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### Adsorption Behavior of Solochrome Black T Dye from Aqueous Solution Using Raw Rice Husk and Rice Husk Biochar: Characterization, Kinetics, and Isotherm Studies

Sabrina, S.T.F, Venujah, P and Arasaretnam, S\*

*Department of Chemistry, Faculty of Science, Eastern University, Sri Lanka*

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#### ABSTRACT

Water pollution and dyes are closely linked, particularly due to industrial activities. Dyes are significant pollutants, often released into water bodies through industrial effluents from textile industries, pulp mills, and dye manufacturing plants. These colored wastewater discharges contain various chemical compounds that can be harmful to aquatic ecosystems and human health. This study deals with the adsorption of Solochrome Black T on rice husk (RH) biochar. The removal of Solochrome Black T dye from aqueous solutions is significant due to its environmental and health impacts. Using pyrolyzed and raw biochar as adsorbents is important because it offers a cost-effective and sustainable solution for removing such contaminants. Biochar has a high surface area and porosity, which make it effective for adsorbing organic pollutants like dyes from water, thereby mitigating environmental contamination and promoting water safety. Characterization of the prepared materials has been conducted by the Fourier transform infrared spectroscopy (FTIR) and X-ray diffraction (XRD). From the analyses, it is revealed that the operational parameters of initial concentration, adsorbent dosage, contact duration, and temperature were analyzed, with one factor changeable at a time and the others constant. A peak at  $3276\text{ cm}^{-1}$  has been observed in raw RH, indicating the presence of free water molecules, which disappear in biochar following the pyrolysis process. This suggests that higher dehydration and aromatization occurred during the pyrolysis process. The XRD pattern identifies a widening peak of silica peak diffraction at  $2\theta = 22^\circ$ . It is shown that the prepared material is more porous with a higher surface area compared with its parent material. The adsorption process mechanism was found as physio-sorption mode dominating over chemisorption mode of removal of dye, following pseudo-second order model and spontaneous nature (indicated from  $-\Delta G^\circ$ ). Maximum adsorption capacity of RH to remove Solochrome dye in optimized conditions is  $46.948\text{ mg g}^{-1}$ . Separation factor 'RL' point outs the feasibility of this process, which is further confirmed by the linearity of equilibrium adsorption isothermal data correlation coefficient values.

**Keywords:** Biochar, Rice husk, Solochrome Black T, Pyrolysis.

#### 1. INTRODUCTION

Biochar has been increasingly essential in the modern era due to its widespread applicability. It is a carbonaceous substance with porous structure. In addition, this will be accomplished through thermochemical conversion of organic molecules in the absence of oxygen. Moreover, this process is known as pyrolysis [1].

Recent years have seen the usage of biochar in a variety of environmental applications as an adsorbent, catalyst, soil conditioner, and energy store. Numerous forms of biomass are accessible for production, including crop residues, wood wastes, animal manures, food wastes, municipal solid wastes, and sewage sludge [2-4].

\* Corresponding author: [s\\_arasaretnam@esn.ac.lk](mailto:s_arasaretnam@esn.ac.lk)

There have been numerous studies undertaken recently to generate biochar and composite materials. Several factors influence how the adsorbent and adsorbate interact. Some of these include the nature of the contaminants, the specific surface area, the pore volume, the adsorbent's hydrophobicity, and surface functionalization. The surface chemistry of modified or unmodified biochar has a high sorption capability for removing different contaminants from wastewater<sup>[5-8]</sup>.

In light of the world population, rice is the most important staple food. Additionally, the husk from the production and processing of rice is regarded as a major agricultural waste worldwide. Adsorption is regarded as a crucial process in the treatment of water because of its flexibility, efficacy, and capacity to remove a wide range of contaminants. It also offers a dependable means of improving water quality and guaranteeing the supply of safe and clean water to communities and industries<sup>[9]</sup>.

Adsorbents with cheap costs provide a workable technique to remove pollutants through the adsorption process. Adsorbents' properties are important for the adsorption process, but they are also useful instruments in a variety of industries because to their economic and environmental benefits. These include cost effectiveness, occurrence, flexibility, ease of customization, and efficient removal of pollutants<sup>[10]</sup>.

According to earlier research, raw biochar has a low potential to adsorb CO<sub>2</sub>. In any case, the biochar's alteration has greatly improved CO<sub>2</sub> adsorption. Chemical activation strategies that produce oxygen-containing functional groups by oxidizing the surface of biochar include exposing it to acidic or alkaline solutions. Additionally, the physical activation methods enhance the surface area of the biochar since they rely on high-temperature steam to enter the material's pores. By employing steam, acidic, or alkaline solutions, these activation methods efficiently establish a network of linked microspores in the biochar, increasing its surface area as well as the number of functional groups that contain oxygen<sup>[11]</sup>.

Nowadays, there is a great deal of scientific and public interest in developing low-cost materials and effective technologies to produce adsorbents for the treatment of dye-polluted wastewater areas. Activated carbon is the traditional adsorbent, but its use is limited due to its higher cost and manufacturing process difficulties. Several low-cost materials have been used to adsorb dyes in the past, including chitosan composites, agricultural solid wastes, clay and modified clay, etc<sup>[12]</sup>.

Water accounts for 71 % of the Earth's surface. And 2.5 % represents merely fresh water, which has the potential to be contaminated by human activities. The most prevalent contaminants are heavy metals, pesticides, metalloids, medicines, polyaromatic hydrocarbons, and dyes<sup>[13]</sup>.

The textile, pulp, and dye-making industries release dyes, a significant class of pollutants, as coloured waste water. These brightly coloured dyes are harmful and have an adverse effect on both human and aquatic life. Because of these adverse effects, synthetic dyes should be properly handled before being discharged into water. These effects include carcinogenic, mutagenic, and teratogenic effects.

Currently, a number of methods, including chemical oxidation, biological treatment, photo-degradation, membrane treatment, and adsorption treatment are used to treat wastewater containing colours. Adsorption was deemed the superior treatment approach among those described for a number of reasons, including being less expensive, more effective, having a simpler production process, and being insensitive to the pollutant<sup>[14]</sup>.

The present study deals with the adsorption of Solochrome Black on RH biochar. The broad objective of this study is to convert RH which is a popular agricultural residue in Eravur, Eastern Province. The removal of Solochrome Black dye from aqueous solutions is significant due to its environmental and health impacts. Dyes like Solochrome Black are often used in various industries but pose serious risks if released untreated into water bodies, causing water

pollution and potential harm to aquatic life and human health. Using pyrolyzed and raw biochar as adsorbents is important because it offers a cost-effective and sustainable solution for removing such contaminants. Biochar has a high surface area and porosity, which make it effective for adsorbing organic pollutants like dyes from water, thereby mitigating environmental contamination and promoting water safety. This research contributes to both environmental protection and sustainable development goals by providing a practical method for treating dye-contaminated wastewater.

## 2. MATERIALS AND METHODOLOGY

### 2.1 Preparation of RH

In this study, RH used as a precursor was sourced from Eravur, Eastern Province, Sri Lanka. The RH was manually cleaned, thoroughly washed with deionized water to remove impurities and dirt, and then dried in direct sunlight for 15 days. After drying, the RH was ground using a grinder and sieved.

The sieved RH was soaked in a HCl ( $0.5 \text{ mol dm}^{-3}$ ) solution (1:20) at room temperature for 48 hours with continuous agitation at 200 RPM on a shaker. Following this, the RH was washed, filtered, and soaked in a NaOH ( $0.5 \text{ mol dm}^{-3}$ ) solution (1:20) under the same conditions. After the NaOH treatment, the RH was repeatedly washed with distilled water and dried at  $40^\circ\text{C}$  for 48 hours. The dried RH was then ground again using a grinder. The ground sample was tightly packed in a porcelain crucible with a fitting lid and pyrolyzed at various temperatures ( $500^\circ\text{C}$ ,  $600^\circ\text{C}$ ,  $700^\circ\text{C}$ ,  $800^\circ\text{C}$ , and  $900^\circ\text{C}$ ) in a furnace (Carbolite Gergo 30-3000  $^\circ\text{C}$ ) under oxygen-limited conditions for 3 hours. The resulting biochar samples, referred to as RH500, RH600, RH700, RH800, and RH900, were stored in dark glass bottles until use [15,16]

### 2.2 Biochar adsorption experiments

Batch Adsorption studies for optimizing parameters to remove the Solochrome Black T from aqueous medium was done with  $100 \text{ mg L}^{-1}$  initial concentration. Parameter optimization was studied step by step while keeping one factor

variable at a time, and other constant as followed: Concentration 50 ppm-250 ppm adsorbent dose (0.5-4.5 g), Agitation time 10 mins-120 mins. After experiment Adsorbent was filtered under a pump and remaining supernatant layer was determined Spectro photochemically by using UV spectrophotometer (BK-D580) at 491nm ( $\lambda_{\text{max}}$ ). The supernatant layers after removal of Biochar which were pyrolyzed at different temperature varying from  $500^\circ\text{C}$ ,  $600^\circ\text{C}$ ,  $700^\circ\text{C}$ ,  $800^\circ\text{C}$  and  $900^\circ\text{C}$  were analyzed same condition at the UV spectrophotometer. Optimized conditions were employed at the same time varying concentration of dye gradually for isothermal investigations, keeping another parameters constant. For kinetic studies, optimized parameters were employed by varying agitation time gradually, keeping others constant.

### 2.3 Characterization of RH

FTIR measurements were performed by using a Perkin Elmer Spectrum One NTS FT-NIR spectrometer (Bruker). FTIR spectra of all samples were taken in the range of  $4000$  to  $400 \text{ cm}^{-1}$  with a resolution of  $16 \text{ cm}^{-1}$  and the temperature was recorded in each measurement. The FTIR spectra of raw RH and RH800 were measured with an average of 20 scans. RH800 was further characterized by XRD (ULTIMA IV).

## 3. RESULTS AND DISCUSSION

FT-IR studies were performed to study about the functional groups responsible for the adsorption of EBT on RH. The FTIR spectra of raw RH, pyrolyzed RH have been shown in Figure 1.

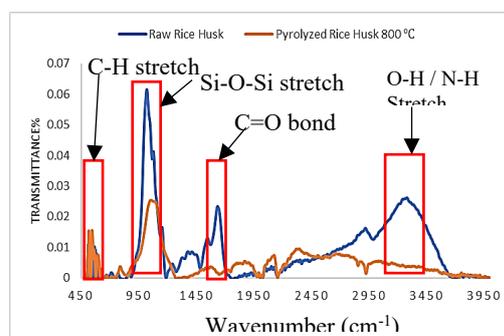


Figure 1. FTIR spectrum of raw rice husk and rice husk biochar.

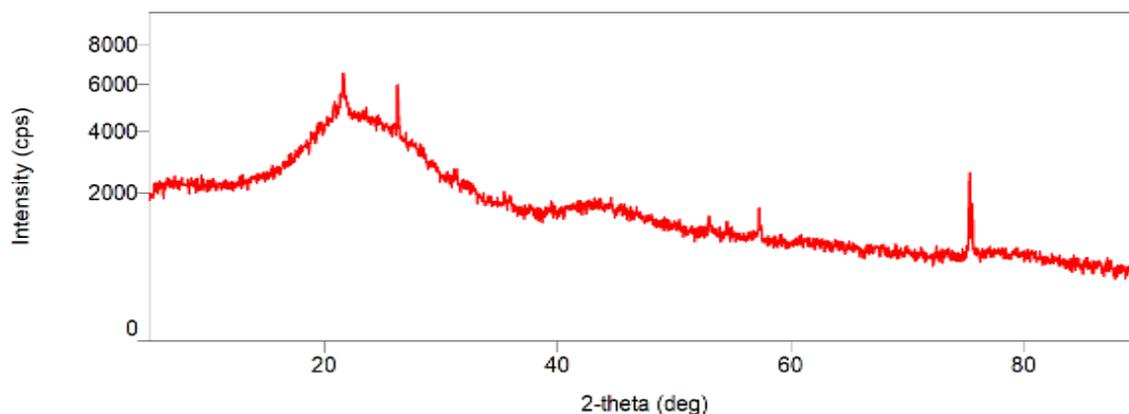


Figure 2. X-ray diffraction of RH pyrolyzed at 800 °C.

The spectrum clearly indicates the presence of carbon, hydrogen, oxygen and nitrogen in RH. The spectra displayed the presence of a strong absorption band at  $3276\text{ cm}^{-1}$  in all two samples. This absorption band indicates the presence of  $\text{-OH/-NH}$  stretching frequencies. So, it can be assumed that the peak at  $3450\text{ cm}^{-1}$  is most probably due to  $\text{-OH}$  stretching vibrations. Furthermore, it is also evident that this peak is not the same shape and size in all two samples. In case of sample RH biochar, it is a bit shallow and less wide while in case of sample RH it is wider and more flattened and it is the smallest in terms of depth and width.

The distortion of this peak in sample RH indicates that  $\text{-OH}$  functional groups may be interacting with EBT through intermolecular or intramolecular H-bonding and is thus playing an important role in the removal of EBT from water samples. The band at  $2870\text{ cm}^{-1}$  denoted the presence  $\text{-C-H}$  stretching vibrations. The shape and texture are almost the same in all two samples which may indicate the lesser role of  $\text{-C-H}$  vibrations in the removal of EBT. The peak at  $1730\text{ cm}^{-1}$  indicates the stretching vibrations of carbonyl group ( $\text{-C=O}$ ). It can be observed from Fig. 1 that this absorption band undergoes a significant decrease in the absorption intensity, as compared to the other sample, which is an indication of the thorough involvement of  $\text{-C=O}$  functional groups with the dye EBT. In RH sample, these two peaks are of weak intensities thus denoting their involvement in the removal

process. Another peak around  $1571\text{ cm}^{-1}$  may be representing  $\text{C-C}$  bond bending vibrations. This peak is more prominent in the RH sample, less prominent in the sample RH biochar. It can be said that in the biochar sample involved the adsorption successfully and broadened the peaks, which will be more functional to dyes removal. Peak at  $3276\text{ cm}^{-1}$  is denoting presence of free water molecule in raw RH which is disappeared in RH biochar. This implies that greater dehydration and increased aromatization occurred during pyrolysis process. Further intensity of peak responsible for vicinal silanol group reduced on pyrolysis which ultimately increases the surface area for adsorption<sup>[17]</sup>.

XRD pattern of the rice husk biochar has been shown in Figure 2. shows the intense peaks at  $20^\circ$  which represent the presence of the  $\text{SiO}_2$  into the sample as expected. The rice husks mostly having silicon. Additionally, in Figure 2 the noticeable peaks are shown at the point  $22^\circ$  and  $43^\circ$ . The XRD pattern identifies a widening peak of silica peak diffraction at  $2\Theta = 22^\circ$ . About  $22^\circ$  detected silica phase corresponding to Silica It is considered that silica has an amorphous form. An amorphous-like pattern that indicated the presence of a significant porous structure in the material<sup>[18]</sup>.

Many parameters influence the adsorbent's adsorption capacity, including pH, sorbent dosage, temperature, contact duration, and initial concentration. In this study, the operational parameters of initial concentration, adsorbent

dosage, contact duration, and temperature were adjusted, with one factor changeable at a time and the others constant.

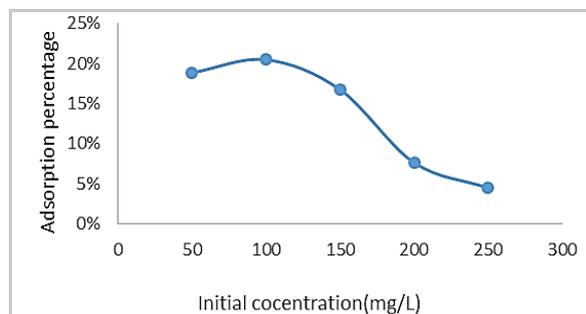


Figure 3. Effect of initial concentration on the removal of Solochrome Black dye by Raw RH

The impact of the initial concentration of solochrome black ( $50\text{-}250\text{ mgL}^{-1}$ ) was tested using 1g of adsorbent and 30 minutes of contact time shows in Figure 3. The dye removal rate increased from 5 to 20 %. The adsorption capacity of the raw RH has been increased initially with the increasing initial concentrations. But, later at the higher initial concentrations the adsorption capacity of the materials does not change significantly with the increasing concentrations. Principally, the more available solochrome black dye create more driving forces to be adsorbed in the available binding sites of the adsorbents and this driving force might break the resistance between the liquid and solid phase [19,20]. But, at the higher concentrations may be due to the saturation of the binding sites into the adsorbents the adsorption capacity does not noticeably increased with further increase in the initial concentrations [19, 21].

For the optimization study, the investigation of the effect of the dosages of the adsorbents is very much needed. In the present study, we investigate the effect of the different dosages of the adsorbents (RH) on the removal percentages, as well as on the adsorption capacity of the adsorbents. In Figure 4, the effect of different adsorbent dosages from 0.5-4.5 g on the adsorption capacity and removal percentages has been plotted. It is found that the removal percentages of the adsorbents were increasing with the increasing number of adsorbents up to

the 55 % removal at the dosage 3 g. Nevertheless, on contrary the adsorption capacity of the adsorbents was gradually decreasing with the increasing amount of the adsorbents. In case of the adsorption capacity, higher amount of adsorption mass may not be saturated [22].

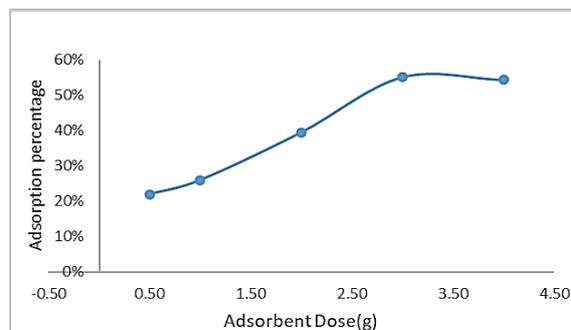


Figure 4. Effect of adsorbent dose on the removal Solochrome black dye by Raw RH.

For checking the effect of contact time between raw RH and Solochrome black dye solution on adsorption phenomenon, dye sample solutions were treated with 3g adsorbent for various interval of time with constant agitation at 100 rpm (Figure 5). It is tangible from this figure that 66 % of dye is removed within 60 min. The increase in adsorption of dye with increasing time is due to the fact that solochrome black dye molecules generally form mono-layer on adsorbent molecule [23]. Thus, adsorption of dye from aqueous media is controlled by the transport rate of adsorbate species from outer to inner sites of adsorbing material.

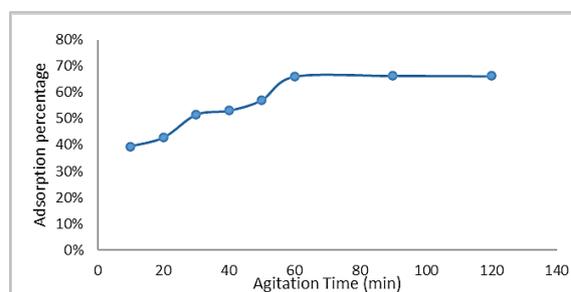


Figure 5. Effect of contact time on the removal Solochrome black dye by Raw rice husk

To check the effect of pyrolysis on removal of Solochrome black, RH was pyrolyzed at different

temperature vary from 500 °C-900 °C. Dye sample solutions were treated with 3g of biochar adsorbent for 60 min of time with constant agitation at 100 rpm (Figure 6).

It evident that dye removal efficiency of biochar was higher than raw RH. The RH biochar sample which was pyrolyzed at temperature 800 °C (RH800) removed higher percentage of dye from solution up to 91 %. Possible reason for this observation, enlargement of surface area with temperature and activation of silicon base functional groups. When temperature was elevated over 800 °C, Significant decline was observed in adsorption efficiency with increasing temperature due to Surface area collapse over 800 °C<sup>[24]</sup>.

Kinetics behaviour of bio sorption of solochrome black dye was evaluated by controlling mechanism such as, mass transfer or chemical reaction, pseudo first and second order kinetic model is used to modulate the experimental data. Lagergren pseudo-first order rate equation.

$$\log[(q_e - q_t)] = \log[q_e] - \left(\frac{K_i}{2.303}\right)t \dots\dots\dots (1)$$

Here ' $k_i$ ' is first order rate constant, ' $q_e$ ' is the dye amount in  $mg\ g^{-1}$  adsorbed at equilibrium, ' $q_t$ ' is the dye amount in  $mg\ g^{-1}$  adsorbed at a specified time. Based on experimental results, linear plot of  $\log(q_e - q_t)$  versus ' $t$ ' is shown in Figure 7 for pseudo-first order kinetic modelling of equilibrium data. The kinetic data was further tested with pseudo-second order kinetics, which is given in equation. 2.

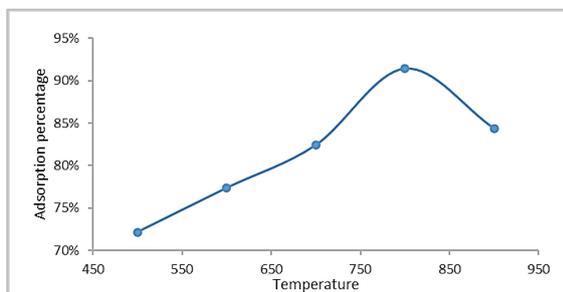


Figure 6. Effect of pyrolysis Temperature on the removal Solochrome black.

It assumes that chemisorption involving valency forces through coordination or switch over of electrons linking adsorbent (RH) and adsorbate

(Solochrome black) is the rate determining step [25].

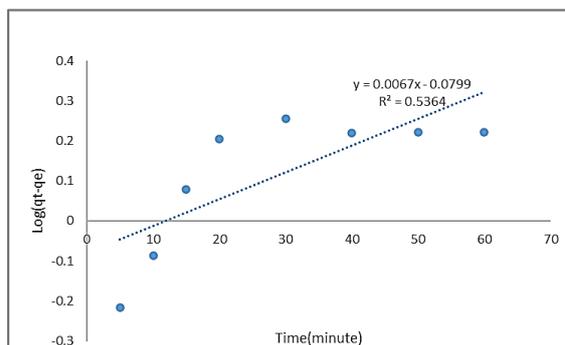


Figure 7. Test of pseudo-first order equation for adsorption of Solochrome Black by pyrolyzed RH.

$$\frac{1}{q_t} = \left(\frac{1}{k_2 q_e^2}\right) \frac{1}{t} + \frac{1}{q_e} \dots\dots\dots (2)$$

Here ' $k_2$ ' is the second order rate constant. Its respective graph of  $1/q_t$  versus  $1/t$  is shown in Figure 8 for pseudo-second order kinetic modelling of equilibrium data.

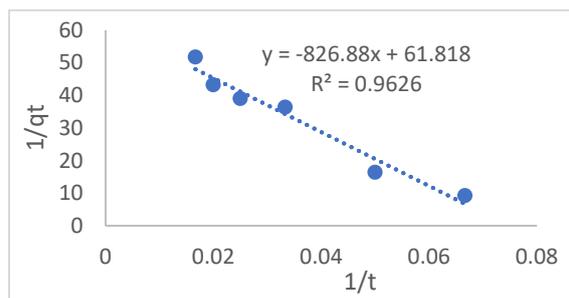


Figure 8. Test of Pseudo-second order equation for adsorption of Solochrome Black by pyrolyzed RH.

It is clear from these graphs that pseudo-first order kinetic model is not applicable on the adsorption of Solochrome Black by pyrolyzed RH, because correlation coefficient  $R^2$  value is far from unity, i.e., 0.5364. It means that concentration of dye and adsorbent quantities, both matters in determining adsorption equilibrium. It is further confirmed by the applicability of pseudo-second order kinetic model with correlation coefficient  $R^2$  value close to unity, i.e., 0.9626.

Langmuir and Freundlich model were applied to investigate the removal of Solochrome Black dye by pyrolyzed RH. The equilibrium data obtained with varying concentration of adsorbate and fixed dose of adsorbent was applied to the Langmuir model as given below in equation 3.

$$\frac{1}{q} = \frac{1}{b q_m C_e} + \frac{1}{q_m} \dots\dots\dots (3)$$

Where  $C_e$  equilibrium concentration ( $\text{mgL}^{-1}$ ) metal solution,  $q_e$  metal adsorption efficiency ( $\text{mg g}^{-1}$ )  $q_0$  maximum adsorption efficiency ( $\text{mg g}^{-1}$ ).  $b$  ( $\text{L mg}^{-1}$ ) is the Langmuir constant representing energy of adsorption. Langmuir models fitted with experimental data with coefficient  $R^2$  0.9818, the assumption of Langmuir model is monolayer adsorption mechanism with no interaction between the molecule adsorbed over homogenous surface [26].

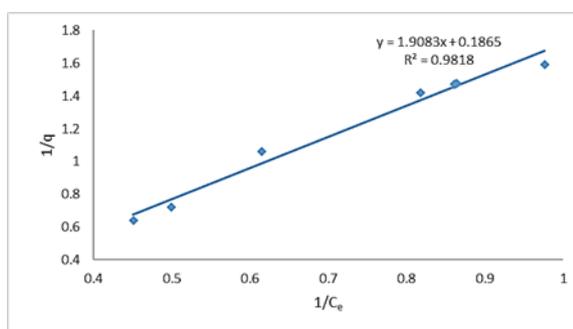


Figure 9. Langmuir isotherm for adsorption of Solochrome Black by pyrolyzed RH.

Based on the  $R^2$  given in the graphs, Dye adsorption by RH biochar conformed fitted to Langmuir isotherm Langmuir (Figure 9).

Separation factor ' $R_L$ ' which is related to the affinity of binding sites, was calculated with equation given below.

$$R_L = \frac{1}{(1 + bC_0)} \dots\dots\dots (4)$$

$R_L$  ranging from 0 to 1 indicates favourable adsorption since the ' $R_L$ ' is found to be 0.4866 which indicates the irreversible nature of favourable adsorption.  $\Delta G^\circ$  came out as  $-16.890 \text{ kJ mol}^{-1}$ , which indicates the nature of this process (Table 1).

The equilibrium data obtained with gradually varying concentration of Solochrome Black T and 3.0 g adsorbent dose was applied to the Freundlich model given in equation. Its basic assumption is that non-uniform distribution of adsorbate on heterogeneous surface with physisorption [27]

$$\log q = \log K_f + \frac{1}{n} \log C_e \dots\dots\dots (5)$$

Where  $K_F$  ( $\text{mg}^{1-1/n} \text{L}^{1/n} \text{g}^{-1}$ ) is adsorption capacity and ' $n$ ' ( $\text{L/mg}$ ) is adsorption intensity, Freundlich constants their values are obtained from graph plotted between  $\log q_e$  versus  $\log C_e$  (Figure 10). The value of ' $1/n$ ' in range of 0 to 1 (Table 2), figure out that the adsorption is compatible with the Freundlich isotherm mode.

Table 1. Langmuir and thermodynamically modelling of equilibrium data for adsorption of Solochrome Black T of RH biochar

Langmuir Isotherm Parameters					Separation factor $R_L$	Thermodynamical parameters $\Delta G^\circ$ ( $\text{kJ mol}^{-1}$ )
Slope	Intercept	$R^2$	$q_m$ ( $\text{mg.g}^{-1}$ )	$b$ ( $\text{L g}^{-1}$ )		
1.9083	0.1865	0.9818	46.948	0.0105499	0.4866	-16.890

Table 2. Freundlich and thermodynamically modelling of equilibrium data for adsorption of Solochrome Black T by RH biochar

Freundlich Isotherm Parameters				
Slope	Intercept	$R^2$	$K_F$ ( $\text{mg}^{1-1/n} \text{L}^{1/n} \text{g}^{-1}$ )	N
0.8498	-0.186	0.9419	0.6516	1.1768

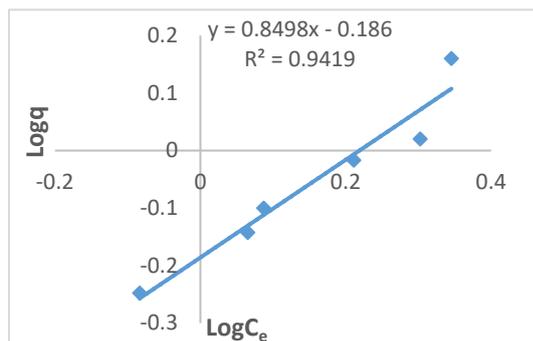


Figure 10. Freundlich isotherm for adsorption of Solochrome Black T by RH biochar.

#### 4. CONCLUSION

In this study, RHs were pyrolyzed to make biochar. The adsorbent's capacity for adsorption was affected by a number of factors, sorbent dose, temperature, length of contact, and initial concentration. The concentration of the dye solution greatly influenced the maximum adsorption of Solochrome Black on raw RH, which happened at 100 mg/L. Pyrolyzing RH biochar at 800 °C resulted in up to 91 % colour removal from the solution. This might be attributed to the increase in surface area with temperature and the activation of silicon base functional groups. Adsorption effectiveness decreased significantly at temperatures above 800 °C owing to surface area collapse. FTIR analysis showed that dehydration and aromatization occurred during pyrolysis process. Further intensity of peak responsible for vicinal silanol group reduced on pyrolysis which ultimately increases the surface area for adsorption.

Rice husk biochar has the active surface charges along with different functional groups (C–O, N–H, –OH, C=C and C–O) revealed from FTIR and XRD analysis might be the reason of the higher cationic dye adsorption capacity.

The XRD pattern showed a broadening peak of silica peak diffraction. Silica is thought to have an amorphous structure. An amorphous-like pattern showed the presence of a large porous structure in the material. The adsorption process mechanism was found as physio-sorption mode dominating over chemisorption mode of removal

of dye, following pseudo-second order model and spontaneous nature (indicated from  $-\Delta G^\circ$ ). Maximum adsorption capacity of RH to remove Solochrome dye in optimized conditions is 46.948 mg g<sup>-1</sup>. Separation factor 'RL' points out the feasibility of this process, which is further confirmed by the linearity of equilibrium adsorption isothermal data correlation coefficient values.

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