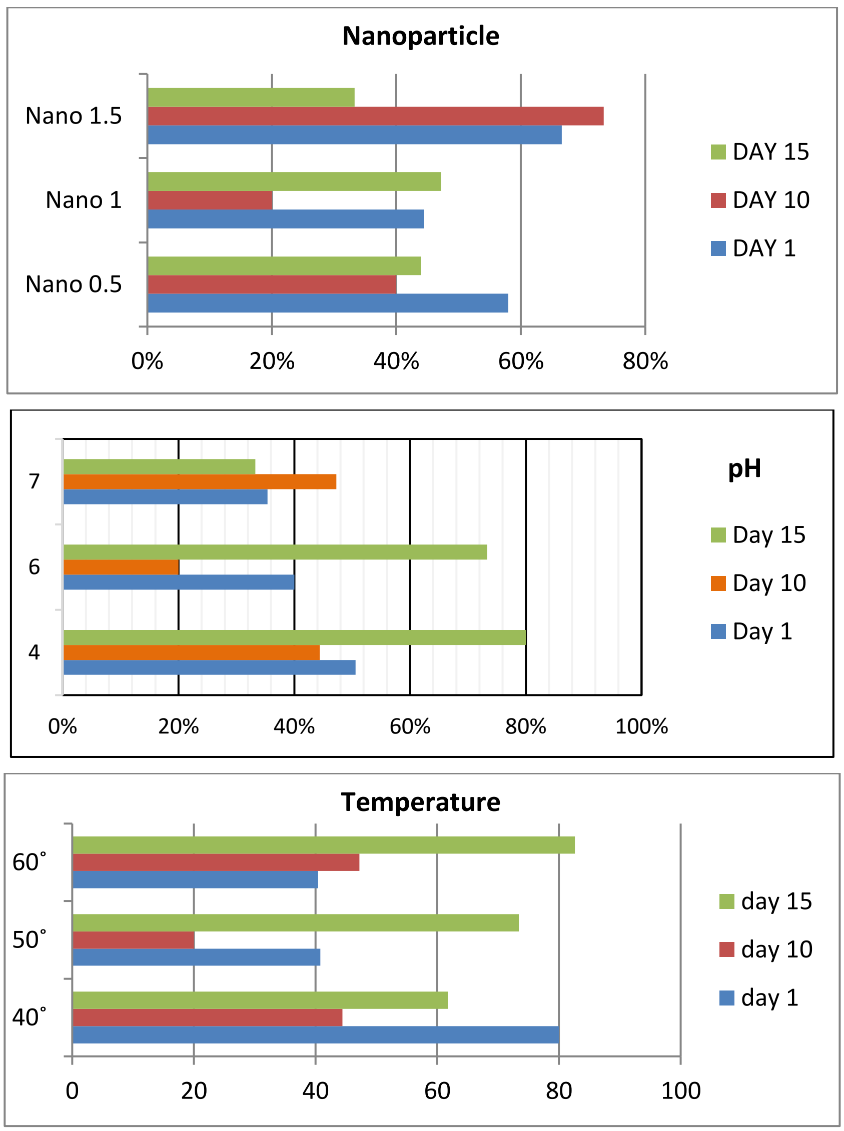


Figure 5: Treatment of waste water (Sample 1) using Zero Valent Iron Nanoparticle



Graph 1: Decolorization of Sample 1

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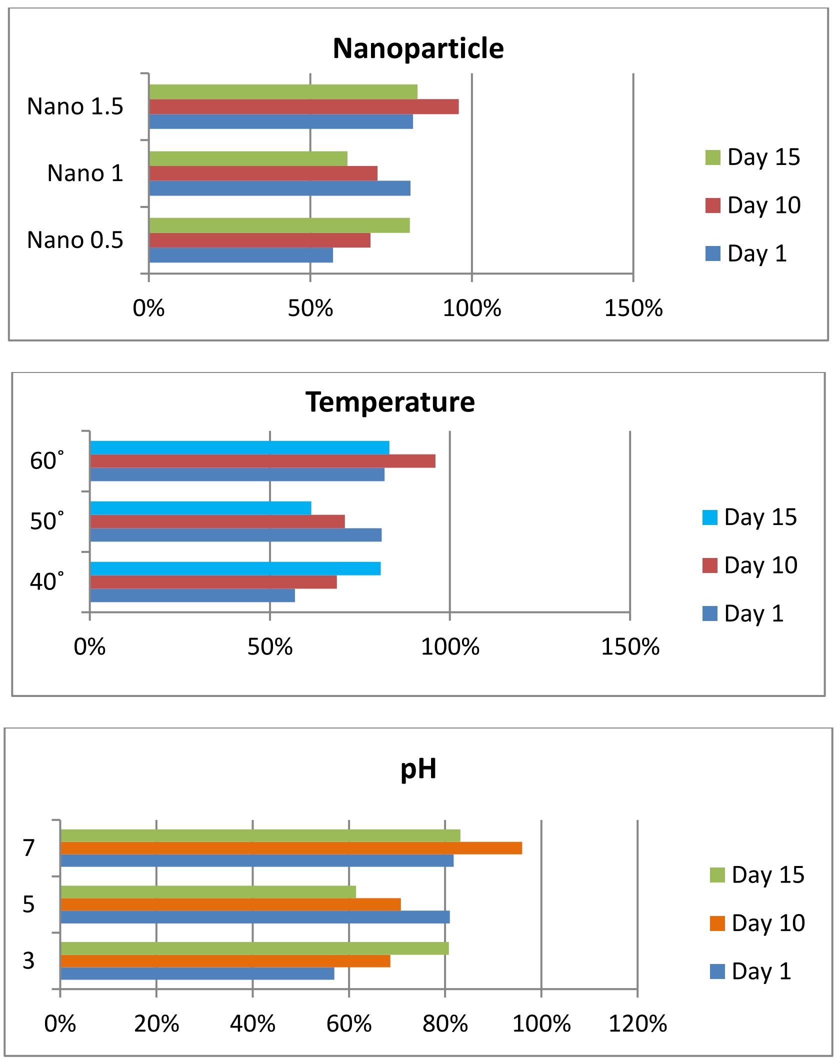
Sample 2 was taken in a 50 ml beaker and was treated with different concentrations of nanoparticle such as 0.5ml, 1ml and 1.5ml, temperature 40^{0}C, 50^{0}C and 60^{0}C and pH 3, 5 and 7. The time was noted at 30 minutes, 60 minutes and 120 minutes.1ml of nanoparticle reported the maximum removal of dye in Sample 2. The percentage of decolorization observed was 81.8%, 96% and 85.2% respectively (Figure 6 and Graph 2).


Figure 6: Treatment of waste water (Sample 2) using Zero Valent Iron Nanoparticle



Graph 2: Decolorization of Sample 2

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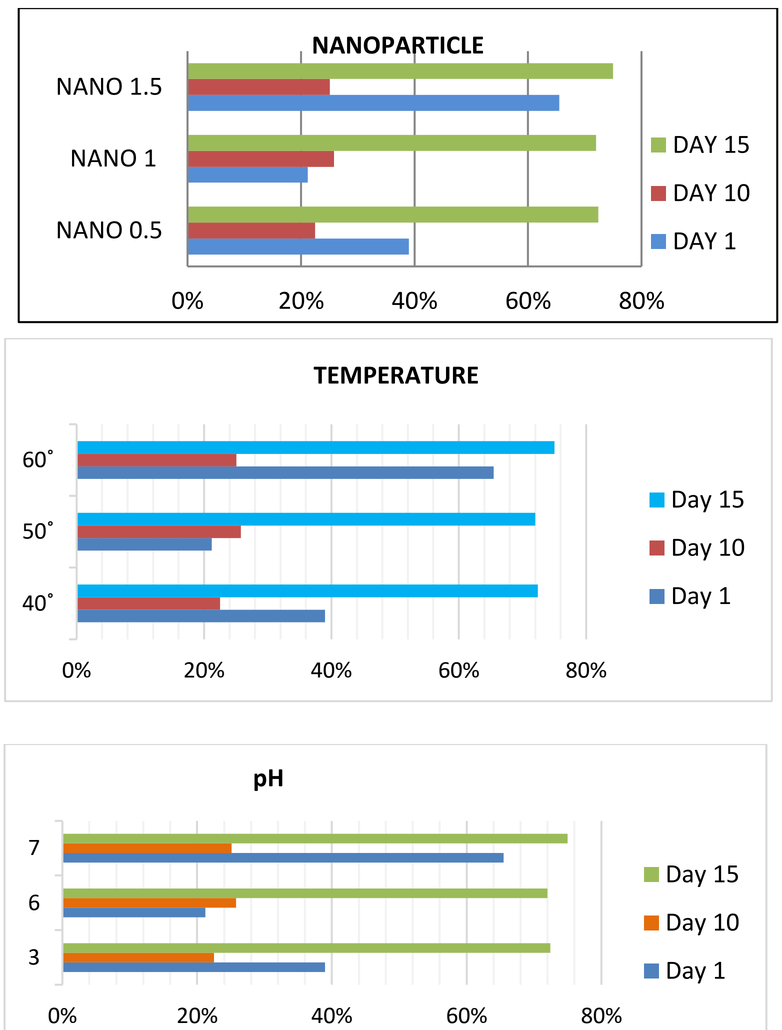
Sample 3 was treated with different concentrations of nanoparticle, temperature and pH. The sample was taken in a 50 ml beaker and was treated with different concentrations of nanoparticle such as 0.5ml, 1ml and 1.5ml, temperature 40^{0}C, 50^{0}C and 60^{0}C and pH 3, 6 and 7. The time was noted at 30 minutes, 60 minutes and 120 minutes. 1.5ml of nanoparticle reported the maximum removal of dye Sample 3. The percentage of decolorization was 72%, 72.7% and 75% respectively (Figure 7 and Graph 3).

Figure 7: Treatment of waste water (Sample 3) using Zero Valent Iron Nanoparticle



Graph 3: Decolourization of Sample 3

Treatment of Textile Waste Water With Nanoparticle

The textile waste water sample treated with the Zero Valent Iron Nanoparticle reported a reduction in Chemical Oxygen Demand (COD) and Dissolved Oxygen (DO) with an increase in the reaction time from 30 to 120 minutes (Table 4).

Table 4: Post Treatment of Textile Waste Water with ZVI Nanoparticle

PARAMETER	SAMPLE 1	SAMPLE 2	SAMPLE 3
COD	0.32mg/L	85mg/L	405mg/L
DISSOLVED OXYGEN	35mg/L	461mg/L	728mg/L

CONCLUSION

Azadirachta indica leaves were subjected to extraction with two different solvents (aqueous and ethanol). The preliminary phytochemical analysis of the extract of Azadirachta indica revealed the presence of compounds such as alkaloids, phenol, tannins, flavonids, steroids, sapponins, terpenoid and glycosides. Zero Valent Iron Nanoparticle was synthesized from Azadirachta indica which was found to remove Dissolved Oxygen and Chemical Oxygen Demand respectively. The synthesized Zero Valent Iron Nanoparticle was characterized by SEM and FTIR. Characterization studies of the textile waste water before and after Zero Valent Iron Nanoparticle treatment showed a profound impact. Zero valent Iron Nanoparticle was found to be very effective in degrading the textile dye and also to remove the color from textile dye waste water by decolorization. Among the three samples used, two of them showed greater color removal efficiency. The Zero Valent Iron Nanoparticle was also used to estimate the Dissolved Oxygen and Chemical Oxygen Demand of the textile waste water. Percentage of color removed was highest in Sample 2 at pH 6 in 60 minutes of contact time and adsorbent dosage of 1ml.

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