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REMEDIATION OF WASTEWATER USING GRAPHENE OXIDE NANOPARTICLE

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ABSTRACT

Population and production increases have increased water use, which results in the rise in waste water quantity. Various methods have been used to remove the pollutants in water however, adsorption are found to be effective, low cost, eco-friendly and highly efficient. The present study aims to explore the approach of synthesis of Graphene Oxide nanoparticle using dry banana stem. The plant, *Musa paradisiaca* is used in this study, the phytochemical studies of the plant were carried out in four extracts finding the presence of Alkaloids, Carbohydrate, Protein, Aminoacid, Phenol, Tannin, Glycosides, Terpenoid, Steroids, Saponins, and Flavonoids and the nanoparticle was synthesized using the dried banana stem. The active phytochemicals present were responsible for this synthesis. The reduced Graphene oxide nanoparticle was characterized by UV – Vis Spectroscopy, FTIR and XRD. The laboratory waste water physico - chemical parameters were analysed. The laboratory wastewater was treated with Graphene Oxide Nanoparticle following the stirring procedure. The reduction of the contaminants and changes in the physico-chemical parameters of the laboratory waste water after treatment were noted. The treated water was checked for different parameters and was found to be very significant thus concluding that treated waste water can be used for recycling purpose. This plant material mediated synthesis of Graphene oxide can be applied extensively in the field of environmental remediation.

Key words : Green synthesis, Banana stem, Graphene Oxide, XRD, FTIR, Treatment.

INTRODUCTION

Nanoscience and nanotechnology primarily deal with the synthesis, characterization, exploration, and exploitation of nanomaterial's (**Paulchamy, Lignesh 2015)**. Nano-biotechnology, Bionanotechnology and Nanobiology is a branch of nanotechnology wherein the Nanomaterial are used for biological applications, for diagnosing disease, targeting drugs, imaging the disease condition and for remediation of water. They are widely used in cancer related studies also. This technical approach to biology allows the scientist to create new and effective system for biological research. The application of nanoparticle in biological field can lead to finding of useful Nano devices and Nano machines, Nano cages and protein based ink. Recently the use of plants and microorganism to synthesize the nanoparticle has been of a great interest, which has opened an alternate method to physical and chemical method of synthesizing nanoparticle. This method of nanoparticle synthesis causes attractive approach of green nanotechnology research towards sustainable development.

In the field of waste water treatment or purification, nanotechnology provides the possibility of effective removal of noxious pollutants and germs. In recent research, Nanoparticle, nanopowder and nanomembrane are used widely for the detection and removal of the chemical and biological substances. Basically four classes of nanoscale materials that are being evaluated as functional materials for water purification are metal -containing nanoparticle, carbonaceous nanomaterial, zeolites, nanotubes, nanofibres and dendrimers. All these show good result in water treatment because of their high surface area (**Dhermendra et al. 2008).**

Graphene Oxide Nanoparticle and its properties

Graphene is a carbonaceous material that appears in repeating patterns of hexagons. The speciality of Graphene is its sp^2 hybridization and very low atomic thickness. Graphene oxide is an oxidized form of graphene, whose surface is laced with oxygen containing groups. Graphene oxide is the by-product of oxidation process as when the oxidizing agents react with graphite, results in increased inter planner spacing between the layers of graphite. This oxidized compound can then be dispersed in a base solution such as water, and graphene oxide is then produced. Since they can be easily dispersed in water and in other solvents they are easy for processes and can be easily mixed with any polymer to enhance the properties of tensile strength, elasticity, conductivity and more **(Long Jiao et al. 2016).** Graphene oxide in solid form can attach to one another to form stable flat structures which can be folded, wrinkled and stretched. They are widely used for applications like nanofiltration membranes.

Need for Green Synthesis

Synthesis of nanoparticle using physical and chemical route leads to production of large amount of by-products such as high radiations, noxious gases, and highly toxic reductants etc. which are harmful to mother earth, plant life, animals and humans. This "Green synthesis" route of nanoparticle synthesis is mainly aimed to reduce and avoid the production of unwanted or harmful by products through sustainable eco-friendly synthesis procedures. Most of the metal oxide nanoparticles are produced by utilizing plant extract which enables to produce at a large scale. Synthesis using the plant material has led to simple, efficient, cost effective and feasible methods to conventional method of synthesis. Green method occurs in single step and relatively requires low energy. Various plants have been used to reduce and stabilize into nanoparticle in one step synthesis. Plants have various biomolecules like carbohydrates, proteins, and enzymes that help in enhancing its properties and aims in synthesis of controlled materials. Nanoparticle that is derived from the biological component is likely to be applied extensively in the field of environmental remediation and also in fields like pharmaceutical, food, cosmetic industry etc.

Application of Banana stem

The banana stem is used both as a food and a non-food related applications like pulp and paper raw material, fillers or structural reinforcement in composite materials, livestock feed, fibres for textile, bioactive compound source and organic fertilizers **(Birdie Scott Padam et al. 2014)**. Banana stem extract have also been used as the natural coagulant for the treatment of water since ancient times. Population and production increases have increased water use, which results in the rise in waste water quantity. Due to development in the field of science the numbers of research laboratories across the globe have raised, the effluent produced from these laboratories includes both organic and inorganic wastes such as organic solvents, toxic dyes, organic chlorine compounds, heavy metal ions, microorganisms, poisonous reagents, plant waste and animal waste. If this water is directly discharged into the sewers without any special treatment there are possible ways to contaminate the water bodies. The amount of water used in the laboratory is relatively high and if treated and recycled they can be reused for garden purposes and others. Thus there is an urge to remove these materials from aqueous solutions. Various methods have been used to remove these organic and inorganic pollutants however adsorption are found to be effective, low cost, eco-friendly and highly efficient. The pollutants are adsorbed on to the particle due to physical or chemical force which mainly depends on the factors like temperature, solution pH, concentration of the pollutant, contact time, particle size, etc. Due to their high surface area and smaller size, nanoparticles have stronger adsorption capacity and reactivity. Benefits of treating water using Graphene Oxide nanoparticle includes reduction in total solids, softening of hard water, pH becoming less aggressive, reduction of colour and turbidity.

MATERIALS AND METHOD

Collection of Water samples from different Science Laboratories

Water samples were collected from 5 different Laboratories [laboratory wash water (from the Departments of Advanced Zoology and Biotechnology, Plant Biology and Plant Biotechnology, Home Science, Chemistry and PG Biotechnology) of Women's Christian College. 50 ml of water from each laboratory was mixed together, made as one wastewater and was used for the analysis

PLANT: Banana stem (matured) [dry]

Dry stem

10 gram of finely powdered Banana stem was taken and was soaked overnight in 100ml of distilled water, chloroform, ethyl acetate and ethyl alcohol. It was filtered using Whatman filter paper. The filtered extract was used for qualitative and quantitative analysis

Synthesis of Graphene Oxide nanoparticle

Dry Banana stem was grounded to obtain fine powder. To 0.5g of the banana stem powder, 0.1g of Ferrocene was added and was directly put into a muffle furnace at 300˚C for 10 minutes under atmospheric condition. Black solid product obtained was further crushed and 5g of this black solid powder was added to 50ml of distilled water and was sonicated for 5 minutes and then centrifuged at 5000 rpm for 5 minutes. The supernatant was used for the experiment and characterisation analysis. This supernatant was made into Graphene Oxide solution **(Thirunavukkarasu Somanathan et al. 2015).**

Treatment of waste water

400ml of the laboratory waste water was filtered to remove coarse solid particles in water. The water was taken in a glass beaker and a magnetic bead was placed on the inside of the beaker with water and placed in the magnetic stirred and was stirred continuous five hours at room temperature. 1ml of nanoparticle was added after every 30 minutes till 5 hours. After 5 hours the water was removed from the stirrer and kept at room temperature for 24 hours. The water was kept for 5 days and each day 0.1ml of Graphene Oxide nanoparticle was added and stirred and each day the amount of waste settled at the bottom was noted. The contaminants which settled down at the bottom was filtered using a paper and the water was used for the further analysis (**Thema et al. 2012**).

RESULT AND DISCUSSION

Collection of the Sample

The sample for the Green synthesis of Graphene Oxide and for phytochemical analysis were taken from the same site in Chennai **(Figure 1).**

Figure 1: Collection of *Musa. sp* (Banana stem)

Sample preparation and processing

The Banana stem were cleaned with water to remove dirt, cut into small pieces and kept for
Sun drying for 7 days after which it was ground to prepare fine powder (**Figure 2).**

Figure 2 : Processing of matured Banana

Qualitative Phytochemical Analysis of dry Banana stem powder

Sample preparation

The Powdered Banana stem was soaked in different aqueous and solvents to obtain the extracts for performing the qualitative and quantitative phytochemical analysis (**Figure 3)**

Figure 3: Water, ethyl acetate, chloroform, ethanol extraction of Matured Banana stem

Qualitative Phytochemical screening of Banana stem

(Table 1) shows the presence of Alkaloid, Flavonoids and Proteins in all the extracts. Tannins and Phenol showed positive results only in aqueous and chloroform extract. Carbohydrate and glycoside showed positive results only in ethanol and chloroform extract. Terpenoids and steroids showed positive results only in ethyl acetate extract. Saponins showed positive result only in aqueous extract and Aminoacid showed positive result only in ethanol extract.

PHYTOCHEMICALS	NAME OF THE TEST	Aqueous	Ethyl acetate	Ethanol	Chloroform
		Dry powder sample			
Alkaloid	Dagendorff	+	+	+	+
Carbohydrate	Fehling's/benedict's	-	-	+	+
Protein and amino acid	Biuret/ ninhydrin	+,-	+,-	+,+	+,-
Flavonoid	Lead acetate test	+	+	+	+
Tannins	Ferric chloride test	+	-	-	+
Phenols	Ferric chloride test	+	-	-	+
Terpenoids	Salkowshki's test	-	+	-	-
Saponins	Foam formation	+	-	-	-
Steroids	Conc. Sulphuric acid test	-	+	-	-
Glycosides	Keller-Kiliani test	-	-	+	+

Table 1: Phytochemical analysis of aqueous and solvent extraction Banana stem

Quantitative Phytochemical Analysis

Quantification of each phytochemicals was carried out. On quantification of these phytochemicals, Carbohydrates was found to be very high. Hence these bioactive compounds would be responsible for the enhancement of the synthesis of Nanoparticle **(Table 2).**

Table 2: Phytochemical Estimation

S. No	PHYTOCHEMICAL	ESTIMATION (concentration mg)
1	ALKALOID	8.13±2.2
2	PROTEIN	11.47±0.4
3	AMINOACID	7.13±1.1
4	PHENOL	0.27±0.0
5	TANNIN	0.74±0.4
6	CARBOHYDRATE	15.43±5.5
7	GLYCOSIDE	10.74±1.2
8	TERPENOID	0.53%
9	STEROIDS	4.20±1.5
10	SAPONINS	10.33±1.2
11	FLAVONOID	2.63±1.1

Green Synthesis of Graphene Oxide Nanaoparticle

The synthesis of Nanoparticle by Green method is by chemical oxidation process at very high temperature of 300˚C and involves the use of Dry Banana stem powder to which ferrocene was added as the precursor. This ferrocene will be thermally decomposed at high temperature breaking down as iron atoms and hydrocarbon molecular fragments. These iron atoms will coalesce with the active compounds in the banana stem producing the nanoparticle (**Figure 4).**

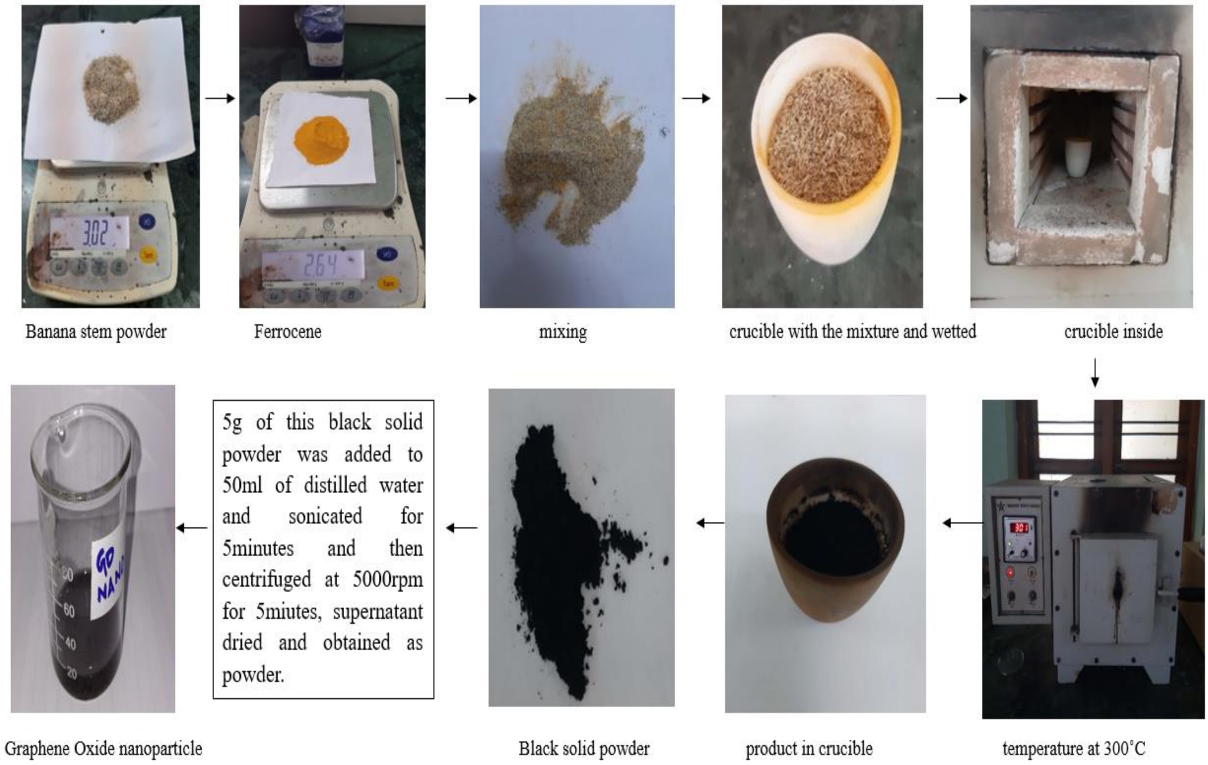


Figure 4 : Synthesis of Graphene Oxide Nanoparticle

UV- Visible spectroscopy

UV- VIS spec characterization of Graphene Oxide Nanoparticle was done using Systronics Double Beam UV-VIS spectrophotometer: 2202. This characterization was done to find the absorption range of Graphene Oxide Nanoparticle. Graphene Oxide absorption spectrum peak was observed at 230nm (**Figure 5)**, this absorption is attributed to aromatic C-C ring transition, which is in accordance to the result obtained by **[6].**

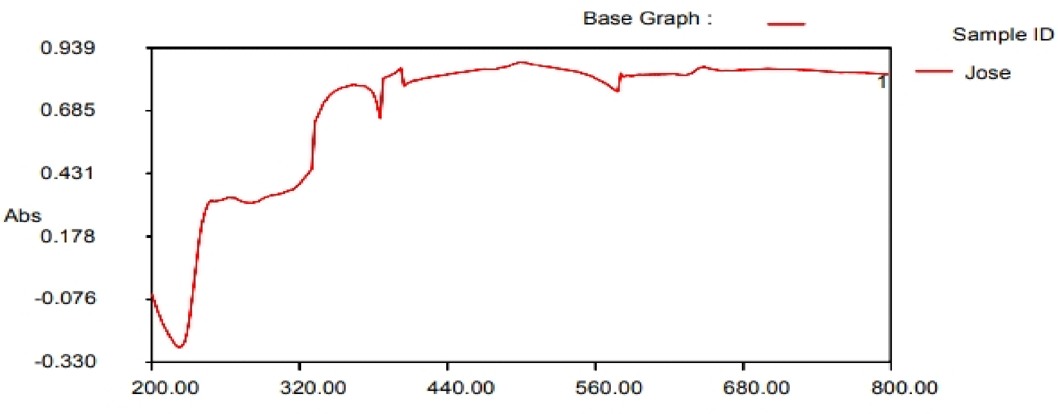


Figure 5 : UV- VIS spec characterization of Graphene Oxide Nanoparticle

Fourier – Transform Infrared spectroscopy

FTIR characterization is done for a nanoparticle to obtain absorption and emission in infrared spectrum of solid, liquid or gaseous compounds. The intensity of the peak in the graph is directly correlated to the amount of sample present, it also gives the details of the bonding configuration in the synthesized Graphene Oxide. FTIR Alpha, Burker, Germany were used for the sample analysis. A strong peak ranging from 3225 cm^{-1} to 3600 cm^{-1} was observed showing the presence of OH group indicating the absorption of water, which gives Graphene Oxide the property to disperse in water and other solvents. The presence of peak stretch from v1005

cm^{-1} to $1300cm^{-1}$ contributes to C-O bond, indicating the oxide functional group which confirms the Graphene Oxide oxidation process (**Figure 6).**

X-Ray Diffraction Measurements

The X-ray diffraction (XRD) is generally used for the analysis of the crystalline material. It also tells about the orientation of the single crystal, measures the spacing between atoms, rows and layers. Powder XRD Diffraction of Graphene Oxide was performed in Bruker, D2 phaser, Germany. Strong peak at 12.3° corresponds to the interlayer spacing about 0.71nm which indicates the presence of oxygen functional group **(Figure 7).**

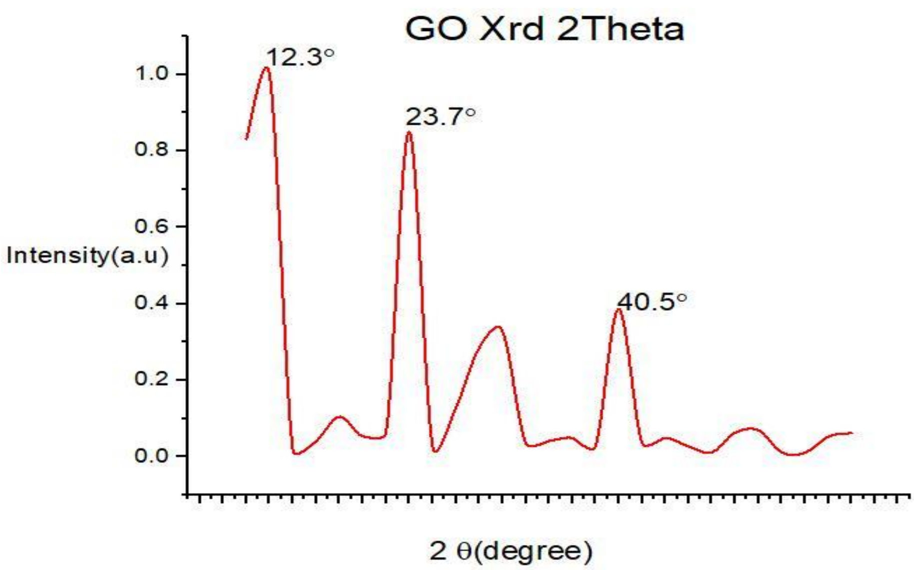
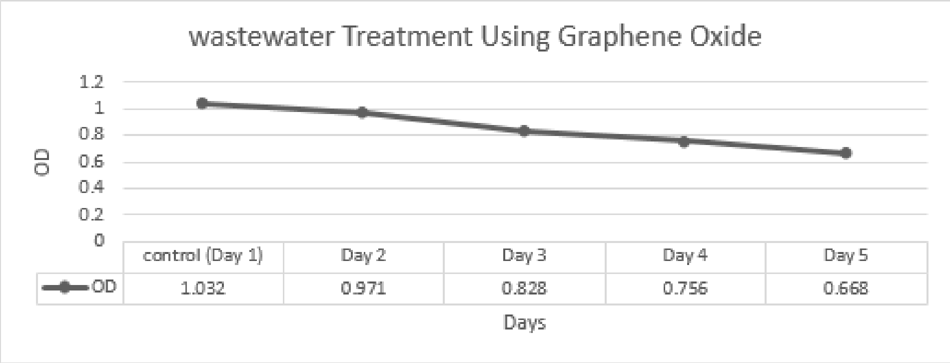


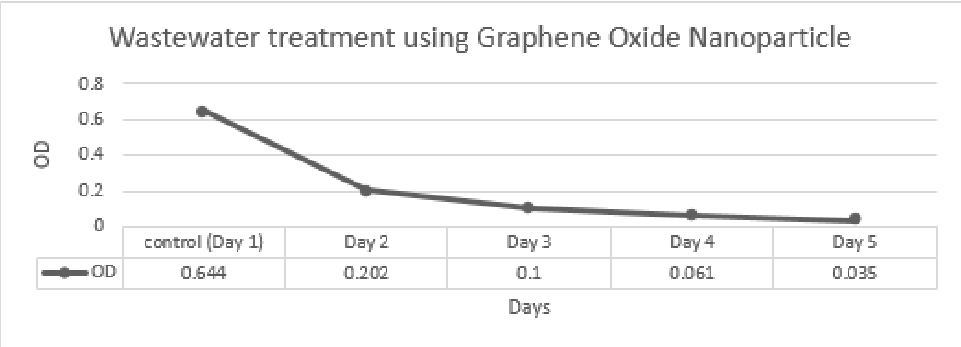
Figure 7 : X-Ray diffraction of Graphene Oxide

Treatment of Laboratory Waste water

On treating the laboratory waste water with Graphene Oxide Nanoparticle, the quality of the water found a drastic change from Day 1 to Day 5. The nanoparticle has magnetic, adsorbing ability that adsorbs heavy metals, ions, solids on to it because of high surface are on nanoscale. BOD, COD, Solids were reduced and remained within standard limits. The change in colour of the water after treatment was visibly noted. However on changing the pH of the water by adding sodium hydroxide pellets the water appears very clear, but the present work aims at treating the wastewater with Graphene oxide nanoparticle without altering the pH. OD was taken using control (wastewater before treatment) as standard **(Graphs 1– 2).**



Graph. 1 Reduction of contaminants in wastewater against wastewater



Graph. 2 Reduction of contaminants in wastewater against distilled water

Physico- chemical characteristics of Wastewater Before and After treatment

The Physico-Chemical parameters of the waste water samples were determined before and after treatment following the standard examination of water, given standard discharge limits as given in **[7]; [8](Table 3).**

Physico-chemical	Before treatment	After treatment	Standard limit
Colour	Brownish grey turbid	Clear light straw coloured	-
pH	2	6-7	6.5-9
Acidity	50mg/L	0.6mg/L	-
Alkalinity	Nil	Nil	-
Hardness	500mg/L	240mg/L	>180 maximum hard
TSS	0.032mg	0.02mg	50
TDS	10.08mg/L	0.8mg/L	
TS	10.832	0.82	
DO	160mg/L	48mg/L	20
BOD	1300mg/l	28mg/L	30
COD	1.7mg/L	0.64mg/L	200

Table 3: Physico-chemical and biological parameters before and after treatment

Serial Dilution

Serial dilution of the water sample will reduce the dense population of the microbial cells in the water enabling to isolate and study the number of organism in that water. In the serial dilution setup, before the treatment the serially diluted water concentration 10^{-4} and 10^{-6} on plating was observed to be 26 and 11 colonies and after the treatment with nanoparticle concentration 10^{-4} and 10^{-6} on plating was observed to be 7 and 2 (**Figures 8 – 10**).

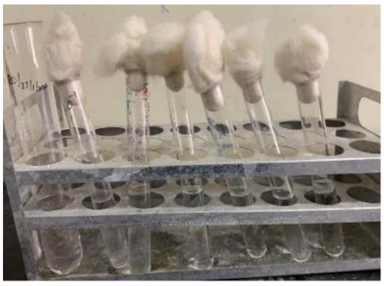


Figure 8 : Serial dilution

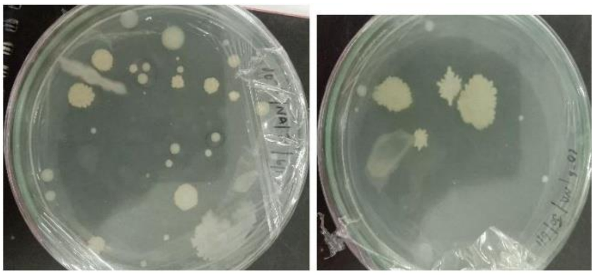


Figure 9 : Colonies found in pre treated

Figure: 10 Colonies found in post treated

CONCLUSION

Banana stem *Musa. spp* was collected processed and prepared for phytochemical analysis and Graphene Oxide synthesis. The banana stem was prepared for phytochemical analysis using different extracts like aqueous, ethyl acetate, chloroform and ethanol. The preliminary qualitative analysis of Dry Banana stem was carried out in all the extracts finding the presence of Alkaloids, Carbohydrate, Protein, Aminoacid, Phenol, Tannin, Glycosides, Terpenoid, Steroids, Saponins, and Flavonoids. The highest amount of Carbohydrate was obtained by each phytochemical estimation. Synthesis of Graphene Oxide Nanoparticle and preparation of Graphene Oxide solution was carried out. Characterization of the synthesized nanoparticle was done using UV-VIS spectroscopy, Infra-Red absorption by FTIR, Powder XRD Diffraction to confirm the presence of Graphene Oxide. The laboratory waste water Physico- chemical parameters were analysed and they were done again after the treatment of wastewater with Graphene Oxide Nanoparticle. The laboratory wastewater was treated with Graphene Oxide Nanoparticle following the stirring procedure and was kept in observation for 5 days and each day OD reading was noted. The reduction of the contaminants and changes in the Physico-chemical parameters were noted.

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