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Degradation of Brilliant Blue Dye (FCF) in Wastewater Using Visible Light Induced Activity of α-Haematite Nanoparticle Synthesized via Green Route

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Abstract

This study investigated the photocatalytic activity of α-Fe_2O_3 nanoparticles (hematite) synthesized from the extract of *Senna siamea* flower and characterized using Transmission Electron Microscopy (TEM), Thermogravimetric Analysis / Differential Thermal Analysis (TGA/DTA), Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray (EDX) Analysis. The photocatalytic activity of the α-Fe_2O_3 nanoparticles was measured by degradation of Brilliant Blue Dye. The α-Fe_2O_3-S.S nanoparticles showed excellent photocatalytic performance, an optimum photocatalytic degradation 82% was obtained within 150 minutes intervals at loading catalyst dose of 150 mg at a concentration of 15 ppm. Thus, the catalyst demonstrated efficient degradation capacity of Brilliant Blue dye in wastewater.

Keywords: Hematite Nanoparticles; Green Synthesis; Senna Siamea Extract; Photocatalytic Degradation; Brilliant Blue Dye

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INTRODUCTION

Water pollution constitutes serious threat to the environment as well as human lives. Pollutant effects may vary depending on their types and source. Heavy metals, dyes, and some other organic pollutants are identified as major pollutants. Particularly, dye pollution in fresh water bodies emanate from effluents discharged from food, pharmaceutical, cosmetic and leather, pulp and paper industries (AttahDaniel et al., 2023; Adeogun *et al.,* 2016). Dyes are coloured substances that are chemically bonded to the substrates to which they are applied. This distinguishes dyes from pigments which do not chemically bind to the materials they colour. Dyes applied on textile materials are generally applied in an aqueous solution, and could require a mordant to improve the fastness of the dye on the fiber (Gerald *et al*., 2000). Globally, it is estimated that about 7×10^5 tons of 10,000 types of dyes are produced. While an estimated 2.8×10^5 tons of these dyes are released into freshwater bodies (Lin, 2015; Mohammed & Jaarrod, 2010; Zhang et al., 2017) without adequate treatment. The presence of dyes in water bodies impede the penetration of sunlight and hinders photosynthesis in aquatic plants and reduce dissolved oxygen, thus leading to the death of aquatic organism and other lives within the aquatic environment (Inyinbor *et al*.,2016). Dyes are basically classified as natural and synthetic, other classification and types of dyes are; acid dyes, basic dyes, direct (substantive) dyes, disperse dyes, sulphur dyes, azo dyes, pigment dyes, vat dyes, reactive dyes, naphthol dyes and premetallized dyes (Inyinbor *et al*.,2016).

Brilliant Blue FCF (Blue 1) is a synthetic organic dye primarily used primarily as a blue colourant for processed foods, medications, dietary supplements, and cosmetics (FD&C Blue 1 (Brilliant Blue), 2019). Brilliant blue dye consists essentially of disodium 3-[N-ethyl-N-[4-[[4-[N-ethyl-N-(3-sulfobenzyl) amino]phenyl](2-sulfophenyl)methylene]-2,5-cyclohexadiene-1-ylidene] ammoniomethyl] benzenesulfonate and its isomers together with subsidiary colouring matters, as well as sodium chloride and/or sodium sulfate as the principal uncolored components. It is synthesized by condensing 2-formylbenzenesulfonic acid with a mixture of 3-[(N-ethyl-N-phenylamino) methyl] benzene sulfonic acid and its 2- and 4- isomers to form the leuco base precursor. Oxidation of the leuco base precursor with either chromium or manganese containing compounds produces the dye, which is purified and isolated as the disodium salt FD&C Blue 1 (Brilliant Blue), 2017). Availability of potable water has become the most challenging task today for researchers and potable water providing professionals (AttahDaniel et al., 2022; Das *et al.,* 2015, Batnagar *et al.,*

2006). Hence, remediation of water is critically necessary in order to compensate for the depletion of potable water supply due to pollution of water bodies via industrial activities.

Many techniques have been employed to eliminate pollutants from wastewater. These techniques usually involve Methods such as biodegradation, oxidation, adsorption, and photo-degradation (Mohammed & Jaarrod, 2010) are used (Ayati et al., 2016; Mohamed *et al.*, 2016; Fallah Shojaei et al., 2018).

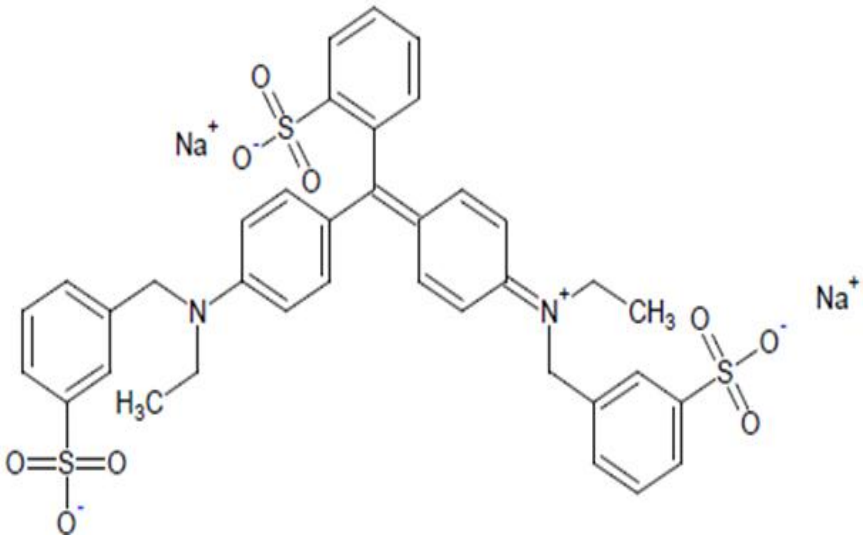

Figure 1: Molecular Structure of Brilliant Blue Dye (Joint FAO/WHO Expert Committee on Food Additives (JECFA), 2017).

Conventional methods of water treatment are either ineffective and/or expensive; hence, there is a clear need for the improvement and development of new materials and techniques for water treatment (Nyamukamba *et al.*, 2019; Gupta et al., 2012). Photolytic catalysis which is cost effective and time saving for removal of pollutant from wastewater, have been reported as a potential remediation technique for dye wastewater. Semiconductor photocatalysis have also been reported to possess the capacity to degrade a variety of dye pollutants in wastewater when UV-visible light or sunlight is deployed as a source of irradiation (Ansari et al., 2015; Ahmed et al., 2017; Taneji et al., 2018 Yerima et. al., 2024).

Nanotechnology, an emerging field that covers a wide range of strategies to development materials at nanoscale. Behavior of materials at the nanoscale as compared to macroscale are often found to be highly desirable properties which are created due to size confinement, the dominance of interfacial phenomena, and quantum effects. These novel properties of nanostructured materials have led to improved properties in catalysts, tunable

photo activity, increased strength and many other interesting characteristics (Mansoori *et al.*, 2008). This plays a major role in the development of innovative methods to produce new products, to substitute existing production equipment and to reformulate new materials and chemicals with improved performance resulting in less consumption of energy and materials and reduced harm to the environment as well as environmental remediation. Environmental applications of nanotechnology addresses the development of solutions to existing environmental problems, preventive measures for future problems resulting from the interactions of energy and materials with the environment, and any possible risks that may be posed by nanotechnology itself (Abdullah, 2016).

In material Chemistry, "green" synthesis have gained extensive attention as a reliable, sustainable, and eco-friendly protocol for synthesizing a wide of materials/ nanomaterial's including metal/metal oxides nanomaterials, hybrid materials, and bioinspired materials (Awwad et al., 2015; Kpega et al., 2023).. As such, green synthesis is regarded as an important tool to reduce the destructive effects associated with the traditional methods of synthesis for nanoparticles commonly utilized in laboratory and industry. The fundamental processes and mechanism of "green" synthesis approaches, especially for metal and metal oxide [e.g., gold (Au), silver (Ag), copper oxide (CuO), and Zinc oxide (ZnO)] nanoparticles using natural products (Singh *et al.*, 2018). For instance, ZnO nanoparticles have been employed as adsorbent due to their low toxicity and efficiency (Azizi et al., 2017). While the plant-based synthesized ZnO nanoparticles enhances metal ion adsorption capacity due to the chemical interactions between ions and functional groups of plant extracts. Previous studies have reported the use of Passiflora foetida extracts-ZnO nanoparticles to efficiently degrade both methylene blue (MB) and Rhodamine blue (RhB) dyes with nearly 93.25% and 91.06% efficiency in 70 minutes (Khan et al., 2021). Likewise, Congo red dye have been degraded successfully using zinc oxide nanoparticles. Green-synthesized metallic nano-particles can be produced by biological methods and provides an environmentally friendly way of synthesizing nano-particles devoid of harmful and toxic chemicals (Singh, 2016; Agarwal et al., 2018).

Senna siamea is an evergreen, medium-sized, many-branched tree legume. It reaches 10-12 m on average, rarely exceeding 20 m, and can exceptionally reach 30 m (Rojas-Sandoval et al., 2013). Its root system consists in few thick and deep roots and in a dense mat of rootlets that spreads rapidly (up to 7 m in one year) (Orwa et al., 2009). *Senna siamea* is easily uprooted by strong winds (Rojas-Sandoval *et al.*, 2013).

Figure 2. Plate I: *Sienna siamea* flowers (Natural Resources Conservation Service Plants Database, 2015)

Photocatalysis has considerable potential to contribute to the degradation of pollutants in aquatic environment and also in wastewater treatment, purification of wastewater using photocatalytic degradation is effective and cost effective compared to other conventional methods. The process involves a photo catalyst (a substance which is activated by adsorbing a photon) capable of accelerating a reaction without being consumed (Fox *et al.*, 1989).

Photo catalysts are mainly semiconductors such as (TiO_2, Fe_2O_3, and ZnO) which when under light exposure (UV light or sun light) degrade organic and inorganic contaminants (Kuriakose *et al.* 2015; Mohamed *et al.* 2017).

Semiconducting oxide photo catalysts have received tremendous attention from researchers in recent years due to their potential applications in solar energy conversion and environmental purification. Semi-heterogeneous photo catalyst have been used in the treatment of organic pollutant in water and in the air (advanced oxidation process (AOP) and are suitable for the oxidation of a wide range of organic compound. They are reported to be very potent and efficient in degrading recalcitrant organic compounds via the acceleration of photoreaction in the presence of semiconductor catalyst (Gaya *et al.*, 2018).

The field of nanochemistry has enormously enhanced the synthesis of semiconductor nanoparticles (Alivisatos, 1996). Semiconductor materials can be manufactured to a very high purity level. Recent advances have reduced impurity levels in the pure material to 1 part in 10 billion. The addition of one-part impurity (of the proper type) per million in a wager of silicon material can change that material from a relatively poor conductor to a good conductor of electric current. (Shree Krishna Khadka, 2018).The unique physical and chemical properties of semiconductor nanomaterials make them suitable for application in emerging technologies, such as Nano electronics, nanophotonics, energy conversion, non-linear optics, miniaturized sensors and imaging devices, detectors, photography (Manas *et al.*, 2019).

Haematite, iron (III) oxide (Fe2O3), widespread in rocks and soils (Cornell *et al.*, 1996) forms $\alpha-Fe_2O_3$ among others are of great importance in technological and industrial applications (Huo, 2000). Iron is found to be very reactive towards oxygen and water in normal air to give hydrated iron oxides (rust), unlike many other metals which form passivating oxide layers, iron oxides occupy more volume than the metal and thus flake off, exposing fresh surfaces for corrosion. Iron oxides and oxide hydroxides are widespread in nature, play a vital role in many geological and biological processes, and are extensively used by humans, e.g., as iron ores, pigments, catalysts, and in blood as hemoglobin. (Abdullah, 2016). In generation of hydrogen, α-Fe_2O_3 enhanced photocatalytic water splitting activity under UV radiation, which is two orders better than that reported for α-Fe_2O_3 powders (Karunakaran, 2006). Another photo catalytic oxidation activity of Fe_2O_3 is in the oxidation of aniline to azobenzene under natural sunlight and UV irradiation in protic and aprotic solvents (Vladimir *et al.*, 2015).

Green chemistry as an inter-disciplinary field is employed by chemists in designing new catalysts that reduces the amount of dyes contained in the textile industrial effluents using different metal based nanomaterials which have been described for the remediation of numerous contaminants, but the vast majority of studies have been dedicated to the removal of heavy metals and chlorinated organic pollutants of water. Metal and metal oxide nanomaterials are highly efficient adsorbent exhibiting advantage such as fast kinetics and high adsorption capacity, nanoparticles are commonly used for environmental remediation (Masciangoli *et al.,* 2003). Metal and metal oxides (such as; gold (Au), silver (Ag), copper oxide (CuO), and Zinc oxide (ZnO)] nanoparticles using natural products. (Singh *et al.*, 2018).In this study, nanoparticles (catalyst) of $\alpha-Fe_2O_3$ was synthesized via green route

using *Senna siamea* flower extract and used in the degradation of brilliant blue dye FCF (a anionic dye) in dye wastewater.

MATERIALS AND METHODS

All reagents used in this work were of analytical grade and used without any further purification: Iron Nitrate nanohydrate, $Fe_2(NO_3)_3.9H_2O$, (321.83g/mol, 99.0%, Sigma-Aldrich), Sodium Hydroxide (NaOH, Sigma – Aldrich, 40g) Hydrochloric acid (Sigma - Aldrich, 37% w/v) and Brilliant Blue (Kem Light Laboratories Pvt. Ltd., 696.68g). Characteristics and molecular structures of Brilliant Blue Dye are described and displayed in figure 1. All solutions were prepared by using double distilled water.

Sample Collection, Identification and Preparation

Senna Siamea flowers were collected in the morning within the Faculty of Physical Sciences, Ahmadu Bello University, Zaria. The flowers were identified at the Herbarium in the Department of Biological Sciences, Ahmadu Bello University, Zaria. The flowers were washed clean under running water from the tap and subsequently with distilled water. The washed lowers were dried at room temperature for 12 days, then ground to yield coarse powder. A portion weighed 5 g was boiled in 50 $cm^3$of double distilled water for 15 min. The aqueous extract was then cooled, filtered using Whatman No.1 filter paper and stored in a refrigerator at 4 °C for further use.

Green Synthesis of α-Haematite Nanoparticles

To 50 ml of the S.S extract in a conical flask, 5 g of Iron nitrate nonahydrate, ($Fe_2(NO_3)_3.9H_2O$ was added. The reaction mixture was evenly stirred for 3 while the temperature was maintained at 70 °C and centrifuged at 4000 rpm for 10 minutes, the solid product was collected and rinsed with distilled water. The α-Haematite nanoparticles were acquired by heating the mixture at 450 °C for 3 h in a hot air oven. The catalyst synthesized was coded as α-Fe_2O_3-S.S.

Characterization of Synthesized Nanoparticles

Transmission emission spectroscopy (TEM)

The internal structure of the synthesized catalyst was examined with the aid of a Nano Mill Transmission Emission Microscope operated at 20 kV. Approximately 0.02g of the sample was dissolved in 10 cm^3 of ethanol, followed by ultra-sonication for 5 minutes.

A drop of the solution was placed on a holey-carbon copper grid and air dried at room temperature and thereafter exposed to photo light for three (3) minutes. After drying, the copper grids with sample were mounted into the electron microscope for analysis.

Thermogravimetric analysis (TGA)

Thermal characteristic and energy profile of synthesized catalyst was investigated using a Perkin-Elmer TGA 400 instrument with heating rate of *10* $℃/min$ up to *950* ℃ in Nitrogen (N_2) atmosphere. Thermal Analysis detects the interatomic and inter-/intra-molecular interactions as related to an imposed external change in temperature. The nitrogen gas was set as the inert gas and oxygen gas as the oxidative. The gas environment was preselected for thermal decomposition (inert-nitrogen gas) and an oxidative decomposition (air or oxygen). The required flow rate was adjusted to provide the appropriate environment for the tests. The samples were then placed in the specimen holder and the temperature of the furnace was raised to*950* ℃. The initial weight was set to read *100 %* before the heating program was initiated.

Scanning electron microscopy (SEM)

A Scanning Electron Microscope (JEOL-JSM 7600F) (SEM) was used to determine the surface morphology of the sample. Minimal preparation includes the acquisition of the sample and placing it on a clean carbon strap that was attached to the sample holder and fitted into the SEM chamber, and some accommodation to prevent charge build-up and also prevent the sample from making contact with the detector.

Energy dispersive x-ray analysis (EDX)

Approximately 0.05 mg of the catalyst was sprinkled on a sample holder covered with carbon adhesive tape for 5 minutes prior to analysis. The sample was characterized using the JEOL-JSM 7600F Scanning Electron Microscope equipped with EDX. The secondary electron mode was activated for imaging and a homogeneous region on the sample was identified. The microscope was operated with electron high tension of 20 kV for EDX. The illumination angle was adjusted to 150º for the determination of the elemental composition of the sample.

Preparation of Reagents and Stock Solution

Preparation of brilliant blue dye stock solution

About 0.5 g of brilliant blue dye was dissolved in a 1000 cm^3 volumetric flask and made to the mark with deionized water to obtain 500 mg/L stock solution. Working concentrations of 5 mg/L, 10 mg/L and 15 mg/L were prepared separately from the stock solution.

Preparation of brilliant blue calibration standards

Calibration standards of 2, 4, 6, 8 and 10 mg/L concentrations were prepared from the stock solution. Their absorbance were measured at a wavelength of 561 nm using distilled water as blank and a calibration curve of absorbance against concentration was plotted which obeyed Beer lambert's law. An equation of straight line was generated in order to determine residual concentration of dye left for subsequent spectrophotometric analysis of the dye degradation.

Photocatalytic Degradation Experiment

Photocatalysis using batch process

The Photocatalytic activity of the synthesized catalyst was evaluated by using the catalyst to degrade brilliant blue dye in batch experiments in a continuous stir tank reactor comprising of a closed Pyrex reactor with an outer diameter of 42.0 mm and a height of 210 mm and thickness 4 mm. 50 cm^3 of 50 mg/L brilliant blue dye solution was added to a 100 cm^3 beaker on a magnetic stirrer. Then 50 mg of the synthesized catalyst (α-Fe_2O_3-S.S) was added to the reactor. The pH of the aqueous dye solution was kept at pH 7.3 during the reaction using a HANNA 4222 pH meter. The suspension was stirred by using a magnetic stirrer at 650 rpm at ambient temperature for 30 minutes in the dark, then 5 cm^3 of the suspension was withdrawn to analyze the equilibrium concentration of dye in the solution using a JENWAY 6705 Ultra violet-Visible Spectrophotometer. The mixture was then exposed to visible light irradiation. At selected time intervals of approximately 30 minutes over a typical 2 hours reaction time, 5 cm^3 of the suspension was withdrawn with a syringe and filtered using a 0.45 μm PTFE membrane syringe filter (Acrodisc CR13mm) and then taken for analysis using JENWAY 6705 Ultra violet-Visible Spectrophotometer. This procedure was repeated using varying concentration of the brilliant blue dye (10 mg/L and 15 mg/L) and varying catalyst dosage (100 mg and 150 mg) respectively.

The percentage of the brilliant blue dye degraded by the catalyst was calculated using the equation 1:

$$\%\, Degradation = \frac{C_o - C_e}{C_o} \times 100 \ldots\ldots\ldots\ldots\ldots\ldots (1)$$

Where: C_o is the initial concentration of the brilliant blue dye before degradation,C_e is the equilibrium concentration (final concentration) of the dye after degradation.

RESULTS AND DISCUSSION

Characterization of α-Fe_2O_3-S.S Nanoparticles

Transmission emission microscope analysis

Plate II (Figure 1a) shows the transition emission spectroscopy of α - Fe_2SO_3 /S.S. The Crystalline features of the catalyst α - Fe_2SO_3 /S.S NPs depicts its crystal structure and features which are in conformity to the rhombohedral lattice system as reported by Rao *et al.,* 1974.

Scanning electron microscope analysis

The Scanning electron micrograph of the prepared catalyst depicted by Plate III (Figure 1b) gives an appreciation of the porosity of the **α - Fe_2SO_3 /S.S** and hence a qualitative assessment of the ability to degrade brilliant blue dye in solution. The prepared sample has many pores in a lump like shape on the surface and are uniformly distributed.

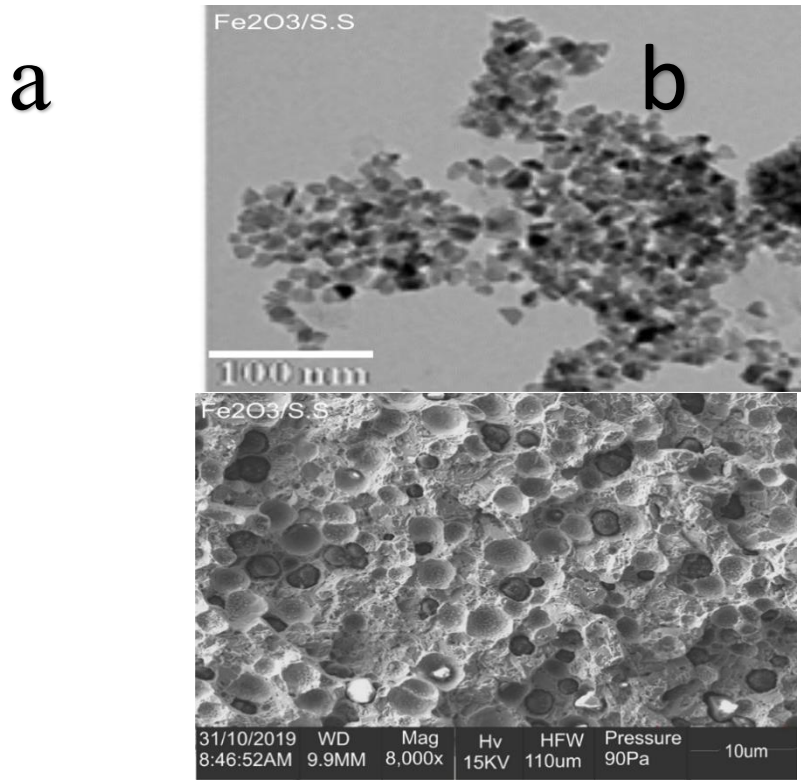

Figure 1 (a) Plate II: Transmission electron microscope of α-Fe_2O_3-S.S nanoparticles (100 nm); (b) Plate III: Scanning Electron Microscope image of α-Fe_2O_3-S.S nanoparticles (× *8 000*)

Thermogravimetric analysis-differential thermal analysis

Thermogravimetric Analysis (TGA) measures the mass of a sample as it is subjected to a selected temperature program in a defined atmosphere. The thermogram is shown in Figure 2a which shows the percentage weight loss of α - Fe_2SO_3 /S.S NPs. There was weight loss of 71.37% as the temperature increased from 25.5 ℃ to 290 ℃ due to loss of water, and combustible organics present in the catalyst. There was a weight loss of 26.63% as the temperature increased from 500 ℃ to 800 ℃.

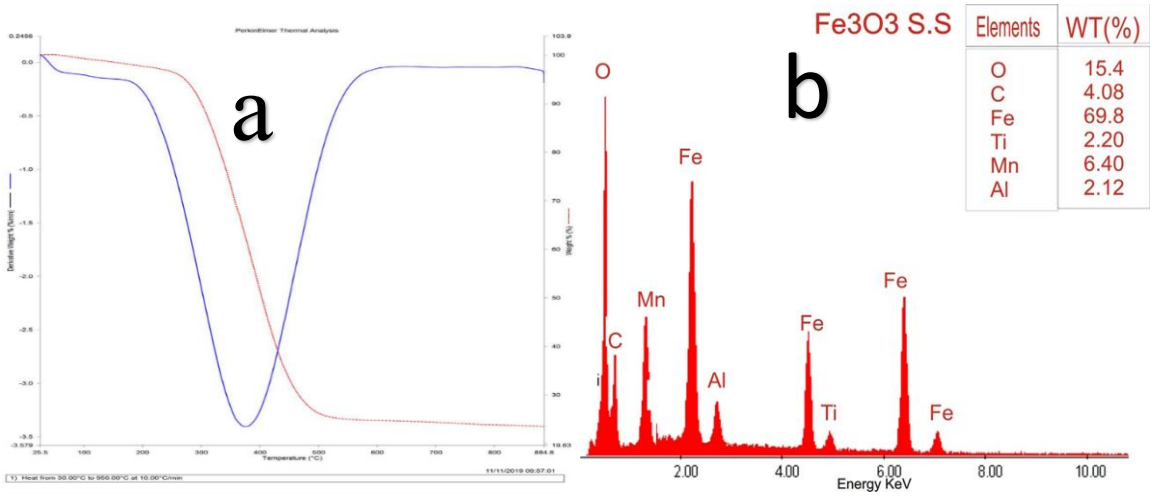

Figure 2: (a) TGA- DTA of α-Fe_2O_3-S.S nanoparticles (b) Elemental composition of α-Fe_2O_3-S.S nanoparticles (EDX)

Energy dispersive x-ray analysis (elemental composition)

The elemental composition of the green synthesized α - Fe_2SO_3 .S.S NPs catalyst was obtained via Energy Dispersed X-ray (EDX) measurement (Figure 2b). The peaks of the elements iron and oxygen show the respective percentage present (69.8% and 15.4%) in the catalyst. The sharp peaks observed in Figure 2b reveals the highly crystalline nature of the prepared α - Fe_2SO_3 -S.S NPs.

Photocatalytic Degradation of Brilliant under Visible Light Irradiation

The effect of the catalyst dosage on the degradation efficiency of the brilliant blue dye photocatalytic degradation was detected by using various amounts of the green synthesized **α** –Fe_2O_3 /SS NPs (50 mg, 100 mg and 150 mg) in aqueous 5 mg/L brilliant blue dye of 50 ml. Before irradiation the system was magnetically stirred for 30 minutes under dark to establish the adsorption-desorption equilibrium between the catalytic surface and the dye.

The pH of the 10 mg/L brilliant blue dye solution was kept at 7.3 at room temperature, the visible irradiation time interval ranged from 30 to 150 min. A maximum photocatalytic degradation efficiency (64%) was obtained with 100 mg catalyst dosage. The results obtained (figure 3a) shows that the degradation efficiency increases by increasing the catalyst dose up to 150mg. This is due to the increase in the number of active sites on the catalyst surface, causing an increase in the number of absorbed photons, which leads to

production of large number of •OH radicals, and increase degradation of the dye molecules. By increasing the catalyst dose greater than 100mg the solution become turbid, thus, decreasing the effectiveness of the catalyst activation during the visible irradiation (Saad *et al.,* 2015).

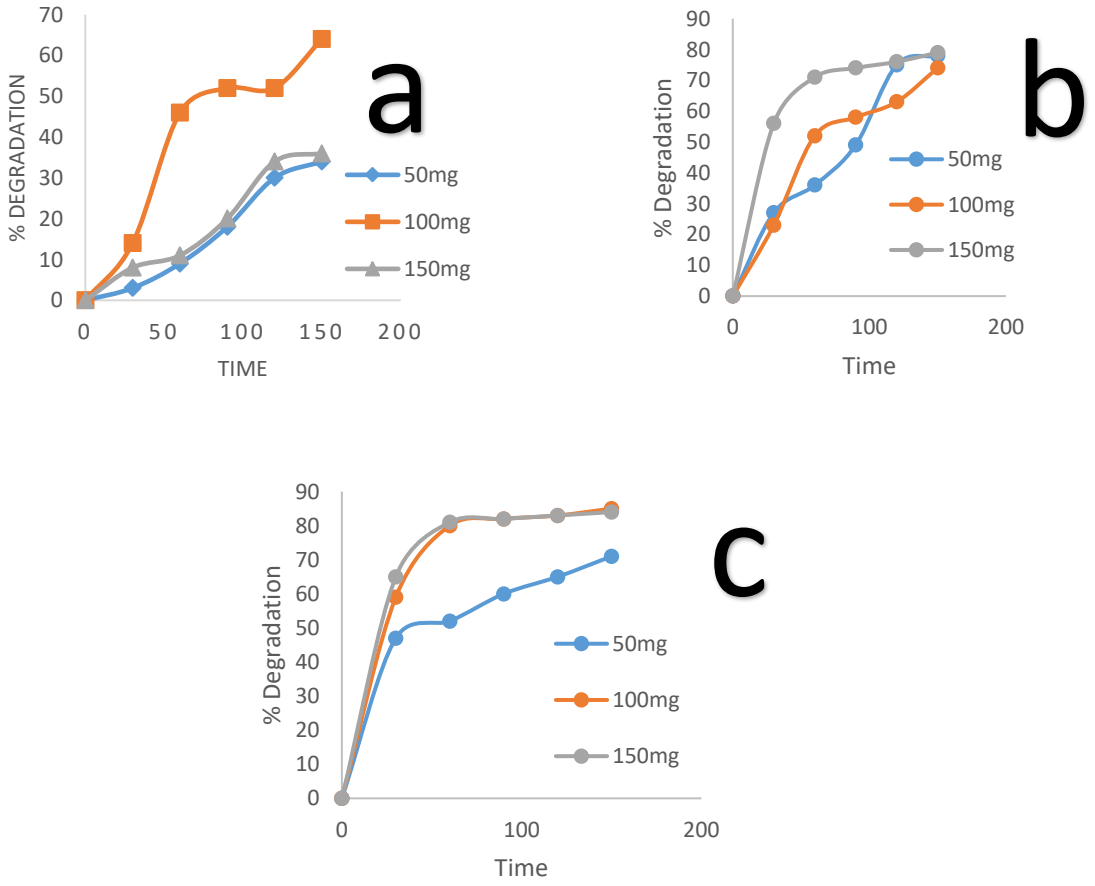

Figure 3b shows the effect of catalyst dosage on the degradation of brilliant blue dye (Concentration=10 mg/L, pH = 7.3).

Figure 3: (a) Effect of catalyst dosage on the degradation of brilliant blue (concentration= 5 mg/L, pH= 7.3) using visible irradiation (b) effect of catalyst dosage on the degradation of brilliant blue (concentration= 10 mg/L, pH= 7.3) (c) effect of catalyst dosage on the degradation of Brilliant Blue (concentration= 15 mg/L, pH= 7.3) using

A maximum photocatalytic degradation efficiency (78%) was obtained with 150 mg α –Fe_2O_3 /S.S catalyst dosage. The results obtained (figure 3b) shows that the degradation

efficiency also increases by increasing the photocatalyst dose up to 100mg. This is due to the increase in the number of active sites on the catalyst surface, causing an increase in the number of absorbed photons, which also leads to production of large number of •OH radicals, and increased degradation of the dye molecules (Saad *et al.*,2015).

Figure 3c show the effect of catalyst dosage on the degradation of brilliant blue dye (concentration=15mg/L, pH = 7.3). A maximum photocatalytic degradation efficiency (82%) was obtained with 150 mg **α** –Fe_2O_3 /S.S NPs catalyst dose at 150 minutes. The results obtained shows that the degradation efficiency also increased the catalyst dosage increased up to 150 mg. This was also due to the increase in the number of active sites on the catalyst surface that caused an increase in the number of absorbed photons, leading to production of large number of •OH radicals, and increased degradation of the dye molecules. (Saad *et al.*, 2015).

CONCLUSION

The study revealed that the green synthesized of **α**-Fe_2O_3 -S.S NPs from *Senna Siamea* plant flower is suitable for the removal of brilliant blue dye from contaminated or waste water. From the result of this study, the following conclusions drawn

The **α**-Fe_2O_3-S.S NPs exhibited good catalytic properties suitable for enhanced degradation of brilliant blue dye in contaminated water.

The degradation of brilliant blue dye increases proportionately with increase in catalyst dose. The optimum dosage for degradation of the brilliant blue dye contaminant was 150 mg with removal efficiency of 82 % respectively achieved in 150 minutes time interval.

Increase in the dosage of the catalyst above 100mg in the case of 5ppm Brilliant blue dye Solution leads to turgidity hence reduces the efficiency of degradation of the dye pollutant.

The maximum degradation of brilliant blue dye contaminant was obtained at a concentration of 15 mg/L with 150 mg dosage implying that at higher concentration and dosage more brilliant blue contaminated wastewater will be degraded.

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