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Utilization of Novel Plant Bio-Sorbents for the Effective Treatment of Textile Dye Effluents

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Abstract

With the emerging inflation in the production of fabrics with the use of automated machines there is also an increase in the production of textile wastewater from these textile plants. Consequently, the increase in environmental pollution created due to discharge of these untreated effluents is inevitable. On the other hand, use of plant source as a biosorbent may provide a solution to the existing textile dye treatment problems. The main aim of study is to utilize three flower extracts namely *Nelumbo nucifera*, *Nymphaea nouchali* and *Musa paradisiaca* as potent biosorbent for the effective treatment of textile dye waste water. The process includes identification, selection and characterization of different plant sources for the use of biosorbent samples. Three different anaerobic sequential batch reactors are used for the study to identify the most effective biosorbent samples. The resulting treated effluent from the bioreactor is characterized and compared with the characterization result of the untreated textile dye wastewater. CETP standards are taken as the reference value and a data analysis is plotted between the standard with the treated and untreated effluents to ensure that the treated effluent follows the regulatory parameters of the Pollution Control Board of India. Among these flower extracts, *Nymphaea nouchali* is found to be more effective than other two flower extracts. This can be evident that development of such waste water plant to treat the effluent from textile industry can lead to sustainable protection over environment.

Keywords

Bio-sorbents, Phyto-remediation, Common Effluents Treatment Plant, Aerobic Sequential Batch Reactors, Textile waste.

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INTRODUCTION:

In the present-day Industrial sectors, among all the other industries present, the textile industry is considered the most polluting due to the amount of waste water produced in terms of volume and composition [1]. Effluent released from the textile industry consists of higher concentration of chemicals in form of organic and inorganic, which are further peculiarized by total dissolved solids (TDS), low dissolved oxygen (DO), chemical oxygen demand (COD), biological oxygen demand (BOD), total suspended solids (TSS) and strong colour of the

effluent [2]. The higher salt content or TDS presence in the water causes problem during the treatment of biological waste water plant, change in taste, contamination of ground and surface water bodies, causes toxicity towards aquatic organisms, quality of irrigation is disturbed due to changes in salt contents which causes changes in sodium proportion towards the other ions. These characteristics makes it necessary for the treatment of effluent from textile industry before discharging directly into the natural water bodies [3]. Present methods applied in treatment of effluents such as Biological decolonization, coagulation and flocculation, ion exchange, oxidation and adsorption [4]. Recently biosorption became hot field of research for treatment of effluent by applying nature's way of remediation of contaminants in soil i.e. is phytoremediation, this approach comes as eco-friendly as well as cost-effective. Several plant species like water lilies [5], water hyacinth [6], water mint, parrot feather, plantain plant [7] etc. have been studied to establish their capability in bio absorption of heavy metals and other toxic pollutants. Development of such system using aquatic plants for treatment of effluents from textile industry which requires lesser energy, easily regenerated and comes as natural system can lead to sustainable better environment.

So, the main aim of the study was to identify a cost effective yet eco-friendly method for the treatment of textile effluents using bio-sorbents. The objective of the study includes design, construct, evaluate and process parameter analysis of a bio reactor consisting of combined phyto-components of different plant species for the effective treatment of textile effluents.

MATERIALS AND METHODS

Collection of Textile dye Industry Effluents

The textile dye industry effluents were collected from a small-scale textile dye industry located at Vellore in Tamilnadu, India. The effluent wastewater was stored at 4°C in a freezer. Then the effluents were characterized in the laboratory for the parameters such as color, chemical oxygen demand (COD), pH, biological oxygen demand (BOD), total suspended solids (TSS), total dissolved solids (TDS) and Heavy metal analysis as per Standard Methods of Analysis, APHA (1992).

Preparation of sorbents

Plant samples were collected from various places around the district of Chennai, Tamilnadu. The collected flowers were tightly packed with Polyethylene bag and then transfer to the laboratory. Then it was washed with distilled water twice followed by rinsing in Sodium hypochlorite solution and kept under room temperature for two weeks in dark condition for complete moisture removal and drying purposes. Then the dried flower samples were made into powder using a homogenizer.

Preparation of plant extracts

The powdered plant samples were weighed 5g and dissolved in 100ml of distilled water and made into homogenized solution by continuous magnetic stirring for 8 hours. The extracts were filtered by Whatmann No1 filter Paper. Then the filtrates were stored in a tight seal pack under 4°C for further use.

PHYTO CHEMICAL ANALYSIS

Qualitative Analysis

Alkaloids:

a. 10ml of extract was taken from the given sample. In that, 8ml picric acid was added. The formation of orange color was observed.

b. And then the appearance of dark orange or purple color was observed. This color change indicates the presence of alkaloids.

Flavonoids:

a. From the given sample 2ml of extract was taken and mixed with few drops of 20% NaOH. Formation of intense yellow color was observed.

b. To that yellow color mixture few drops of 70% diluted HCL was added. The disappearance of yellow color was observed. The formation and disappearance of yellow color indicate the presence of flavonoids.

Glycosides:

a. 1ml of given sample was taken. In that 0.5ml of glacial acidic acid and 1% aqueous ferric chloride was added.

b. The formation of brownish ring was observed. This indicates the presence of glycosides.

Phenolic Compounds:

a. 2ml of extract was taken. In that 2ml of 5% ferric chloride was added.

b. The formation of Blue color was observed. This indicates the presence of phenolic compound.

Saponins:

a. From the given sample 2ml extract was taken and 5ml of distilled water was added, which is need to be vigorously mixed.

b. The formation of bubbles and persistent form foam indicates the presence of saponins.

Tannins:

a. 2ml of extract was taken. In that 10% of Alcoholic ferric chloride was added.

b. The formation of brownish blue or black color was observed. The color change indicates the presence of tannin.

Terpenoids:

a. From that given sample 1ml of extract was taken. In that 0.5ml chloroform followed by few drops of con. H_2SO_4 was added.

b. The formation of reddish-brown color was observed. This indicates the presence of terpenoids.

Quantitative Analysis:

Estimation of phenolic Compounds:

200µl of plant extract samples were taken (sample concentration mg/ml) and 800µl of Folin Ciocaltea reagent was added (1:1 dilution). Then 2ml of 7.5% sodium carbonate was added. Finally, the mixture was incubated for 4 hours in the dark place. Gallic acid was used as a standard in varying concentrations. The absorbance reading of the samples and the standard were taken at 765 nm and a standard curve was plotted using the reading from the Gallic acid standard. The slope value was calculated. From that value, total phenolic content present in the sample was calculated [8].

DPPH radical scavenging assays

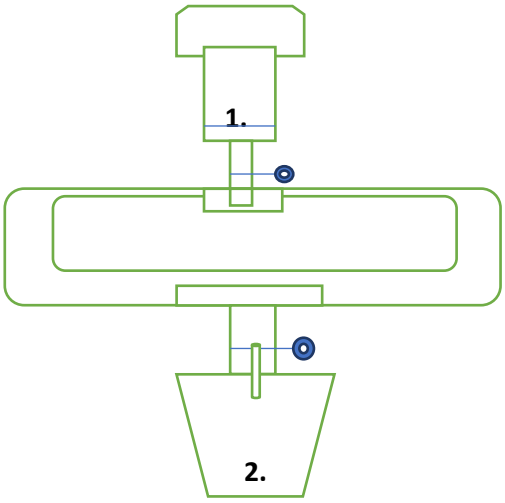
The DPPH assay, as previously reported by [9], was employed to determine the radical scavenging activity of the extracts. Aliquots of extract dissolved in dimethyl sulfoxide (DMSO) were plated out in triplicate in a 96-well microtiter plate. The methanolic DPPH (50 µM) solution (Aldrich) was added to alternating columns of the test samples and methanol was used for control of test samples, in the remaining columns. The plate was shaken for 2 minutes and incubated for 20 minutes in darkness at 37˚C, in a water bath. The percentage of decolourisation was obtained spectrophotometrically at 517 nm. The percentage of decolourisation was plotted against the concentration of the sample. Gallic acid was used as antioxidant standard. At least three independent tests were performed for each sample. The DPPH absorbance decreases with an increase in DPPH radical scavenging activity. Results were expressed as % inhibition of the DPPH radical which is obtained with the equation:

$$\text{DPPH radical scavenging activity (\%)} = \frac{ABS_{Control} - ABS_{Sample}}{ABS_{Control}} \times 100$$

Where $ABS_{control}$ is the absorbance of DPPH radical + methanol and ABS_{sample} is absorbance DPPH radical + sample extract/standard.

Aerobic Sequential batch reactor (ASBR)

Three ASBRs are used for conducting the research work. The reactors are identical in all aspects. The descriptive schematic of one of the three reactors is shown in Fig 1. For treatment with plant materials individually, the acrylic tank alone is used. The capacity of the Acrylic Tank is 5 litres. The sample that is to be treated is allowed to enter the Acrylic tank through a valve. The flow rate of the sample (effluent) is 5ml / min. The plant materials are subjected to the treatment of about 36 hours each.



1. Sample Holding Chamber 2. Reactor Chamber

Figure 1 Diagram of Aerobic sequential batch reactor

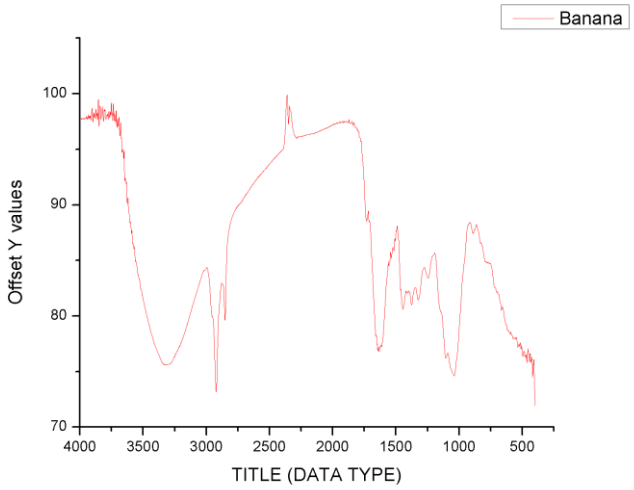


Figure 2 FTIR graph of *Musa paradisiaca* flower

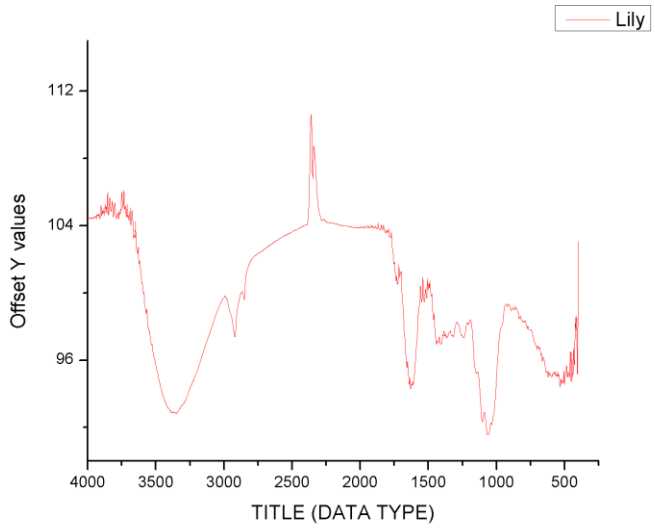


Figure 3 FTIR graph of *Nymphaea Nouchali* flower

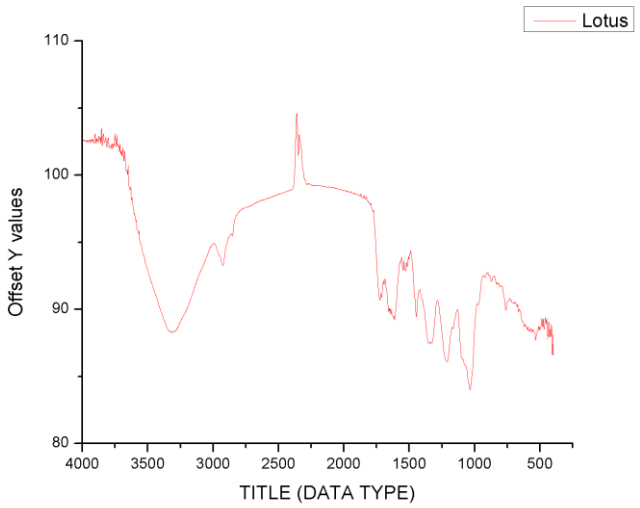


Figure 4 FTIR graph of *Nelumbo Nucifera* flower

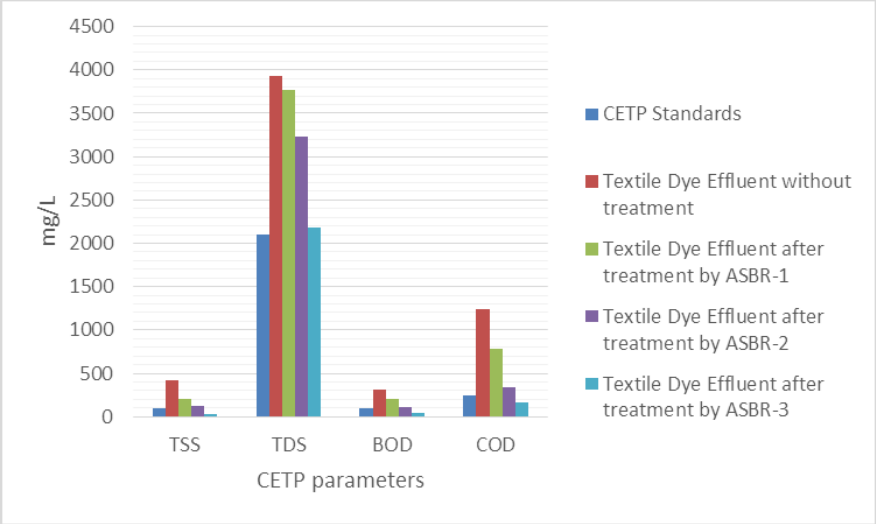


Figure 5 Comparison of CETP standard with effluents treated by ASBR's results-Graph 1

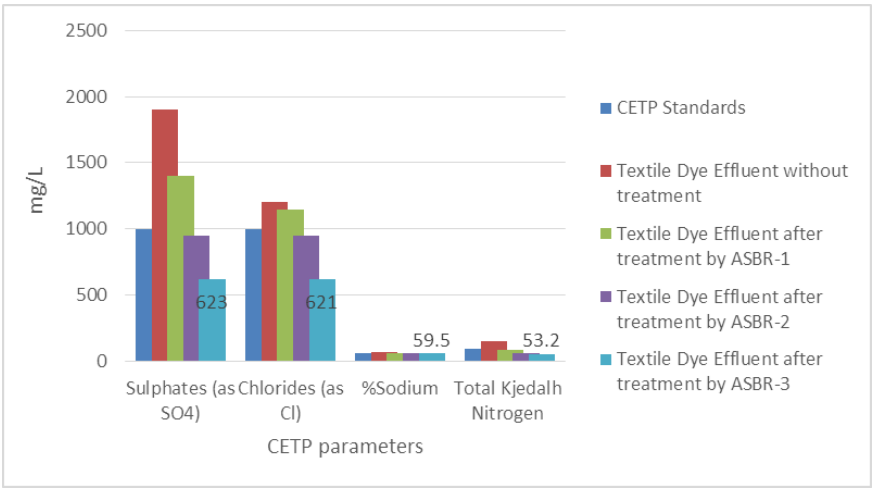


Figure 6 Comparison of CETP standard with effluents treated by ASBR's results-Graph 2

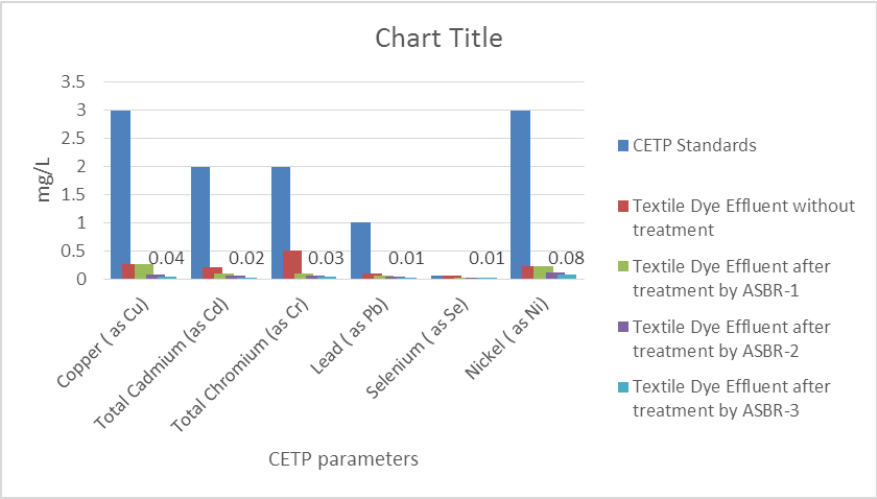


Figure 7 Comparison of CETP standard with effluents treated by ASBR's results-Graph 3

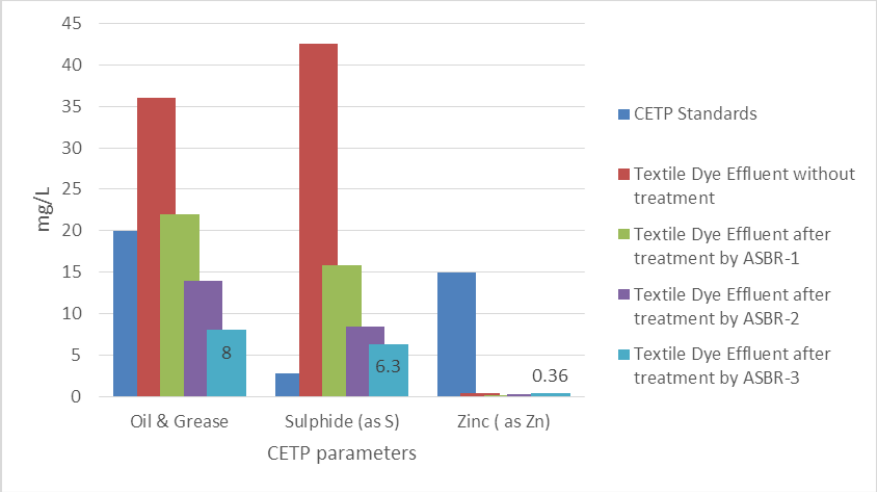


Figure 8 Comparison of CETP standard with effluents treated by ASBR's results-Graph 4

Evaluation of the various constituents of the Treated Textile Effluent by ASBRs.

The Treated Textile effluent samples from all the three ASBR were taken up for analysis of different physiochemical parameters [10, 11, 12].

Estimation of TDS

The samples were filtered and the sediments leftover on the filter were scraped off and dried in an oven. Then the dry weight of the sediments was measured.

Estimation of TSS

TSS of effluent is determined by pouring one litre of effluent through a pre-weighed Whatmann filter paper of a specified pore size, then weighing the filter again after drying to remove all water.

COD

2.5 ml of the sample was taken in the tube, 1.5 ml of 0.25 N $K_2Cr_2O_7$ (potassium dichromate), one spatula of mercuric sulphate $HgSO_4$ and 3.5 ml of COD acid were added and kept in COD reactor for 2 hrs at 150°c. After cooling the sample, it was titrated against FAS (standard ferrous ammonium sulphate 0.1N) ferroin is used as an indicator. The endpoint is reddish brown colour. In the blank tube, 2.5 ml of distilled water was taken and then follow the same procedure in the sample. Finally, below calculation is used for COD analysis:

$$\text{COD} = \frac{(\text{Blank value-titrated value}) \times \text{N of FAS} \times 8000}{\text{Volume of sample}}$$

The percentage COD reduction is calculated by

$$\%\ \text{COD Reduction} = \frac{\text{COD Initial-COD Final}}{\text{COD Initial}} \times 100$$

BOD

Take 5 litres of distilled water and aerate for 3.5 hours. Later added 1 ml of nutrient which contains FeCl, CaC_{12}, PO_4, $MgSO_4$, domestic water to 1 litre aerated distilled water. The solution was further aerated for 30 minutes. BOD was calculated from the formula:

$$\text{BOD} = \frac{(\text{Blank value-titrated value}) \times 300}{\text{Volume of sample}}$$

The percentage colour removal is calculated by

$$\%\ \text{Decolorization} = \frac{\text{Initial OD-Final OD}}{\text{Initial OD}} \times 100$$

Determination of pH

The pH is determined by measurement of the electromotive force of a cell comprising an indicator electrode (an electrode responsive to hydrogen ions such as glass electrode) immersed in the test solution and a reference electrode (usually a calomel electrode). Contact is achieved by means of a liquid junction, which forms a part of the reference electrode. The emf of this cell is measured with pH meter.

Analysis of Sulphate Concentration

About 100ml of the sample was treated with 20ml of buffer solution A (30 g of MgC_{12} and was dissolved in 5g of sodium acetate, 1g of KNO_3 and 20ml of CH_3COOH in 500ml distilled water). A spoonful of BaC_{12} was added to it. The turbidity was measured. Using standard graph, the concentration of sulphate was measured.

Estimation of Chloride

Ten millilitres of effluent samples in a conical flask was taken and 1ml potassium chromate was added to get light yellow colour. It was then titrated with standard silver nitrate solution till colour change from yellow to brick red.

Colour – Hazen Units

Colour can be measured by spectrophotometrically or using a visual comparator. In both cases, the standard unit of measurement is the Hazen unit (HU). (True colour is often quoted as True Colour Units, or

TCU) Hazen units are defined in terms of a platinum–cobalt standard (APHA Method 2120B 1992). Colour values obtained using a spectrophotometer are dependent on the wavelength used for the measurement. For spectrophotometry method, the British Standard which uses 436 nm (BSI Method BS6068 1986), is suitable.

Total Kjeldahl Nitrogen

Aqueous samples: The sample was mixed thoroughly and transferred to a kjeldahl flask and a few boiling chips or glass beads were added. To remove the excess Ammonia in the sample, 25 mL borate buffer and then 6N NaOH were added until the pH is 9.5. Then the solution was boiled until 10-20 mL remains and cooled to room temperature. The volume was made upto 250 mL with reagent water and finally 50 mL digestion reagent was added to the kjeldahl flask. The resulting solution was boiled until the volume was reduced to approximately 25 mL and dense fumes appear above the liquid. The flask was cooled, and the solution was dilute to 300 mL with reagent water. 50 mL sodium hydroxide-sodium thiosulfate reagent was added to it and mixed thoroughly. The pH was maintained at 11. The tip of the condenser was placed below the surface of solution in the receiving flask. The receiving flask was added with 50 mL of 0.04N H_2SO_4. The solution was distilled at a rate of 6-10 mL/min and 200 mL of distillate was collected and diluted to 300 mL with reagent water. Since the intensity of the color used to quantify the concentration is pH dependent, the acid concentration of the wash water and the standard ammonia solutions should approximate that of the sample. A stable baseline with all reagents was obtained and feed with reagent water through the sample line and the reading for the sample line was recorded. The solid sample dry weight/wet weight ratio must be determined separately.

Oil & Grease

For oil and grease concentration determination, gravimetrical method (APHA, 1998) was used after solvent extraction with xylene.

Heavy Metal Analysis

Heavy metal (Sulphide, Cadmium, Chromium, Lead, zinc, Selenium, Nickel and copper) determination was carried out using atomic absorption spectrophotometer (AAS). APHA 22 nd Edition 3111 B & C

FTIR study

FTIR technique is an interesting application for studying the interaction between an adsorbate and the active groups on the surface of adsorbent. Infrared spectral data obtained from Perkin Elmer FTIR spectrophotometer (Spectrum BX-II) is used to determine the interaction between the sorbents and dyes. For FTIR analysis, pellets are prepared in KBr disks. Sorbent powder and dye loaded sorbent pellets are prepared using the same ratio of sorbent (1 mg) and dye + sorbent (1 mg) in KBr (100 mg, dried at 110oC). The infrared spectra in the range (4000 – 400 cm−1) are recorded for sorbent and dye loaded sorbent pellets [12].

RESULT AND DISCUSSION

Qualitative Analysis of sorbents

To study the presence of various functional group present in the sorbents phytochemical screening was done. (+ = present, - = absent) and the results are tabulated in Table 1. Both Flavonoids and Phenols are seen in most of the sorbents which plays a very important role in case of the phyto-remediation of the textile wastes. Similarly, Terpenoid and Glycoside are absent in all of the sorbent's samples. The bio-sorption property of the plant sorbents can be enhanced by the increased presence of large number of phenolic derivatives present. Due to the oxidizing activity of the phenols, the decolorisation of the effluents will be more effective in process.

Table 1 Phytochemical Screening of flower of the plant Sorbents

Sample	Flavonoid	Alkaloid	Saponin	Tannin	Phenolic compound	Terpenoid	Glycoside
Musa Paradisiaca	-	-	+	+	+	-	-
Nymphaea Nouchali	+	+	+	+	+	-	-
Nelumbo Nucifera	+	+	-	-	+	-	-

Quantitative Analysis of sorbents

The presence of the phenol compounds is evident in all the three different sorbents and it also plays an important role in the process of the phytoremediation and Bio-reduction of toxic compounds and heavy metals. So, the Total phenol content of all the sorbents were quantified with Gallic acid equivalence and are tabulated in Table 2. The Total Phenol content of the *Nymphaea nouchali* was comparably higher than the *Musa paradisiaca* and *Nelumbo nucifera.* So, on conclusion out of all the three different sorbent, Flower sample of the

Nymphaea nouchali has the largest quantity of phenolic compounds which is 80 mg Gallic acid equivalence per gram of the sample following by *Nelumbo nucifera* flower which has 70 mg Gallic acid equivalence per gram of the sample and finally *Musa paradisiaca* flower having 53 mg Gallic acid equivalence per gram of the sample.

Table 2 Total phenol content of the Flower samples

S.No	Sample	Gallic acid standard equivalence
1	*Musa paradisiaca*	53 mg GAE/g of sample
2	*Nymphaea Nouchali*	80 mg GAE/g of sample
3	*Nelumbo Nucifera*	70 mg GAE/g of sample

Anti-oxidant activity of sorbents

The presence of high phenolic content indicates the one of the important activities for phytoremediation which is the Antioxidant property. The antioxidant property of sorbents was studied by method of [9] and resulted are tabulated in Table 3. Since having a high presence of the phenolic compound, the antioxidant activity is also seen in high in-case of the *Nymphaea nouchali* flower sample and it is followed by *Musa paradisiaca,* finally *Nelumbo nucifera* in the last. The *Nymphaea nouchali* flower has the highest antioxidant capacity which is 155 mg/g of the sample which is followed by 93 mg/g of *Musa paradisiaca* Flower. *Nelumbo nucifera* flower has the lowest antioxidant capacity which is 65 mg/g of the plant sample.

Table 3 Antioxidant capacity of the sorbents

S.No	Sample	mg/g of the sample
1	*Musa paradisiaca*	93
2	*Nymphaea Nouchali*	155
3	*Nelumbo Nucifera*	65

The antioxidant capacity is one of the most important property responsible for the phytoremediation and heavy metal absorption capacity of the sorbents, and on-considering the Table 4, it is evident that the *Nymphaea nouchali* flower will have the highest activity against the Heavy metal reduction and decolorisation process. Similarly, *Musa paradisiaca* flower can also show Heavy metal reduction and Decolorisation process due to its anti-oxidant activity.

Table 4 Decolorisation percentage and COD reduction percentage

S.NO	Plant Sample	% Decolorisation	% COD
1	*Musa paradisiaca* flower	68.5	74.5
2	*Nymphaea nouchali* flower	71.2	81.7
3	*Nelumbo nucifera* flower	64.4	71.5

Decolorisation and Chemical Oxygen Demand

The Treated samples from the ASBR's were evaluated for effective decolorisation and COD reduction and the results are tabulated in table 4. Out of all three ASBR's, the reactor having *Nymphaea nouchali* flower showed high percentage of decolorisation and COD reduction which is 71.2% and 81.7% respectively, followed by *Musa paradisiaca* flower having 68.5% of decolorisation and 74.5 % of COD reduction. Finally, the flower sample of *Nelumbo nucifera* plant has a activity of 64.4% of decolorisation and 71.5% of COD reduction.

Table 5 Peaks observed in the FTIR analysis of various Sorbents

Peaks for *Musa paradisiaca*	Peaks for *Nymphaea Nouchali*	Peaks for *Nelumbo Nucifera*
3303.46- PHENOLS ALCOHOLS, O-H	3306.36- Phenols	3353.6- Phenols, alcohols
	2926.45- CH_2, CH	2921.63- CH_2, CH
2922.59- CH_2, CH	2855.1- CH_2, CH	2853.17-C-H
2853.17- CH_2.CH	1725.98- C=0 (acid)	1732.73 C=O (saturated aldhehyde)
1732.73- C=O ACID	1650.77- C=C	
1637.27- C=C	1617- C=C	1629.55- C=C
1446.35- ALPHA CH_2 BEND	1536.02 C=C	1531.2 C=C

	1446.35- ALPHA CH_2 BEND	1436.71 ALPHA CH2 BEND
1375.96- O-C	1351.86- O-C	
1323.89- O-C		1405.85 C-O-H BEND
1247.72-C-O PHENOLS, C-O ACID		1365.35 O-C

Table 6 Comparison of the Treated & untreated Textile Effluent characteristics by ASBR's.

Parameters*	CETP Standards	Textile Dye Effluent without treatment	Textile Dye Effluent after treatment by ASBR-1	Textile Dye Effluent after treatment by ASBR-2	Textile Dye Effluent after treatment by ASBR-3
pH	5.5 – 9.0	7.24	7.56	7.5	7.41
Color (Hazen units)	25	500	350	225	100
TSS	100	418	200	121	32
TDS	2100	3932	3764	3236	2180
BOD	100	310	210	116	42
COD	250	1245	778	340	163
Sulphates Sulphates (as SO_4^{2-})	1000	1905	1400	950	623
Chlorides (as Cl^-)	1000	1200	1150	947	621
Sodium	60	73.98	66	62	59.5
Total Kjedahl Nitrogen	100	153.2	84	63	53.2
Oil & Grease	20	36	22	14	8
Sulphide (as S^{2-})	2.8	42.6	15.8	8.4	6.3
Copper (as Cu)	3.0	0.26	0.26	0.08	0.04
Cadmium (as Cd)	2.0	0.2	0.1	0.05	0.02
Total Chromium (as Cr)	2.0	0.5	0.1	0.05	0.03
Lead (as Pb)	1.0	0.1	0.05	0.03	0.01
Selenium (as Se)	0.05	0.05	0.02	0.015	0.01
Zinc (as Zn)	15	0.36	0.18	0.22	0.36
Nickel (as Ni)	3.0	0.22	0.22	0.12	0.08

*** All values except pH and color are in mg/L**

FT-IR Analysis

The FTIR of the samples revealed functional groups present in them due to phyto -constituents like phenolic, flavonoids, tannins, and carbohydrate compounds present in them. Bands at 3303.36, 3306.46, 3356 cm^{-1} are due to presence of alcohols or phenolic compounds, as the qualitative test showed presence of phenolic compounds in the plant, this bending must be due to presence phenols and flavonoids. The other absorption bands of IR spectrum like C-O (1351.86 cm^{-1}), C=C, C-H (2950-2800 cm^{-1}), =CH were in consistent with presence phyto-constituents analysis [14]. From IR and qualitative analysis of plants, presence of many O groups must be attributing to the phytoremediation property of the plants, as plants in general, use flavonoids, amino acids, polysaccharides, phenols as growth and support supplements, in a form of intermediates which makes pollutants to bind them and make them bioavailable for microbes to degrade them. Besides sometimes plants transfer the contaminants to shoots, leaves or flower for accumulation [15].

Evaluation of the various constituents of the Treated Textile Effluent by ASBRs.

The characterization of the Textile dyes treated by the ASBR's reveals that out the three different plant samples, the sample system which contains flower of the plant *Nymphaea nouchali* showed maximum efficiency of treatment against the Textile dye which

also correlates with the Decolorisation and COD reduction data. The data comparison of the ASBR's was done in order to study the relation between various parameters of the untreated textile dye effluents and treated effluents by ASBRs. From the graphical representation (fig 5, fig 6, fig 7, and fig 8) it has been clearly visible that most of the bio-reduction activity of plant samples are mainly due to the synergic effect of combination of different phytochemicals together in a single ASBR. And out of the three different flower samples, the sample system in ASBR-3 has the overall effective efficiency when compared with rest of the sample systems. So out of 3 different ASBR, it was evident that flower of the plant *Nymphaea nouchali* can be suitable to treat the textile dye effluent before letting it to the environment.

CONCLUSION

Development of such biosorption system becomes necessary for the maintenance of the effluent, before discharge into the natural water bodies. One such system was found out after optimization of various parameters of different plant samples, which led selection of the ASBR-3 system which contains the flower of plant Nymphaea nouchali. Due to high presence of different groups of phyto-constituents in Nymphaea nouchali flower, which has reflected in its higher antioxidant property compared to the rest of plant samples.

The results obtained from the FTIR spectrometric analysis, it was found out there were presence of many O- groups bonds which was responsible antioxidant properties. All biosorbents showed peak in the range of 3300-3400 cm^{-1}, a prominent region known as the detection range for the phenolics and alcohol groups.

From the experimental analysis and data interpretation of the fabricated bioreactor it was evident that the usage of plant materials as biosorbents holds a promising remedy to the textile wastewater treatment and also provides a future potential as a design for the common effluent treatment plants with cost effective utilization of plant sources as biosorbents.

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