



## REMOVAL OF EOSIN STAIN FROM AQUEOUS SOLUTION BY RICE HUSK

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### Abstract

This paper aims to determine Rice Husks (RH) as a bio-adsorber for eosin stain removal from aqueous solutions by adsorption and the adsorptive features of eosin Y are available and to assess the capacity of eosin y dye in aqueous solutions. The RH particles prepared were characterized by X-ray diffraction (XRD), Scanning Electron Microscopy (SEM) and Fourier-transform infrared spectroscopy (FTIR). The RH particles were produced and the parameters for adsorption were analyzed including the time of touch, adsorbent dosage, dye concentration, pH and turmoil. The best adsorption from the color dye concentration between 1 to 50 mg/l was determined using batch method, calibration curves were drawn and dye adsorbent amount calculated. The results demonstrated that the best eosin dye concentration had a maximum sorption ratio of 1 mg/l and a sorption ratio of 100%. The indication from Langmuir and Freundlich isotherms for rice husk has been fitted with adsorption isotherms. In comparison to alkaline pH, rice husk showed better adsorption at acidic pH. all, the rice husks showed the potential to be used as an adsorbent in treatment systems of biological effluents.

**Keywords:** Adsorption, Eosin stain, Rice husk, Dye, Pollution.

### Introduction

Many non-ferrous manufacturing and chemical plants produce a large amount of dye wastewater. Due to the high concentrations of chroma in dye wastewater, it is difficult to dissolve and even dangerous, with specific employees very active in the handling of dye wastewater. Membrane isolation, ion exchange, biodegradation, chemical precipitation, and adsorption are the common treatment methods for wastewater. Adsorption is considered a very effective and inexpensive method of treatment for large industrial applications. It has the properties of acid and alkali resistance, its initial production and its relatively high ratio as one of the most commonly used adsorbents. There are many of source use as adsorbent, recent investigations focused on effectiveness of low cost adsorbents like pearl millet husk Selverani (2000), neem leaf powder Walker and Weatherly (1998), coconut husk Low and Lee (1990), wheat straw Robinson *et al.* (2002), and Verma and Mishra (2006), sewage sludge Otero *et al.* (2003), maize cobs Lin (1987), perlite Dogan *et al.* (2000), wood Poots *et al.* (1978), banana pith Namasivayam and Kanchana (1993), peat Poots *et al.* (1976), natural adsorbent Nassar and Guendi (1991), agricultural waste Nawar and Doma (1989), chitin Annadurai and Krishnan (1996), and rice husk Kadirvelu *et al.* (2000), Feng *et al.* (2004), and Chuah *et al.* (2005) in the removal of dyes from wastewater effluent. Rice husk meets the requirements in terms of active carbon production as a cheap and plentiful biomass resource. Rice is in nature tough, insoluble in water, woody and characterized by its abrasive resistance and structural arrangement of silica-cellulose Daifullah *et al.* (2004). High demands for the adsorption capacities of all the above adsorbents, low in cost and biodegradable, are high. Its major organic components for cellulose, hemicellulose, lignin or inorganic components of rice husks include SiO<sub>2</sub>, H<sub>2</sub>O, Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>, K<sub>2</sub>O, Na<sub>2</sub>O, CaO and MgO Purkait *et al.* (2005), Foo *et al.* (2009), and Hameed (2009). In order to avoid the environmental hazards Eosin Y, a coal tar- xanthene dye was selected as the model anionic dye Chatterjee *et al.* (2005) It is widely used in textile, color solvents, inks, paints, varnishes, paper, and

plastics industries because it cannot be biodegraded by traditional activated sludge processes Zhou, He (2008). The adsorption of the above stain from its aqueous solutions to rice husk was investigated in this study. The impact on the batch experiments was investigated for various parameters, including pH, agitation time, temperature, concentration and adsorbent doses. Figure1 shows the chemical structure of the eosin stain.

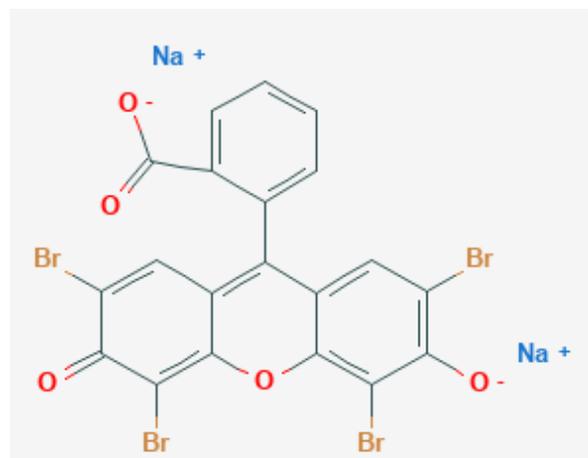


Fig. 1: Chemical structure of Eosin stain.

### Material and Method

#### Preparation of Adsorbent and Adsorbate

The Al Mashkhab Rice Mill located at Al-Najaf Government gathers organic rice husks. To remove impurities such as sand, dirt and other impurities, they are washed 7-10 times with distilled water. Then dried in a hot air furnace for 2 hours at 95-100 °C. At room temperature, rice husk was cooled and rice husks were then saved for further use in the dry, clean plastic bottles. Before adsorption trials, no other physical or chemical approaches were used. Eosin Y was ordered from Ricca Chemical and used without further purification for adsorption experiments. The correct amount of eosin of 1 g in 100ml within a ratio of 50:50 of water and ethanol with magnetic rings has been dissolved to

prepare 100 ml of adsorbate solution (stock solution). Then a 0.4 ml of acetic acid solution was added in the blending phase drop-by-drop at a concentration of 1 mol/l to boost eosine dyes. It was then placed in a volumetric container for the preparation of other diluted solutions. The dilution from the stock solution was used to prepare samples of different dye concentrations. A 250 mL beaker was taken at the required concentration. The stock solution is diluted by 50 times, which means that 2 ml of stock solution is diluted to 100 ml for the preparation of 20 ppm of solution. Solutions of 1, 5, 10, 20, 30, 40, and 50 ppm are typically have been taken in a variety of labeled bottles for 24 hours to homogenize. Such solutions were used to create calibration curves and to test the kinetic and balance. For determining the calibration curve of  $\mu$  max eosin color and the total absorbance to be determined, i.e., 517 nm, for eosin (UV) spectrometer (Shimadzu U.V-visible, Japan.1800). The percentage of dye adsorption from the aqueous solution can be calculated according to the equation:

$$\% \text{ Removal} = \frac{[C_0 - C_e]}{C_0} \times 100\% \quad \dots(1)$$

Where, C (mg/l) is the initial concentration and Ce (mg/l) is the concentration at any time respectively.

#### Batch adsorption

Batch experiments have shown that the solid/liquid interface adsorbing effect leads to a change in the adsorbate concentrators (i.e., the color to be studied) in the solution. Adsorption isotherms are constructed by measuring the adsorbate concentration of medium in a batch model at room temperature before and after adsorption. Batch adsorption to test several parameters such as the concentration effect of 1-50 ppm, a contact time of 60-180 min, adsorbate dose (RH) of 0.25-2 g, pH of 2-10, agitation frequency of 100-500 rpm.

Batch adsorption experiment was performed by removal for different intervals of 60, 90, 120, 130, 150, and 180 minutes from several 100 mL glass bottles sampling wastewater with various quantities of 0.25, 0.5, 1.0, 1.25, and 2.0 g. On a flat orbital mechanical shaker, each solution was mixed for lasted 2 hours until the equilibrium was reached. The solution was then filtered (with Gelman membrane filter of 0.45  $\mu$ m). The UV-VIS spectrophotometry scanners (SHIMADZU) at a wavelength of 517 nm to eosin Y were used to determine filtrate absorbance. Apply acid aliquots and simple solutions of HCl and NaOH before applying RH modified the pH values for solutions. The quantity of adsorption at balance  $Q_e$  (mg/g) was determined using the following equation Li Z, Wang *et al.* (2018).

$$Q_e = \frac{C_0 - C_e}{m} \times v_{\text{sol}} \quad \dots(2)$$

Where,  $Q_e$  is an amount of solute adsorbed per unit weight of adsorbent in mg per gram,  $C_0$  is a dye initial concentration in mg per liter,  $C_e$  is a liquid-phase concentration of dye at equilibrium in mg per liter,  $V_{\text{sol}}$  is the volume of solution in liter and m is the mass of dry sorbent used in gram.

## Results and Discussion

### Photoabsorption Spectrum

Eosin Y is subject to a tautomeric equilibrium between many structures, pH-impaired (lactone and quinonoid), and other solution conditions. Uses SHIMADZU UV SPECTROPHOTOMETER UV-1800 to measure the optical absorption of Eosin Y. Samples are prepared for normal illumination of the specimen in 1 cm quartz cells of pathologic duration with absorption below 0.1 at excitation, and of all radiation wavelengths, and to prevent internal filter action. The absorption peak was measured to be 517 nm.

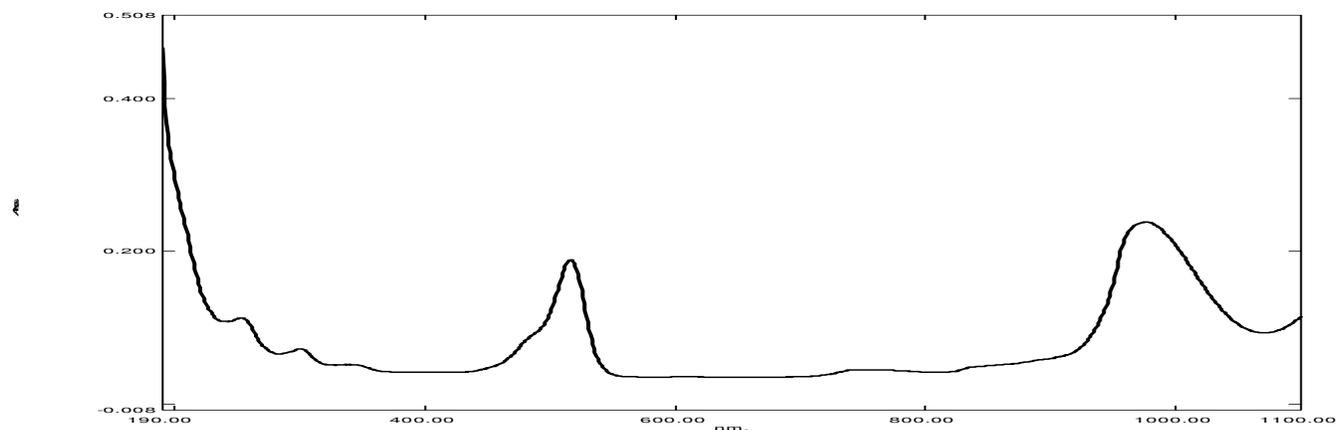


Fig. 2 : Photoabsorption spectrum of Eosin Y in basic ethanol.

### Characterization of rice husk

Figure 2 shows the rice husk analytical spectrum FTIR. Infrared spectrums have been obtained using a diffuse reflective supplement with a Fourier transform spectrophotometer (IRPrestige-21, SHIMADZU, Japan). Typically, a dried specimen of 0.006 g used in these analyses. A spectrum of between 4000 and 400  $\text{cm}^{-1}$  with a resolution of 1  $\text{cm}^{-1}$  was observed in the medium IR range. It was also found that there is a peak was observed at 2900 to 3750  $\text{cm}^{-1}$  with a slight volume of about 3400  $\text{cm}^{-1}$ , which indicates the presence of free OH and Si-OH groups, hydrogen-bonded. This is due to the quantities of silanol (Si-

OH) as well as the adsorbed water at the surface area (about 3400  $\text{cm}^{-1}$ ) Abou-Mesalam (2003). In the focus area of 1625  $\text{cm}^{-1}$ , which indicates the presence of an aldehyde and ketone CO band. The peak band of 1625  $\text{cm}^{-1}$  can also be caused by conjugated carbonyl groups with hydrocarbon. Furthermore, at 1512  $\text{cm}^{-1}$  the sharp peak indicates the presence of primary amides and implies C-N retention and N-H curvature. The symmetrical- $\text{NO}_2$  band extension can also be attributed to the flat top at 1512  $\text{cm}^{-1}$ . Band of rice husk in a region of 1250-1000  $\text{cm}^{-1}$  was considered peaks in a region of approximately 1053  $\text{cm}^{-1}$ , and the Si-O bond in siloxane (Si-O-Si) groups was realized as a result of this. Sharma *et al.* (2010). The bands around 1512  $\text{cm}^{-1}$  in TRH are also

attributed to carboxylic carbonate structural structures, thereby the RH-cellulose from rice husk is very similar to that of – cellulose in the FTIR spectrum. At a peak of  $1072.42\text{ cm}^{-1}$  an intense band corresponds to the extending vibrations of tetrahedrons of silicon-oxygen as ( $\text{SiO}_4$ ). The superposition of the extended vibrating of the COH bond in the  $1250\text{-}1000\text{ cm}^{-1}$  interval and the extended vibrations of the Si-O bond probably led to the high intensity of this pitch. Due to the bending movement of siloxane waves, the absorption peak of  $783\text{ cm}^{-1}$  was observed.

The dry rice husk and pistachio nutshell were crushed using a crushing mill. The resulting recovered product was washed several times with distilled water to eliminate water-soluble impurities and then dried in an oven at  $105\text{ }^\circ\text{C}$ . 500 g of the small pieces from the pistachio, nutshell was placed in a horizontal furnace in an inert atmosphere at  $1000\text{ }^\circ\text{C}$  for 3 hours. It was then removed from the furnace and cooled in a desiccator. After cooling, the rice husk and the activated pistachio nutshell was subsequently subjected to a homogenization treatment using a three-dimensional shaker for 1 hour.

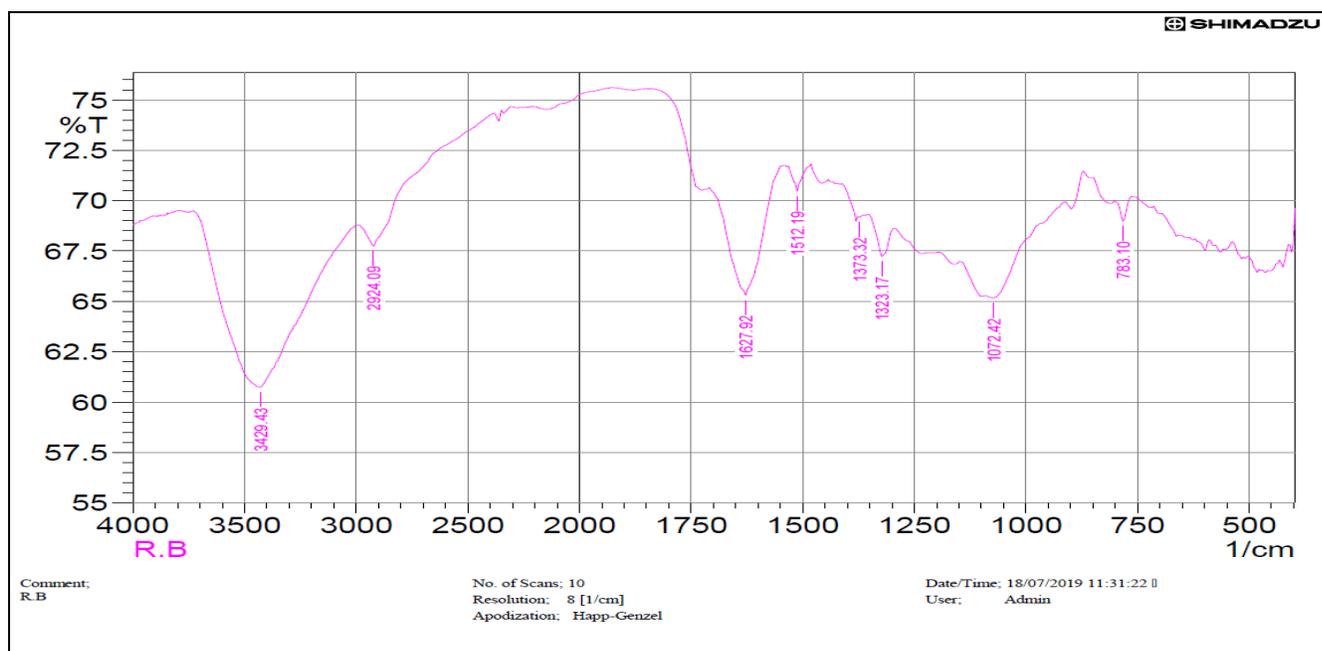


Fig. 2 : FT-IR spectrum for rice husk

### Effect of initial concentration

The effect of initial concentration had been studied to determine the change of percentage dye removal. Six types of solutions of 100 ml for Eosin dye were prepared with different concentrations of 1, 5, 10, 20, 30, 40, and 50 ppm and the optimized contact time, dosage and pH from experiments. The applied shaking speed was carried out at 200 rpm. The highest removal percentage was indicated at 1 ppm concentration with a denoted value of 99%, as shown in Figure 3.

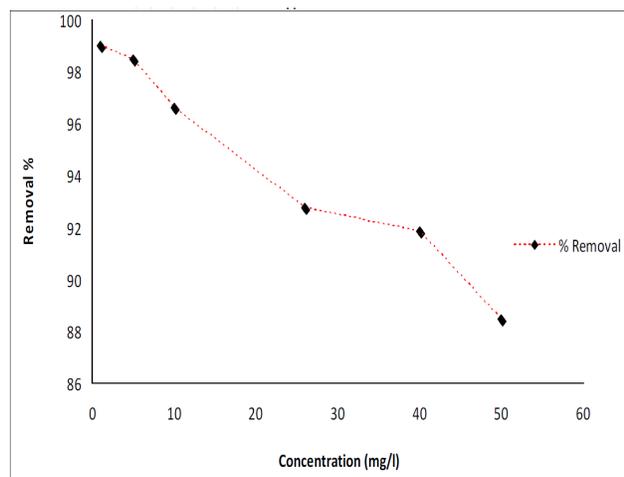
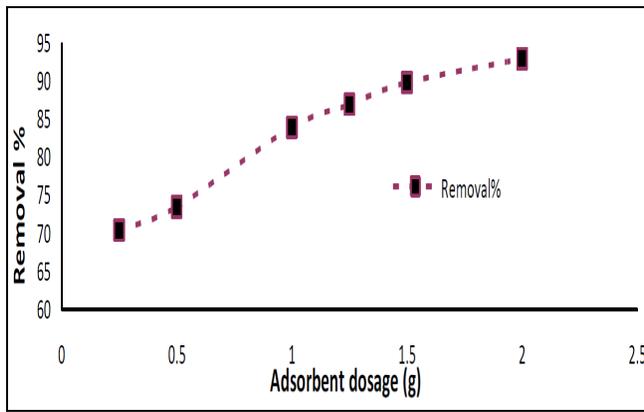


Fig. 3. Effect of initial concentration on adsorption of eosin dye.

As can be seen in the figure, IR spectra of rice husk in the region  $1200\text{-}1000\text{ cm}^{-1}$  were considered to result from superposition of vibrations of the C-OH bond and Si-O bond in the siloxane (Si-O-Si) groups. An intense band at  $1053\text{ cm}^{-1}$  corresponds to the stretching vibrations of silicon-oxygen. High intensity of this peak was probably due to superposition of the stretching vibrations of the C-OH bond in the interval  $1200\text{-}1000\text{ cm}^{-1}$  and the stretching vibrations of the Si-O bond. The absorbance peak at  $442\text{ cm}^{-1}$  was due to the bending vibration of the silo

### Effects of the amount of adsorbent

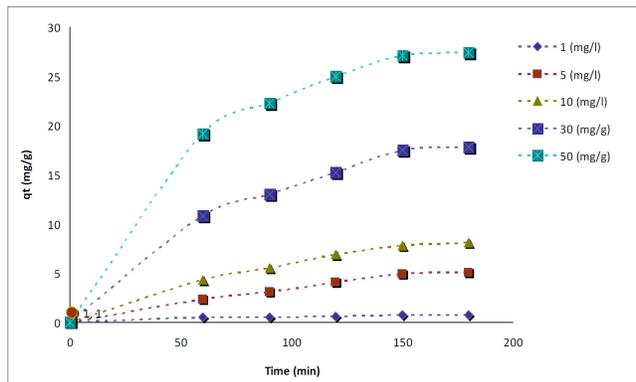
Figure 4 illustrates the percentage of dye removal in wastewater samples at a pH value of 2 for adsorbent dosages of 0.25-2 g. The experiment was conducted at room temperature and the time of contact was 130 minutes. It was found that the amount of waste disposal was initially low, and tend to increase with lower adsorbent dose. This was because the adsorbent's active site initially struggled to reach an adsorbate effectively as there was lower adsorption. It was also revealed that the optimum dosage of rice husk is 2 g to remove the maximum amount of dyes, nevertheless, the average level of exclusion is nearly 99%. However, the extraction percentage decreased over 2 g of the rice husk dose Shirsath and Shrivastava (2012), Maruca *et al.* (1982), Attia *et al.* (2003), Dursun (2003), Kim and Rajapakse (2005), and Marthur, Narang (1990).



**Fig. 4 :** The effect of adsorbent dosage for the removal of Eosin dye; when the pH is 2,  $C_0$  is 1 mg/l, and 130 min as contact time.

#### Effects of dye-adsorbent contact time

The duration of the experiment was in the range of 60 min to 180 min within an interval period of 30 min. The dye sample was taken separately and tested at room temperature under controlled operating conditions of 1 ppm, a pH of 2, and a mass of 1 g. The result showed that the rate of dye removal was very low at the early stage of the experiment because of the inappropriate interaction between the adsorbent and dye in wastewater. In addition, the percentage of dye removal increased linearly as contact time increased. The extraction percentage was found to be highest at a contact time of 180 minutes and the superiority was approximately 99%. The contact of the 180 min was the optimum time that the equilibrium is achieved. Nevertheless, the deletion proportion decreased with time increased after 180 minutes of contact time Dutta *et al.* (2002), and Majeti, Kumar (2000).

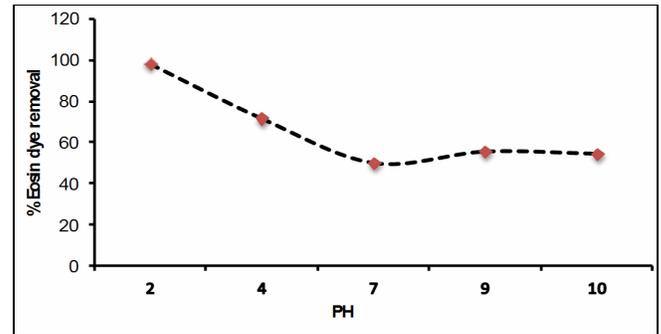


**Fig. 5 :** The adsorption versus contact time of eosin dye

#### Effect of PH

The pH of the solution represents an important operating factor, a 1 mg/l aqueous solution of dyes was treated with an adsorbent dose of 1 g at 130 minutes with taken into the consideration the disparity of pH in the range of 2-10. At a specific pH considered the optimum pH for a dye is 2, the maximum extraction effectiveness is achieved. The highest value of adsorption was achieved at the acidic pH value of 2, while at the basic pH of 10, the minimum value was recorded. The pH has been preserved with the assistance of 0.1M NaOH and 0.4M HCl. Through increasing and/or decreasing the pH of the solution, the efficiency of pollutant extraction decreases. This is because the eosin dye molecules become nucleophilic at higher pH (basic pH) which results in less adsorption on the nucleophilic sites of

the RH. Similar results have been documented in literature other investigators Lakshmi *et al.* (2009), Hameed *et al.* (2008) and (2009).



**Fig. 6 :** The removal dye of eosin versus the pH at a concentration of 1 mg/l.

#### Adsorption behavior

It is important to establish the most adequate link to balance curves to optimize the design of the adsorption system to remove dyes from aqueous solutions. The balance isotherms in this trial were applied to experimental data in order to describe the dye-RH relationship by two commonly employed isothermal models, namely, Langmuir and Freundlich. An analysis of the best model that explains Eosin's adsorption characteristics on rice husk is the Langmuir isotherm, besides that its spectacles the best match for Eosin dye's adsorption on rice husk. The equation of the Langmuir isotherm can be expressed as follows Kumar (2006), Islam *et al.* (2015), Mahmoud *et al.* (2016), Manna *et al.* (2017), Peláez Cid *et al.* (2013), Shakoor, Nasar (2016), and Verma (2008).

$$\frac{C_e}{q_e} = \frac{1}{q_{mb}} + \frac{C_e}{q_m} \quad \dots(3)$$

Where,  $C_e$  (mg/l) is the equilibrium concentration,  $q_e$  (mg/g) is the amount of adsorbate adsorbed per unit mass of adsorbate, and  $b$  and  $q_m$  are Langmuir constants, in which they are mainly related to adsorption capacity and rate of adsorption, respectively Langmuir (1918). The Langmuir adsorption plot for the studied dye on the rice husk is shown in Figure 8. From this isotherm, the Langmuir values  $b$  and  $q_m$  have been determined and their values are shown in Table 1. The following formula has also been used to measure a dimensionless constant separation function of Langmuir isotherm (LD) Kalyani and Gurunathan (2016):

$$R_1 = \frac{1}{(1 + K_L C_0)} \quad \dots(4)$$

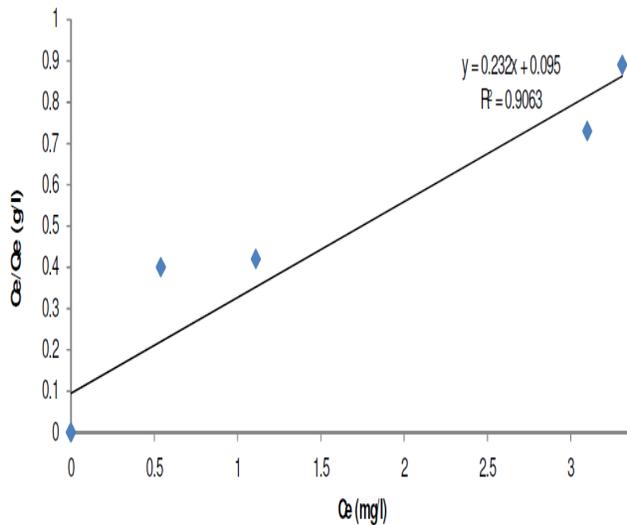
Whereas,  $C$  is the initial concentration of Eosin dye (mg/L) and  $K_L$  (l/mg) is the Langmuir adsorption constant given in Table 1. The adsorption of Eosin dye on the rice husk surface is promising since  $R_1$  is less than unity but greater than zero Deniz, Saugideger (2010). It is clearly to be noticed that the adsorption isotherm of eosin was found to conform with the Freundlich model, as displayed in Figure 6. The Freundlich isotherm Freundlich (1906) have represented as follows:

$$\log q_e = \log kf + 1/n \log C_e \quad \dots(5)$$

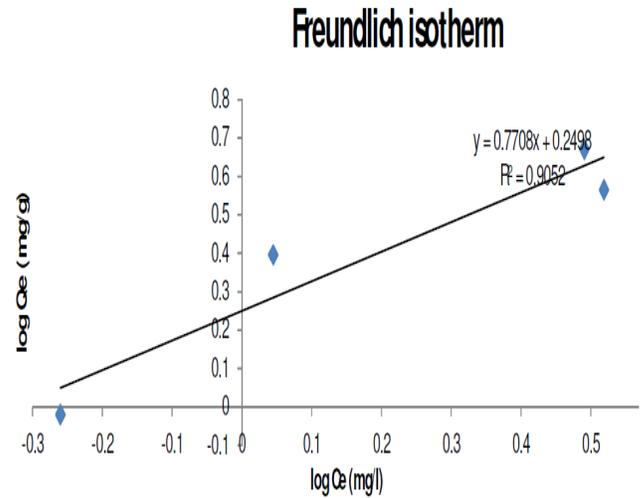
Where  $K_f$  and  $n$  are Freundlich constants incorporating all factors affecting the adsorption capacity and intensity of adsorption respectively.

**Table 1:** Isotherm parameters for removal of dye by RH.

Type Dye	Langmuir isotherm				Freundlich isotherm			
	Q max (mgg <sup>-1</sup> )	b (l/mg)	K <sub>L</sub>	R <sub>L</sub>	R <sup>2</sup>	K <sub>F</sub> [(mg <sup>-1</sup> g) (mgL <sup>-1</sup> ) <sup>n</sup> ]	N	R <sup>2</sup>
Eosin	4.31	0.095	2.44	0.291	0.906	1.777	1.297	0.905



**Fig. 7 :** Langmuir isotherm for the adsorption of Eosin dye on rice husk

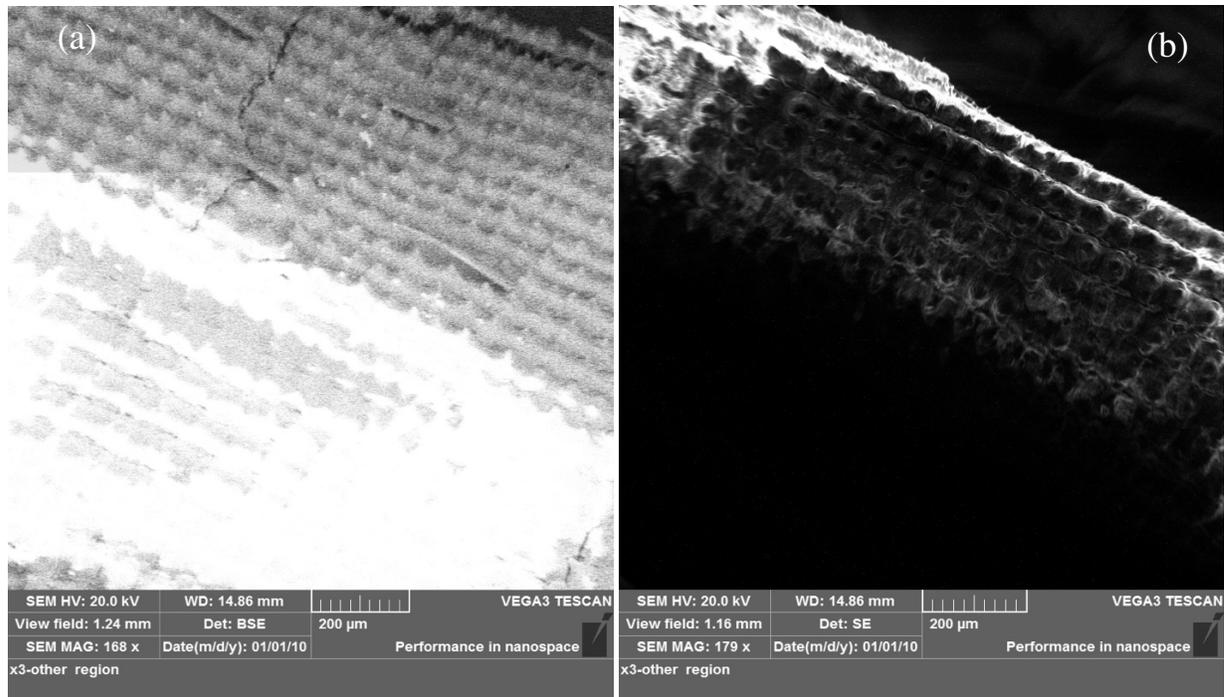


**Fig. 8 :** Freundlich isotherm for the adsorption of Eosin dye on rice husk

**Morphological behavior of RH**

The adsorbent surface morphology (RH) was presented by Scanning electron microscopy (SEM) and the obtained results are revealed beneficial information on the morphological texture of the adsorbent surface M.A. Garcia-

Morales *et al.* (2013). The SEM morphologies of RH before and after adsorption of the dye solution are presented in Figure 9. Based on the obtained micrographs, the RH particles are formed into two morphologies; engulfed or adsorbed on the surface of the dye molecules.



**Fig. 9 :** Scanning electron microscopy of Rice Husk: (a) Before adsorption (b) Eosin adsorption

The representative bands of the elements in the EDS spectra are as shown in Figure 10, in which it was revealed that the elements of potassium, calcium, nitrogen, and iron are not presented in the EDS spectrums. The observations in Figure 10b, on the other hand, indicate that the nitrogen and

potassium, which could have been released after the adsorbent process, however, the biomass surface before adsorption contains, C, O, Si, Al, and Cu, in which they are characterized as fibrous materials. The elements C, O, Si, N<sub>2</sub>, Al, P, and Mg are also found on this spectrum range.

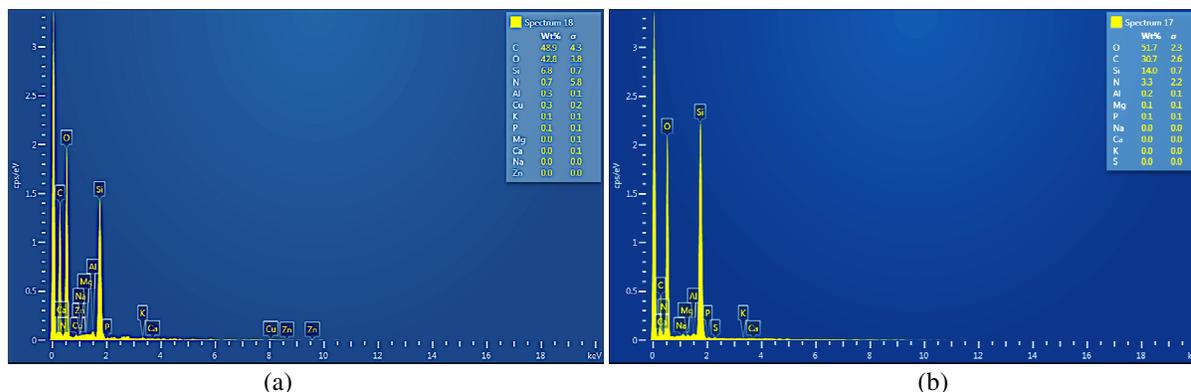


Fig. 10 : EDS pattern of rice husk for (a) before adsorption (b) after adsorption

### Conclusions

Simple equipment, easiness of service, low cost, lower time and reduced charging are characterized by adsorption. Aquatic solution dye removal was shown by analyzing water quality parameters like pH, pre-and post-treatment values. The results of this study are consistent with the fact that rice husk is an effective adsorbent for the removal of eosin color. Utilizing an adsorbent weight, contact time, pH, adsorbate concentration, UV-and IR spectrums recorded before and after the adsorption, the dyes have undergone some structural changes during procedures, thereby can be optimized for adsorption efficiency. It can be also concluded the use of the inexpensive RH adsorbent to remove eosin color from the aqueous solution is efficient. Furthermore, the rice husk obtained to study rice husk efficiency color adsorption displays that RH particles are either engulfed or adsorbed on the surface of dye molecules.

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