



## New bio-sorbents in the removal of brilliant green dye from polluted waters

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### ABSTRACT

Powders of leaves, stems and their ashes of *Thespesia populnea*, *Pongamia pinnata* and *Aeschynomene aspera* are found to have strong affinity towards cationic Brilliant Green Dye at high pH values. The sorption characteristics of these bio-products in controlling the Dye pollution in waste waters, are studied by varying physicochemical parameters such as pH, sorption concentration and equilibration time using simulated waste waters. The conditions for maximum extraction of the Dye have been optimized. Fivefold excess of common anions ions present in natural waters, have marginal interference on the % of extraction while Cation like  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$  and  $\text{Cu}^{2+}$  have shown some interference but  $\text{Fe}^{2+}$  and  $\text{Zn}^{2+}$  have synergistically maintained the maximum extraction of the Dye at optimum conditions of extraction. The methodologies developed in this work have been applied to diverse waste water samples collected in different industries and are found to be remarkably successful.

**Key Words:** Pollution Control, Brilliant Green Dye, *Thespesia populnea*, *Pongamia pinnata*, *Aeschynomene aspera*, applications.

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### INTRODUCTION

Increasing interest is being envisaged by the Environmental researchers in using adsorbents derived from bio-materials, either in their native form or chemically activated form, in controlling toxic pollutants. These biological approaches have stimulated continuous and expanding research in this field and these methods offers a potential alternative to the existing methods of detoxification and the recovery of toxic and valuable ions from industrial discharges/ polluted waters.

The use of these bio-methods in the removal of Dyes from polluted water is an important aspect and is increasingly probed by environmental researchers [1-25]. Such studies are important as the effluents from the Dye-based industries possess substantial amounts of the synthetic Dyes [26-28] which are non-degradable and stable to light, heat and oxidizing agents [29]. The accumulation of the synthetic toxic Dyes are posing problem to the environment as they are toxic to aquatic life and furthermore, they affect the photosynthetic activity due to their color [29].

Brilliant green is an important cationic Dye and is used in various manufacturing industries such as paper, green ink, and textile industries [6]. Further, it is also used in biological straining, and as dermatological agent, veterinary medicine, and an additive to poultry feed to inhibit propagation of mold, intestinal parasites and fungus [8].

The Dye is toxic and its consumption causes gastrointestinal problems, vomiting, diarrhea, nausea and dermatological problems [8, 30-32].

The various biomaterials such as Orange peel [1], banana pith [2], bottom ash [3-5], deoiled soya [6], rice husk [7], kaolin [8], bentonite clay [9], neem leaf powder [10,11], powdered activated sludge [12], perlite [13], powdered peanut hull [14], natural and modified clays like sepiolite [15], zeolite [16], bamboo dust [17], coconut shell [18], groundnut shell [17], Indian Rose wood saw dust [19], rice straw [20], duck weed [21], sewage sludge [22], saw dust carbon [23], agricultural waste and timber industry waste carbons [24] and gram husk [25], have been explored as sorbent in the control of Dyes from wastewaters.

*While we are probing the affinity of different plants materials for their sorption abilities towards cationic Brilliant Green Dye, we noticed affinity between them and sorbents derived from *Thespesia populnea*, *Pongamia pinnata* and *Aeschynomene asperin* plants. The present work is a comprehensive study in optimizing the physicochemical parameters such as pH, sorbent dosage and agitation time, in developing an eco-friendly procedure for the quantitative removal of the Dye from polluted water by effectively evoking the sorption nature of these sorbents and further, the developed methodologies have been applied for some industrial effluent samples.*

### EXPERIMENTAL SECTION

**(A) CHEMICALS:** All chemicals used were of analytical grade.

**Stock solution of Brilliant Green Dye:** 50 ppm of Brilliant Green solution was prepared by dissolving a requisite amount of A.R. grade Brilliant Green Dye in double distilled water. It was suitably diluted as per the need.

**(B) ADSORBENTS:** The plants materials pertaining to *Thespesia populnea*, *Pongamia pinnata* and *Aeschynomene aspera* have been explored in this present study.



A: *Thespesia populnea*

B: *Pongamia pinnata*

C: *Aeschynomene aspera*

**Fig No. 1: Plants showing affinity towards Brilliant Green Dye**

*Thespesia populnea*, commonly known as the *Portia Tree* is a species of flowering plant in the mallow family, Malvaceae. It is a small tree or arborescent shrub that has a pantropical distribution, found on coasts around the world and is supposed to be originated from India [33]. It grows in the wide range of soil types that may be present in coastal environments, including soils derived from quartz, limestone and basalt and favors neutral soils (pH of 6-7.4) [34]. The heartwood of the Portia Tree is dark reddish brown to chocolate brown and is used in woodworks. Traditionally it is planted in sacred groves and used for religious sculpture.

*Pongamia pinnata* is a species of tree in the pea family, Fabaceae, native in tropical and temperate Asia. The tree grows to about 15–25 meters in height with a large canopy which spreads equally wide and be deciduous for short periods. The tree is well suited to intense heat and sunlight and its dense network of lateral roots and its thick and long taproot make it drought-tolerant. The dense shade it provides slows the evaporation of surface water and its root nodules promote nitrogen fixation. The plant is also used in the preparation for diesel for generators.

*Aeschynomene aspera* is a species of flowering plant in the family of Fabaceae It is also known by the names *Sola*, *Sola Pith Plant*, *Pith Plant*, *Laugauni* or *Netti (Tamil)*. **Pith** of low density from this plant is used to make hats known as pith helmets.

The leaves and stems of *Thespesia populnea*, *Pongamia pinnata* and *Aeschynomene aspera* were cut, washed with tap water followed by distilled water and then sun dried. The dried materials were powdered to a fine mesh of size: < than 75 microns and activated at 105<sup>o</sup> C in an oven and then employed in this study. Further these leaves or stem materials were burnt to ashes and these ashes were also used in this work.

#### (C) ADSORPTION EXPERIMENT:

**Batch system of extraction procedure was adopted [35-37].** Carefully weighted quantities of adsorbents were taken into previously washed 1 lit/500 ml stopper bottles containing 500 ml/250 ml of Brilliant Green Dye solution of predetermined concentrations. The various initial pH values of the suspensions were adjusted with dil HCl or dil NaOH solution using pH meter. The samples were shaken vigorously in mechanical shakers and were allowed to be in equilibrium for the desired time. After the equilibration period, an aliquot of the sample was taken for the determination of Brilliant Green Dye using Spectrophotometric method. The Dye has  $\lambda_{max}$  at 628 nm and obeys Beers-Lamber's law at low concentrations. The O.D. measurements were made at the said  $\lambda_{max}$  using UV-Visible Spectrophotometer (Systronics make). The obtained O.D value for un-known solution was referred to standard graphs (drawn between O.D and concentration) prepared with known concentrations of Brilliant Green by adopting method of Least Squares.

The adsorption nature of the three sorbents was probed with respect to the time of equilibration, pH and sorbent dosage. At a constant sorbent concentration, the % removal of Brilliant Green from sample waters was studied with respect to time of equilibration at various pH values. The results obtained were presented in the Graph Nos. A: 1-12. To fix the minimum dosage needed for the maximum removal of the Brilliant Green Dye for a particular sorbent, extraction studies were made by studying the % of extraction with respect to the sorbent dosage at optimum pH and equilibration times. The results obtained were presented in the Graph Nos. B: 1-3.

#### (D) EFFECT OF OTHER IONS (INTERFERING IONS):

The interfering ions chosen for study were the common ions present in natural waters viz. Sulphate, Fluoride, Chloride, Nitrate, Phosphate, Carbonate, Calcium (II), Magnesium (II), Copper(II), Zinc(II) and Nickel (II). The synthetic mixtures of Brilliant Green and of the foreign ions were so made that the concentration of the foreign ion was maintained at five fold excess than the Dye concentrations as cited in the Table: 1. 500ml of these solutions were taken in stopper bottles and then correctly weighted optimum quantities of the promising adsorbents (**as decided by the Graph Nos. A and B**) were added. Optimum pH was adjusted with dil. HCl or dil. NaOH using pH meter. The samples were shaken in shaking machines for desired optimum periods and then small portions of the samples were taken out, filtered and analyzed for Brilliant Green. % of extraction was calculated from the data obtained. **The results were presented in the Table: 1.**

#### (E) APPLICATIONS OF THE DEVELOPED BIO-SORBENTS:

The workability of the developed bio-sorbents for removing the cationic Brilliant Green Dye was tried using real sewage/effluent samples collected from some Dyeing industries at Hyderabad and Bombay. For this purpose, samples were collected from effluents of industries and the samples were analyzed for actual amounts of Brilliant Green Dye and samples were fed with known amounts of Brilliant Green Dye. Then these samples were *subjected to extraction for the Dye adopting the methodologies developed in this work at optimum conditions of extraction as given in the Table 2. The results obtained were presented in the Table 2.*

### RESULTS AND DISCUSSION

Our pilot experiments revealed that *Brilliant Green Dye* has affinity towards the biomaterials of *Thespesia populnea*, *Pongamia pinnata* and *Aeschynomene aspera*. So, the present work is a comprehensive report on the extraction of the said Dye with respect to various physicochemical parameters such as *pH, time of equilibration and sorption concentration*. The results obtained have been depicted in the Graph No.A: 1-12 and Graph No. B: 1-3. *The following observations are significant:*

**1. Time of equilibration:** Time of agitation has been found to have profound effect on the % of removal of the Dye. % removal is found to be increasing with the increase in the contact time at a fixed pH until a saturation state is reached and after such a time, the extractability remains constant (vide Graph Nos. A: 1-12). In other words, there is no further adsorption. As for example, in the case of powder of leaves of *Thespesia populnea*, % of extraction of *Brilliant Green Dye* has been found to be 70.0% at 15min, 80.4% at 30 min, 92.5% at 45 min, 100% at 60 min or

above, at pH: 8 (vide Graph No. A: 1) Similarly with the stem powders of *Pongamia pinnata*, the % of extraction of the Dye is found to be 74.6 at 15 min, 90.3% at 30 min and 100 at 45 min or above (vide Graph No. A: 7). **The same trend is notice in other sorbents (vide Graph Nos. A: 1-12).**

**2. Effect of pH:** The sorbents of the present study have been found to be pH sensitive. The removal of the Dye is almost 'nil' or marginal at pH: 1 and below but substantially increased with the increase in the pH conditions of agitation system (vide Graph No. : 1-12). As for example in case of powders of *Thespesia populnea* leaves, the maximum extractability has been found to be 2.0% at 1.0 N HCl; 2.5 % at pH: 1; 86.7% at pH: 2 ; 100% at pH: 4 and above, at 60 minutes of equilibration time and with sorbent dosage of: 1.0 g/500 ml (vide Graph No.A:1; B: 1). With the ash of leaves of *Thespesia populnea*, the % of extraction has been found to be 0.5% at 1.0 HCl, 0.4% at pH: 1; 80.2 % at pH: 2; 95.0% at pH: 4; 100% at pH: 6 and above, after an equilibration time of 45 minutes with a sorbent concentration of 0.5 gm/500ml (vide Graph No.A:2 and B: 1). With stem powders of *Thespesia populnea*, 100% extractions has been observed throughout the pH range : 2 to 10 after an equilibration time of 45 min with 0.5 g/500 ml of the sorbent dosage while with its ash, even 0.25 gm/500ml of sorbent concentration is sufficient to remove completely the Dye after the same equilibration time (Graph No.A:3 & 4; B:1).

Plant materials derived from *Pongamia pinnata* have shown good sorption characteristics towards Brilliant Green Dye with the increase of pH. In the pH range 2-10, 100% removal of the Dye has been observed for the powder of leaves at an equilibration time of 60 minutes and sorbent dosage of 0.75 gm/500 ml; and with their ashes at an equilibration time of 45 minutes and sorbent dosages of 0.50 gm/500 ml. With sorbents derived from stem powders of *Pongamia pinnata* plant, 100% of removal has been observed in the pH range 2-10 but with varying sorbent concentrations: 0.75 gm/500ml for raw stem powders and 0.5 gm/500 ml for their ashes (vide Graph No. A: 7 & 8; B: 2).

With the sorbents pertaining to *Aeschynomene aspera*, the complete removal of the Dye has been observed above pH: 2, at optimum conditions of time of agitation: 60min for leaves powder and 45 min for their ash; 45 min for both stems powder and their ash at the optimum sorbent concentration: 0.5gm/500 ml for raw powders (both leaves and stems) and 0.25 gm/500 ml for their ashes (vide Graph Nos. A: 9-12 and B: 3).

**3. Optimum equilibration time:** The minimum time needed for the maximum extraction of the Dye under optimum conditions of extraction, is found to be low for ashes than with raw leaves powder: 60 minutes with all the plants while 45 minutes with their ashes (vide Graph No.A: 1, 2, 5, 6, 9 and 10). It is interesting to note that in the case stems or their ashes for all the plants of study, the optimum agitation time has been found to be same viz., 45 min. (vide Graph No .A: 3, 4, 7,8,11 and 12).

**4. Sorbent Concentration:** The minimum optimum concentration of sorbent needed for the maximum extraction of the Brilliant Green Dye is observed to be less for ashes than the raw plant materials: in the case of *Thespesia populnea*, 1.0 gm/500ml for leaves powder while 0.5 gm/500 for their ashes; 0.5 gm/500ml for stem powders while 0.25 gm/500ml for their ashes. In the case of *Pongamia pinnata* plant, the optimum sobent dosage has been found to be: 0.75 gm/500ml for leaves powder and 0.5 gm/500ml for their ashes; 0.75 gm/500 ml for stems powder and 0.5 gm/500ml for their ashes. With the *Aeschynomene aspera* as sorbent, the dosage has been found to be 0.5 gm/500ml for leaves powder while 0.25/500ml for their ashes; 0.5 gm/500ml for stem powder while 0.25 gm/500ml for their ashes (vide Graph No.: B:1-3).

**5. Interfering Ions:** The effect of the presence of foreign ions on the extraction of the Brilliant Green Dye from simulated waters has been presented in the Table No.1. It can be inferred that the common ions present in the waters viz., Sulphate, Phosphate, Chloride, Carbonate, and Fluoride have marginal effect while Cations namely,  $Ca^{2+}$ ,  $Mg^{2+}$  and  $Cu^{2+}$  have interfered to some extent but  $Fe^{2+}$  and  $Zn^{2+}$  have synergistically maintained almost the complete extraction of the Dye.

## DISCUSSION

The surface morphology of bio-sorbents plays an important role. The surface functional groups present in these biomaterials are either -OH-or -COOH groups. The pH sensitive dissociation of these groups imports charge on the inter surface and thereby, an urge for oppositely charged ions prevails on the surface of the sorbents. At low pH values, the dissociation of the functional groups is less favored and even protination of the functional groups may

occur and due to this, there is a surface electrostatic thrust for anions. But at high pH values, the functional groups dissociate imparting negative charge to the interface and thereby a thrust for cations prevails.

Brilliant Green Dye being a cation especially in the pH range 2 to 10 is binding to the surface of the adsorbent at high pHs and this results in higher % of removal. As pH of extraction system decreases, the binding nature of the surface decreases due to non-dissociation of the functional groups and even protonation of functional groups, which imports some positive charge to the surface and this manifests in the decrease in % of extraction of the Brilliant Green Dye.

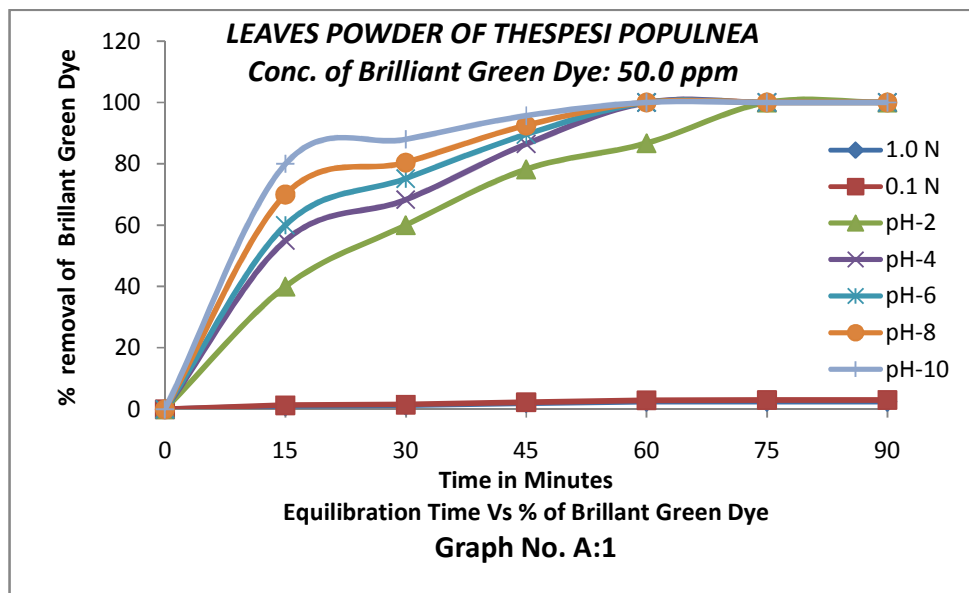
Ashes are the oxides of some heavy metals containing large amounts of silica. The ashes, contains '-OH' groups and '-O-'. and observed surface sensitivity may be accounted in the same lines as described in the case of raw leaves or stem powders. In fact, in the literature it is reported that the silica possesses cation exchanging nature [38-40] and this supports the proposed logic for the observed behavior.

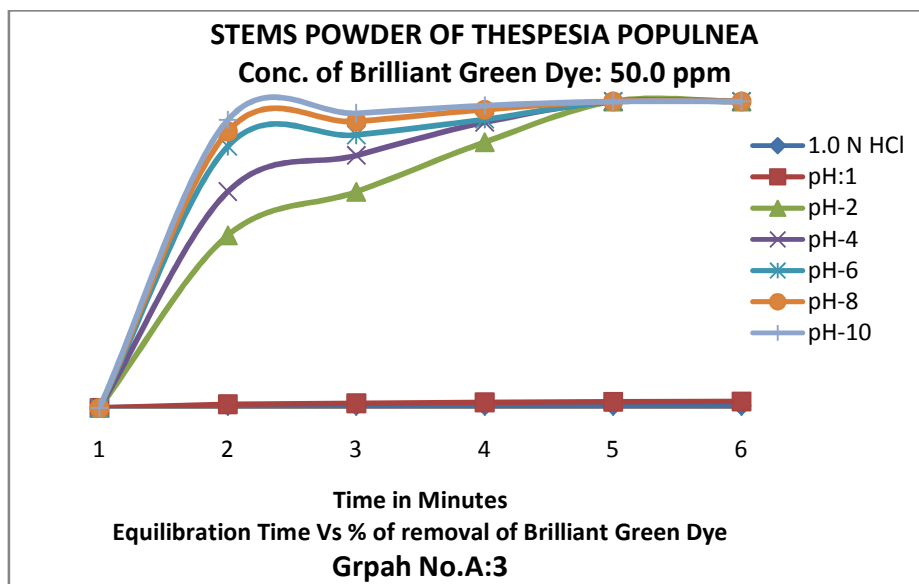
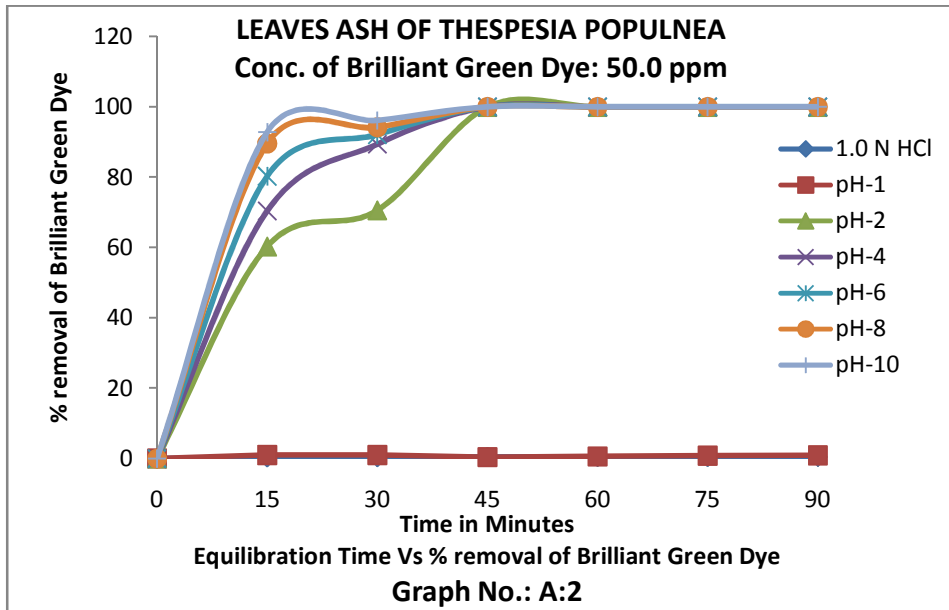
The decrease in the rate of adsorption with the progress in the equilibration time may be due to the more availability of adsorption sites initially and are progressively used up with time due to the formation of adsorbate film on the sites of adsorbent and thus resulting in decrease in surface sorption ability of the adsorbent.

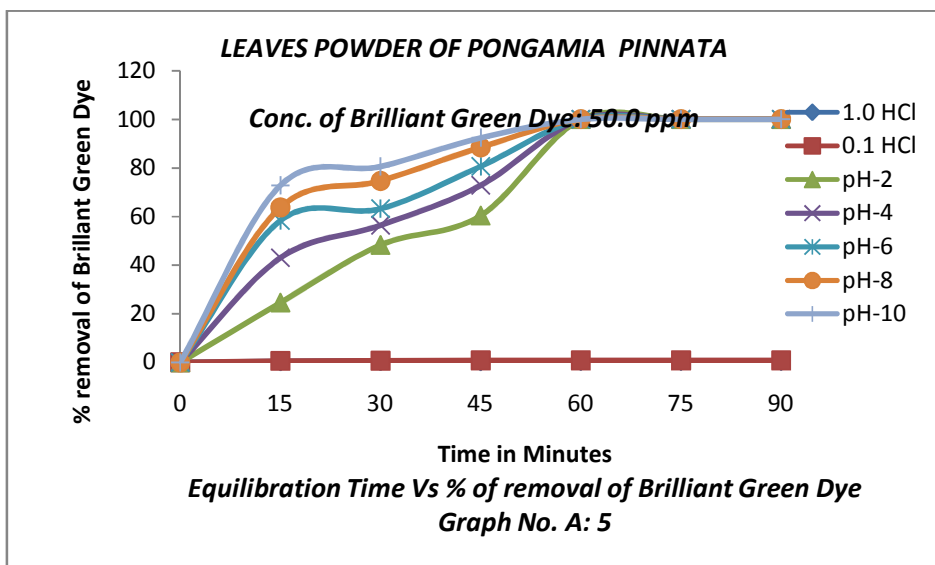
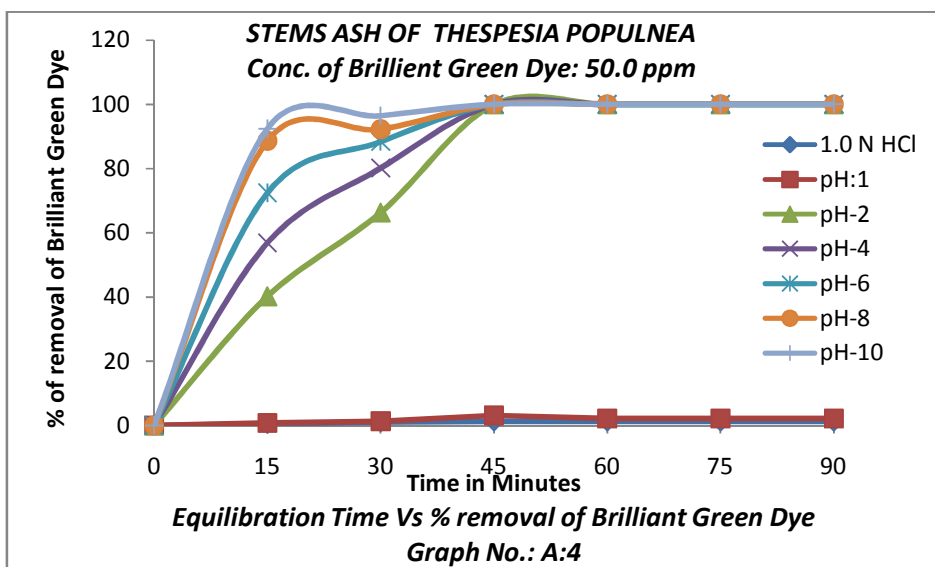
The observed data pertaining to the effect of foreign ions on the extraction the Dye confirms this concept. Foreign anions are found to be less innocuous to the extractions while some Cations like  $Ca^{2+}$ ,  $Mg^{2+}$  and  $Cu^{2+}$  are found to be interfering. Anions being negative in nature cannot be sorbed to negatively charged surface of the sorbent at high pH values while Cations being positive in nature compete with cationic Dye for sorption sites resulting inference. In the case of  $Zn^{2+}$  and  $Fe^{2+}$ , the % removal of the Dye is not affected because the  $Zn^{2+}$  ion forms negatively charged zincate at the high pH values resulting seldom affinity towards the sorbent while  $Fe^{2+}$  gets precipitated as its hydroxide at high pH conditions of extraction and thus resulting precipitate also adsorbs or traps the Dye effecting the complete removal.

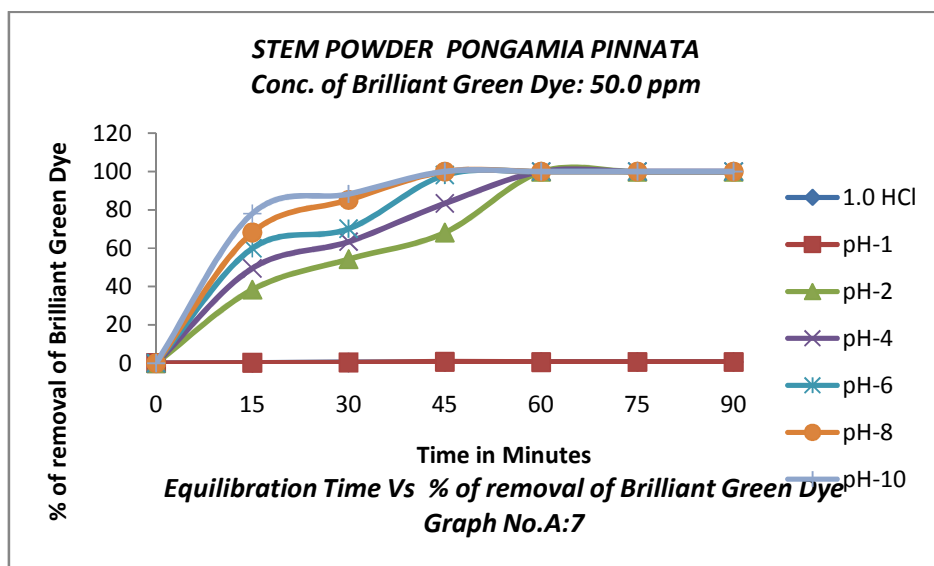
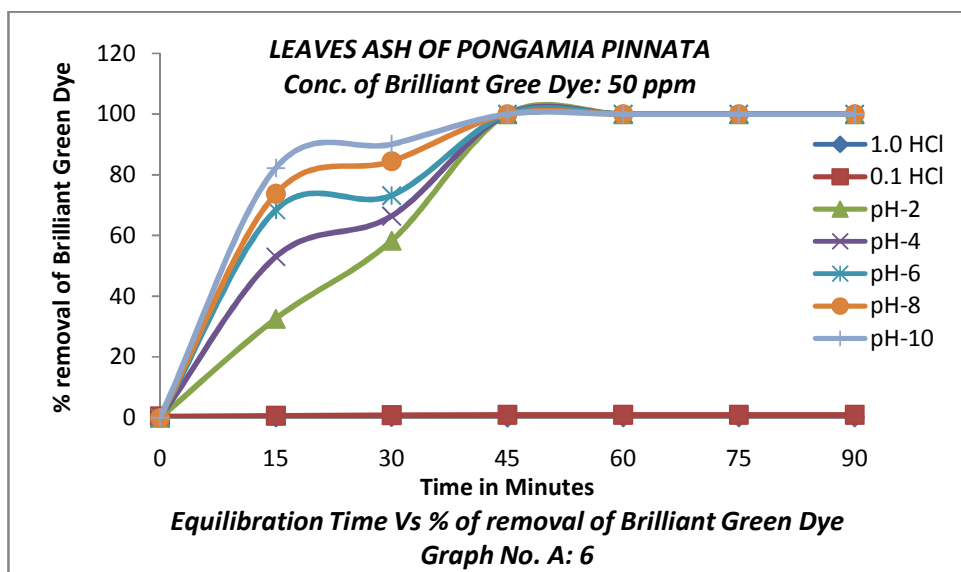
#### APPLICATIONS

The procedures developed in this work have been applied for samples collected from the sewages/effluents of Dyeing industries which are fed with varying quantities of the Brilliant Green Dye. **The results have been presented in the Table No: 2.** It can be inferred from the data that the procedures are remarkably successful.

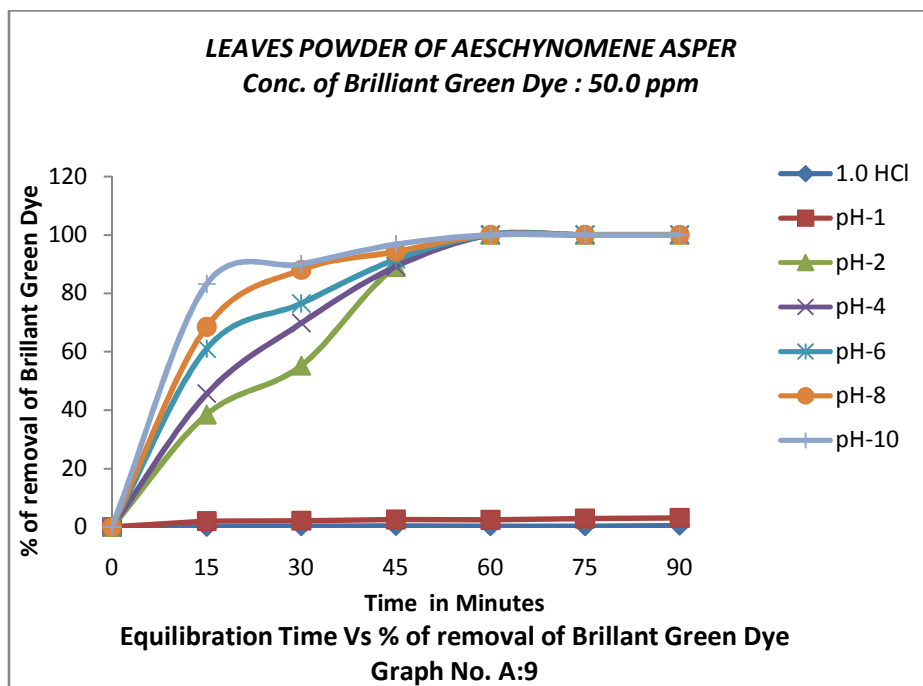
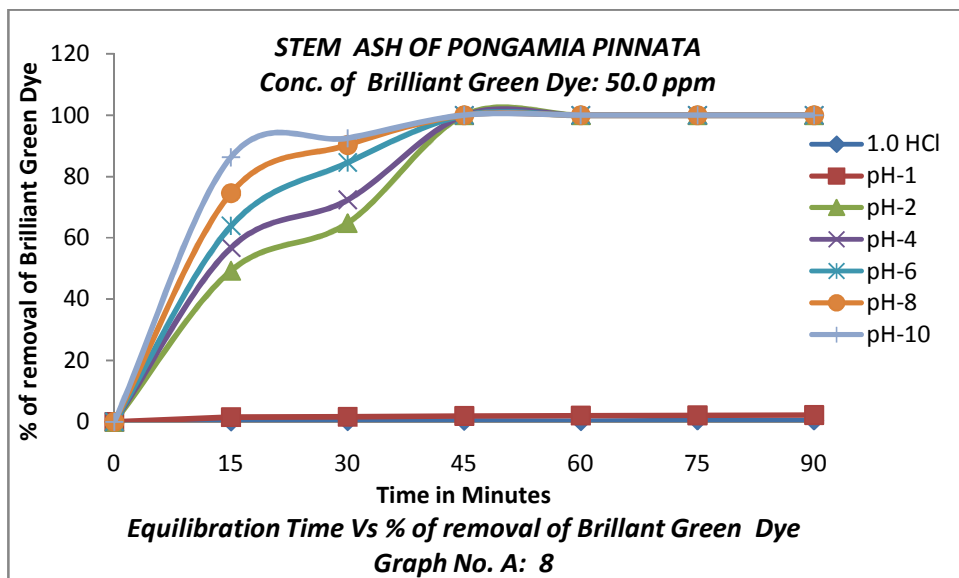


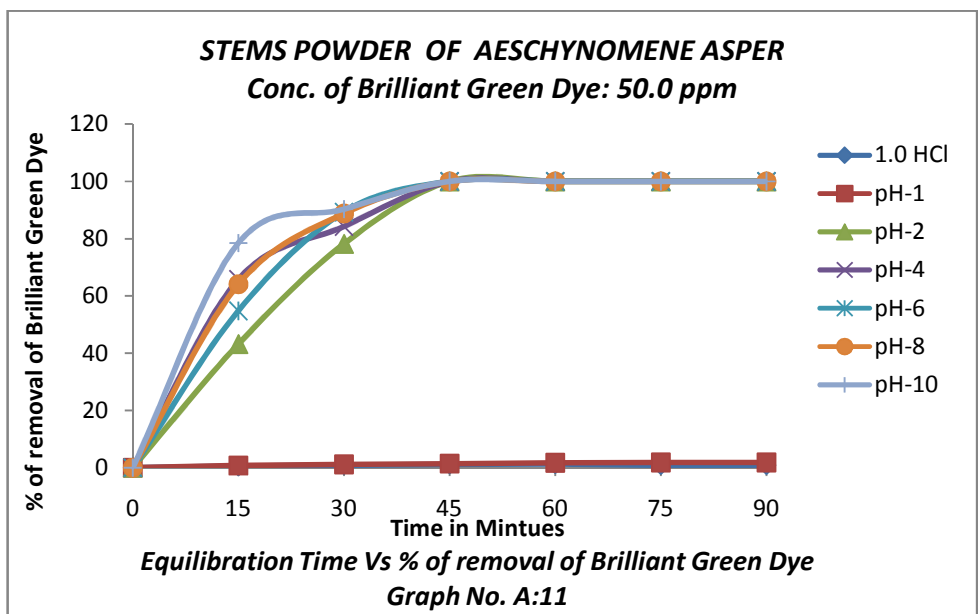
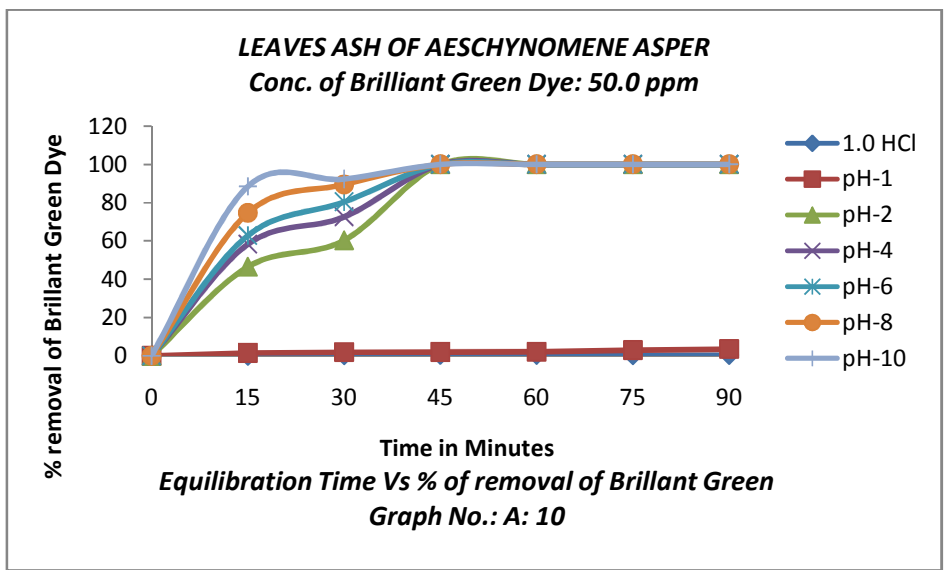


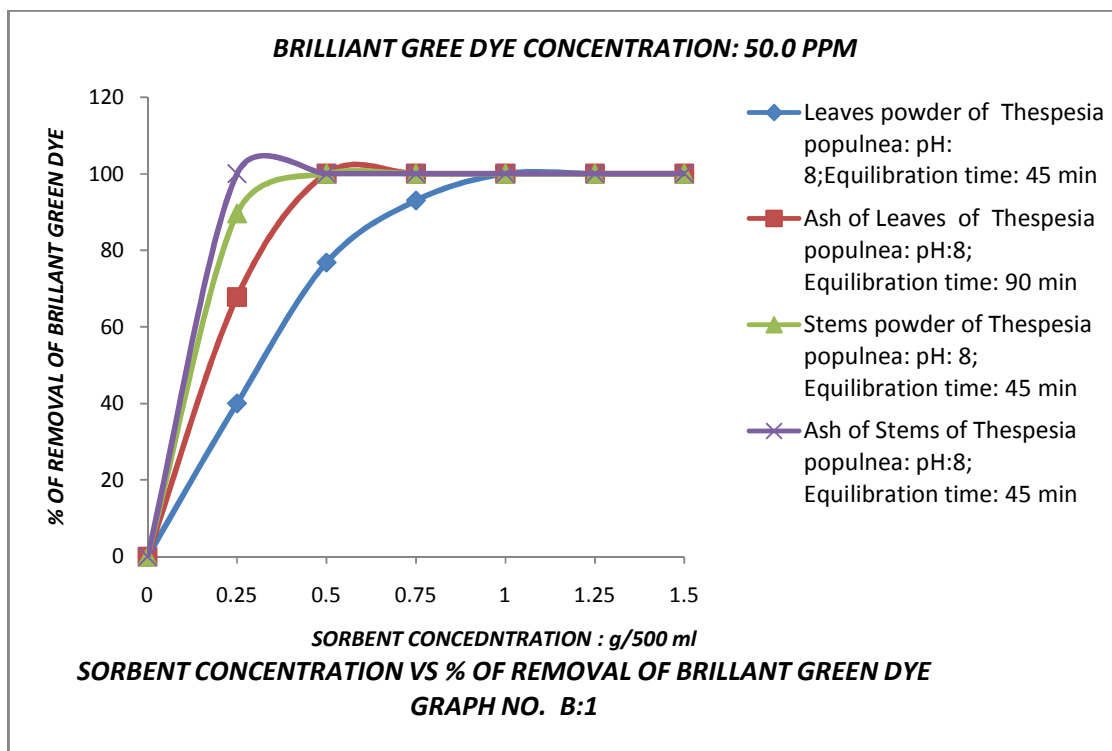
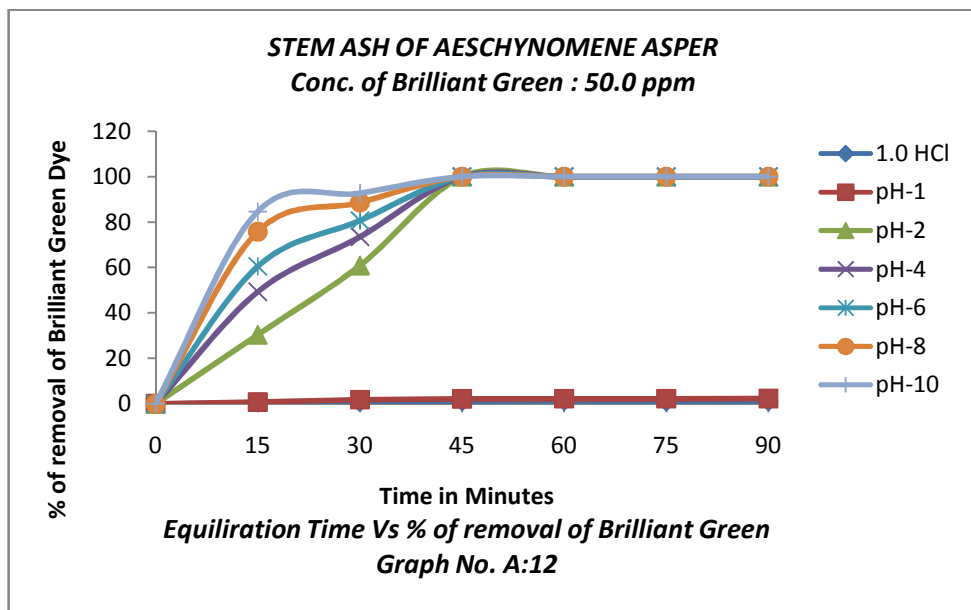












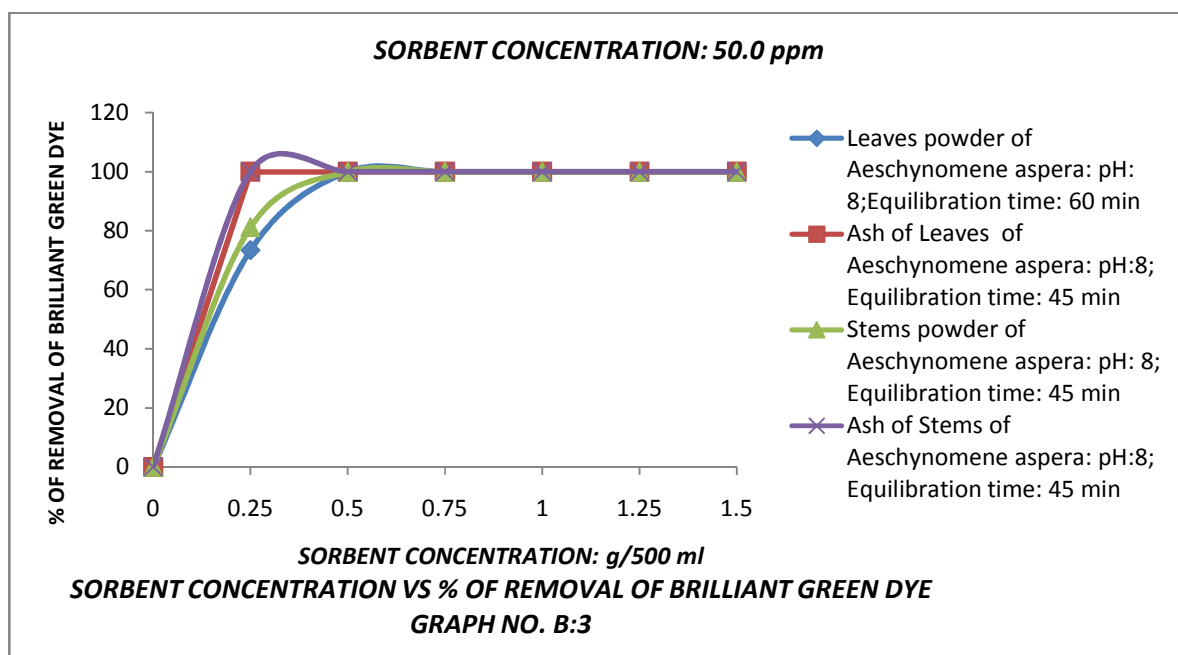
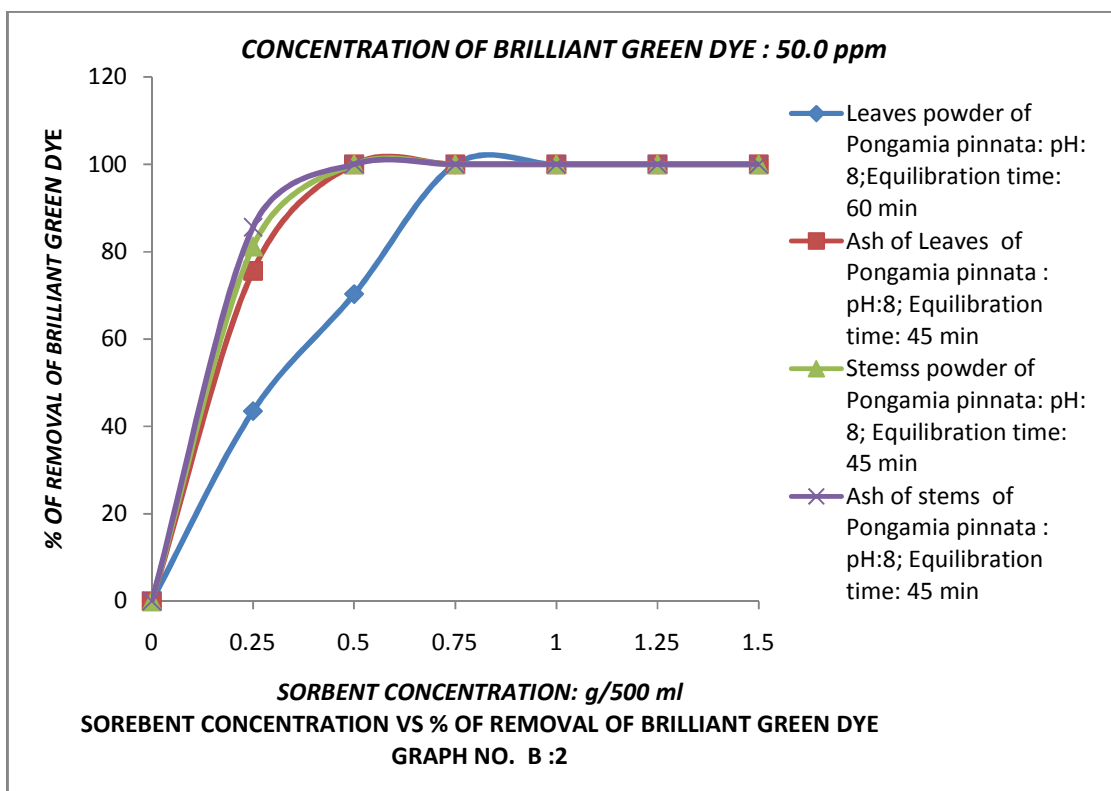


Table No. :1: Effect of interfering Ions on the Extractability of Brilliant Green Dye with different Bio-sorbents

Adsorbent and its concentration	Maximum Extractability at optimum conditions	% of Extractability of Brilliant Green Dye in presence of fivefold excess of (50 ppm) interfering ions at optimum conditions: Conc. of Brilliant Green Dye: 50 ppm									
		SO <sub>4</sub> <sup>2-</sup>	PO <sub>4</sub> <sup>3-</sup>	Cl	CO <sub>3</sub> <sup>2-</sup>	F	Ca <sup>2+</sup>	Cu <sup>2+</sup>	Fe <sup>3+</sup>	Zn <sup>2+</sup>	Mg <sup>2+</sup>
Leaves powder of <i>Thespesia populnea</i>	100.0%; pH: 8; 60 minutes; sorbent conc.: 1.0 g/500 ml	97.5	97.0	100.0	96.5	94.0	85.0	85.2	100.0	99.5	83.0
Leaves Ash of <i>Thespesia populnea</i>	100.0%; pH:8;45 min; Sorbent conc.:0.5g/500 ml	96.4	96.5	100.0	97.3	93.0	78.0	83.2	100.0	99.0	75.5
Stem powders of <i>Thespesia populnea</i>	100.0%; pH:8; 45 minutes; Sorbent conc.:0.5 g/500 ml	97.0	95.5	100.0	100.0	96.5	83.0	80.0	100.0	100.0	73.0
Stems ash of <i>Thespesia populnea</i>	100.0%;pH:8, 45 miutes; Sorbent conc.: 0.25 g/500 ml	97.5	94.5	100.0	97.9	95.5	81.5	79.0	100.0	100.0	80.5
Leaves powder of <i>Pongamia pinnata</i>	100.0%; pH:8; 60 minutes; Sorbent conc.:0.75 g/500 ml.	96.7	95.0	100.0	100.0	92.0	80.0	84.0	100.0	99.0	81.0
Leaves Ash of <i>Pongamia pinnata</i>	100.0%; pH:8; 45 minutes; 0.5 gm/500 ml	95.5	94.0	100.0	100.0	93.5	79.0	78.0	100.0	98.9	78.5
Stem powders of <i>Pongamia pinnata</i>	100.0%; pH:8; 45 minutes; Sorbent Conc.: 0.75 g/500 ml	95.2	96.5	100.0	95.0	97.5	78.0	74.0	100.0	100.0	76.5
Stem ash of <i>Pongamia pinnata</i>	100.0%; pH:8; 45 minutes; Sorbent Conc.: 0.5 g/500 ml	94.2	94.0	100.0	100.0	93.5	73.5	72.0	100.0	100.0	73.0
Leaves powder of <i>Aeschynomene aspera</i>	100.0 %; pH: 8; 60 minutes; Sorbent conc.:0.5 g/500 ml.	97.8	93.5	100.0	100.0	96.0	72.0	86.7	100.0	100.0	72.5
Leaves Ash of <i>Aeschynomene aspera</i>	100.0%; pH:8; 45 minutes; Sorbent Conc.: 0.25 g/500 ml	96.5	94.8	100.0	100.0	95.0	75.5	87.0	100.0	100.0	77.0
Stem powders of <i>Aeschynomene aspera</i>	100.0%; pH:8; 45 minutes; Sorbent Conce.: 0.5 g/500 ml	97.0	96.0	100.0	100.0	92.0	80.5	80.0	100.0	100.0	78.5
Stems ash of <i>Aeschynomene aspera</i>	100.0%; pH:8; 45 minutes; Sorbent Conc.: 0.25 g/500 ml	96.8	97.0	100.0	100.0	930	83.0	81.0	100.0	100.0	82.5

TABLE NO.2: % OF EXTRACTABILITY OF BRILLIANT GREE DYE FROM DIFFERENT INDUSTRIAL EFFLUENTS WITH BIO-SORBENTS DEVELOPED IN THIS WORK

Bio-Sorbent	% of Extractability of Brilliant Green Dye				
	Sample 1: Fed with 10.0 ppm of Brilliant Green Dye	Sample 2 Fed with 15.0 ppm of Brilliant Green Dye	Sample 3 Fed with 20.0 ppm of Brilliant Green Dye	Sample 4 Fed with 25.0 ppm of Brilliant Green Dye	Sample 5 Fed with 30.0 ppm of Green Dye
Leaves powders of <i>Thespesia populnea</i> :at pH: 8; Equilibration time: 60 minutes; sorbent conc.: 1.0 g/500 ml	89.5	83.0	85.0	82.0	91.0
Leaves ashes <i>Thespesia populnea</i> : at pH: 8; Equilibration time: 45 min; Sorbent conc.:0.5g/500 ml	88.0	82.5	89.0	83.5	89.0
Stem powders of <i>Thespesia populnea</i> :at pH:8; Equilibration time: 45 minutes; Sorbent concentration: 0.5 g/500 ml	92.5	82.0	81.0	84.0	88.0
Stems ash of <i>Thespesia populnea</i> :at pH:8; Equilibration time:45 minutes; sorbent concentration: 0.25 gm/500 ml	94.0	84.0	86.5	85.0	87.0
Leaves powders of <i>Pongamia pinnata</i> :at pH:8; Equilibration time: 60 minutes; sorbent concentration: 0.75 g/500 ml	90.0	85.0	84.5	86.2	86.5
Leaves ash of <i>Pongamia pinnata</i> :at pH:8; Equilibration time: 45 minutes; sorbent concentration: 0.5 g/500 ml.	91.0	86.2	87.0	90.0	87.0
Stems powder of <i>Pongamia pinnata</i> :at pH:8; Equilibration time: 45 minutes; sorbent concentration: 0.75 g/500 ml.	89.0	84.0	88.0	91.0	92.0
Stems ash of <i>Pongamia pinnata</i> :at pH:8; Equilibration time: 45 minutes; sorbent concentration: 0.5 g/500 ml.	92.5	87.5	86.0	83.5	86.0
Leaves powder of <i>Aeschynomene aspera</i> : pH: 8; Equilibration time: 60 minutes; Sorbent conc.:0.5 g/500 ml.	95.2	90.5	83.0	88.9	85.0
Leaves Ash of <i>Aeschynomene aspera</i> : at pH:8; Equilibration time: 45 minutes; Sorbent Conc.: 0.25 g/500 m	93.5	92.0	82.5	87.0	87.0
Stem powders of <i>Aeschynomene aspera</i> : at pH:8; Equilibration time: 45 minutes; Sorbent Conc.: 0.5 g/500 ml	90.5	88.0	89.0	92.0	82.0
Stems ash of <i>Aeschynomene aspera</i> : at pH:8; Equilibration time: 45 minutes; Sorbent Conc.: 0.25 g/500 ml	93.5	87.0	90.0	93.0	83.0

### CONCLUSION

1. Bioadsorbents derived from plant materials of *Thespesia populnea*, *Pongamia pinnata* and *Aeschynomene aspera* have been found to be effective in the extraction of Brilliant Green Dye from polluted waters at high pH values.
2. Physicochemical parameters such as pH, time of equilibration and sorbent concentration have been optimized for the maximum removal of Brilliant Green Dye.
3. Methodologies have been developed to remove substantial amounts of the Dye from simulated waters in all the sorbents probed at optimum conditions of extraction.
4. The procedures developed are successfully applied for some industrial samples.

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