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Removal of acid yellow 17 dye from aqueous solutions using activated water hyacinth (*Eichhornia crassipes*)

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ABSTRACT

Dye-containing wastewater has been known as a serious environmental treat. There are many treatment methods, but they are not practical in many developing countries. In this study, batch adsorption of acid yellow 17 dye from aqueous solution has been carried out on a laboratory scale using activated water hyacinth root powder. The dye removal efficiency was measured using a UV-Vis spectrometer. Adsorption experiments were carried out in a batch process at different operating parameters including initial dye concentration, adsorbent dose, contact time, and solution pH. From the experimental data, the maximum dye removal efficiency achieved was 92.26% at pH 2, initial dye concentration 50 mg/L, contact time 120 min, and adsorbent dose 20 g/L. Experimental studies indicated pseudo-second-order kinetic ($R^2 = 1$) and Langmuir isotherm ($R^2 = 0.953$) models could better describe acid yellow 17 dye adsorption on the activated water hyacinth. Adsorption experiments revealed that activated water hyacinth prepared from locally available infesting water bodies had a high potential for removing acid yellow 17 dye from aqueous solution.

Key words: acid yellow 17 dye, activated water hyacinth, adsorption, textile wastewater treatment

HIGHLIGHTS

- Activated carbon was prepared from water hyacinth root using phosphoric acid.
- The removal efficiency of acid yellow 17 dye was evaluated using activated carbon at different operation conditions.
- Isotherm and kinetic studies of the removal of acid yellow 17 dye onto activated carbon from water was investigated.

GRAPHICAL ABSTRACT
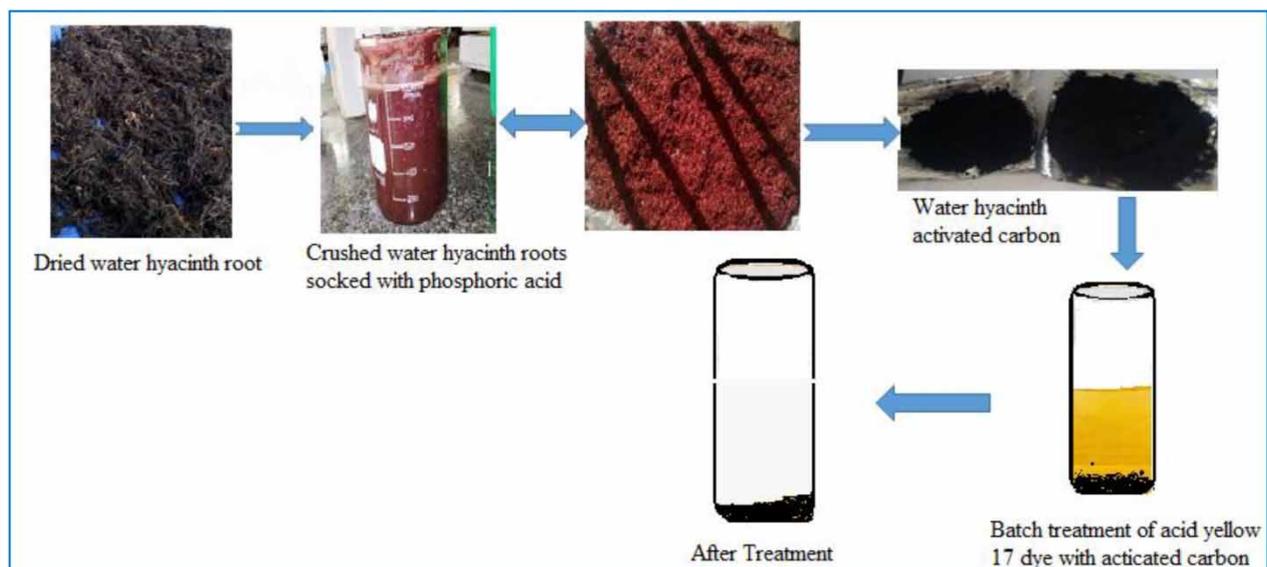
INTRODUCTION

In the world, numerous industries including textile, leather, plastics, paper, food, cosmetics, etc., use dyes and pigments to paint their products. The colored wastewaters of these industries are harmful to aquatic life in rivers and lakes, reducing light penetration and also enhancing extremely virulent metals in the environment (Ozacar 2002).

Dyes can be classified into natural and synthetic colorants. The synthetic dyes are characterized by their variety of colors and fastness, which makes them more widely used than natural dyes (Scholz 2019). Dyes can also be classified as cationic (basic) dyes, anionic (acid, direct, and reactive) dyes, and non-ionic (vat and disperse dyes) (Khan *et al.* 2018). Dye-containing wastewater is usually composed of dissolved solids, acids, bases, color, and toxic compounds. Color is noticeable at very low concentrations. Therefore, it needs to be treated before it is discharged to the environment.

The various methods for dye removal can be classified into three categories including biological, physical, and chemical methods such as chemical oxidation, electrochemical oxidation, photo degradation, biological treatment, membrane filtration, ion exchange, adsorption, precipitation, and coagulation (Ferrarini *et al.* 2016). Among these wastewater treatment techniques, adsorption is superior to other techniques for water reuse in terms of initial cost, flexibility, and simplicity of design, ease of operation, and insensitivity to toxic pollutants (Alemu *et al.* 2019). It also does not result in the formation of harmful substances (Mousa & Taha 2015). Adsorption is a natural process by which molecules of a dissolved compound collect on and adhere to the surface of an adsorbent solid. Commercially available activated carbon is effective in removing various types of contaminants (li *et al.* 2007; Owalude & Tella 2016), but it costs a lot and there is a continuous search for low-cost adsorbents prepared from agricultural or industrial wastes such as rice husk, sawdust, coir pith, wheat husk, ash, egg shell, orange fruit peels, peas waste, cassava peel, and eucalyptus bark (Ashraf *et al.* 2013; Ukanwa *et al.* 2019).

Acid yellow 17 (AY-17) dye is a reactive and anionic dye having a general structure shown in Figure 1. It is usually used in the textile, printing, soap, detergent, and cosmetic industries (Jedynak *et al.* 2019). It can damage the respiratory, nervous, and cardiovascular systems in humans and animals. Its thermal degradation can produce oxides of nitrogen and carbon that are toxic to living organisms. Moreover, it can cause mutagenic and tumorigenic effects in yeast, bacteria, and mammals (Choy *et al.* 2000).
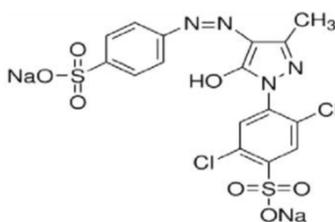

Figure 1 | Chemical structure of AY-17 dye.

Water hyacinth is a free-floating plant on aquatic bodies. It also grows in marshy areas in abundance. It is an exotic species with rapid and invasive proliferation resulting in choking of water bodies. This plant is very common in East African countries including Kenya, Uganda, and Ethiopia. It has been studied as an important biosorbent for the treatment of dyes, trace metals, and other physicochemical parameters (total suspended solids, total dissolved solids, biological oxygen demand, and chemical oxygen demand) (Nath *et al.* 2014; Priya & Selvan 2017; Yi *et al.* 2019).

The aim of this study was to evaluate the adsorption of AY-17 dye from aqueous solutions using activated water hyacinth (AWH) root under different operational conditions, such as adsorbent dose, pH, contact time, and initial dye concentrations. The equilibrium isotherm and adsorption kinetics models of AY-17 dye onto the AWH were also investigated.

MATERIALS AND METHODS

Materials

Powder of AY-17 dye (dye content 60%, chemical formula: $C_{16}H_{10}Cl_2N_4Na_2O_7S_2$), H_3PO_4 ($\geq$ 85%, Merk), HCl (37% concentration, Fisher Chemical), NaOH (98%, Fine Chemical Industries) used to adjust the pH of the

solution during batch adsorption experiment), distilled water for the solution preparation and rinsing purposes. All the chemicals used were of an analytical grade. Water hyacinth root (collected from Lake Tana) was used for the preparation of activated carbon. KBr pellet was used for FT-IR spectra development of the adsorbent.

Preparation of activated carbon

Water hyacinth was collected from Lake Tana, Ethiopia. The stem parts were cut off and the roots were collected and then washed thoroughly with distilled water to remove all the earthly impurities. Then it was dried in the open air for 6 days. The dried roots were crushed by using mortar and pestle. The crushed samples were chemically activated with mol L^{-1} phosphoric acid ($\geq$ 85%) for 24 hours and were dried by using an oven (PH 030A) to the next carbonization step. The activation/carbonization was performed at 600 °C for 2 hours using a furnace (Nabertherm B180, Germany). The activated samples were then washed with distilled water repeatedly to remove the acid from the surface of activated carbon, then the samples were oven-dried at 105 °C and grinded and sieved between 1 and 1.7 mm mesh and stored in clean containers.

FT-IR and SEM analysis of water hyacinth root

A Fourier transform infrared spectrometer (Spectrum 65 FT-IR, PerkinElmer) was used to illustrate the functional group of activated carbon made from water hyacinth before and after adsorption. It was recorded in the mid infrared region (4,000–400 cm^{-1}) using a pressed KBr pellet technique. SEM (SEM – JEOL, JSM 6360 LV) was used to investigate the surface morphology of the AWH adsorbent using a voltage of 15 kv and 1–3 nA beam current.

Batch adsorption experiments

One gram of the AY-17 dye powder was dissolved in distilled water to prepare the stock solution (1,000 mg/L), which was kept in dark-colored glass bottles. This dye stock solution was used to prepare intermediate and working solutions for batch adsorption experiments.

Batch experiments were conducted in a series of 250 mL jar containers. In all of the experiments 100 mL of AY-17 dye solution was added and agitated by using a rotary incubator shaker at 200 rpm (Excella E-24 Model). The pH of the solution was adjusted with 0.1 mol L^{-1} HCl or 0.1 M NaOH before adding the adsorbent. It was measured using an electronic pH meter (Jenway 430 Model). The experiments were carried out at room temperature.

To evaluate dye removal efficiency and capacity of the adsorbent optimization experiments were done. The effects of adsorbent dose, contact time, pH, and dye initial concentrations were investigated by varying any one of the process parameters and keeping the other parameters constant. All the experiments were performed in duplicate and the mean values were reported to investigate the influence of contact time (10–150 min), pH (2–11), adsorbent dosages (1–6 g/L), and initial dye concentration (50–300 mg/l). In this study 100 mL of the AY-17 dye solution with different concentrations (50, 100, 150 200, 250, and 300 mg/L) and a known amount of adsorbent were added into each jar at different conditions. It was shaken continuously at a constant oscillation at room temperature. Then the solution was filtered by using Whatman No. 41 filter paper (0.45 μm pore diameter). The amount of dye that might be retained in the filter paper was not taken into consideration. The removal efficiency of the dye was analyzed using a UV-VIS spectrometer at an adjusted wavelength of 402 nm.

The percentage of removal of AY-17 dye was determined using Equation (1).

$$R(\%) = \frac{(C_o - C_e)}{C_o} \times 100 \tag{1}$$

The amount of dye adsorbed, at time t (q_t) and at equilibrium (q_e), are shown below:

$$q_e = \frac{V(C_o - C_e)}{M} \tag{2}$$

$$qt = \frac{V(C_o - C_t)}{M} \tag{3}$$

where, q_t and q_e denote the amounts of AY-17 dye adsorbed (mg/g) at time t and at equilibrium, respectively. C_o, C_e, and C_t are the concentration of the dye in the solution (mg/L) at the initial, equilibrium and at time t, respectively; M is the mass of the adsorbent (g) and V is the volume of the solution (L).

RESULTS AND DISCUSSION

FT-IR analysis of AWH

The FT-IR spectra of AWH before and after adsorption of AY-17 dye is shown in Figure 2. The FT-IR spectrum of AWH was recorded in the range of 400–4,000 cm^{-1} helped to identify the functional groups on the material surface. These functional groups help to bind the dye molecules onto its surface (Figure 2(a)). The appearance of a peak around 3,418 cm^{-1} represents OH stretching vibrates suitable for inter-and intra-molecular hydrogen bonding of polymeric compounds such as carboxylic acids, alcohols, or phenols as in hemicellulose, and cellulose (Samiey & Ashoori 2012). The peak observed at 2,371 cm^{-1} owing to $C = O$ stretching vibration of the carboxylic acid. A signal produced at 1,600 cm^{-1} is attributed to the C-C stretch vibration of carboxylates (Liu *et al.* 2006; Sundari & Ramesh 2012). The bands observed at 1,094 and 1,160 cm^{-1} indicates C-O stretch vibrations of an ester carboxyl acids, phenols, and alcohols (Segneanu *et al.* 2012). In this range the peaks appeared may be the presence of phosphate groups in the AWH sample, such as $P = OOH$ (hydrogen bonded, $P+ -O - ($in acid phosphate esthers), P-H (in phosphine group) (Zhang *et al.* 2017). The FT-IR band at 500 and 766 cm^{-1} indicates $= C$-H bending vibrations. After adsorption, a shift was observed on the characteristic peaks of FT-IR spectra, which indicates the biosorption of AY-17 dye onto the AWH surface (Figure 2(b)).
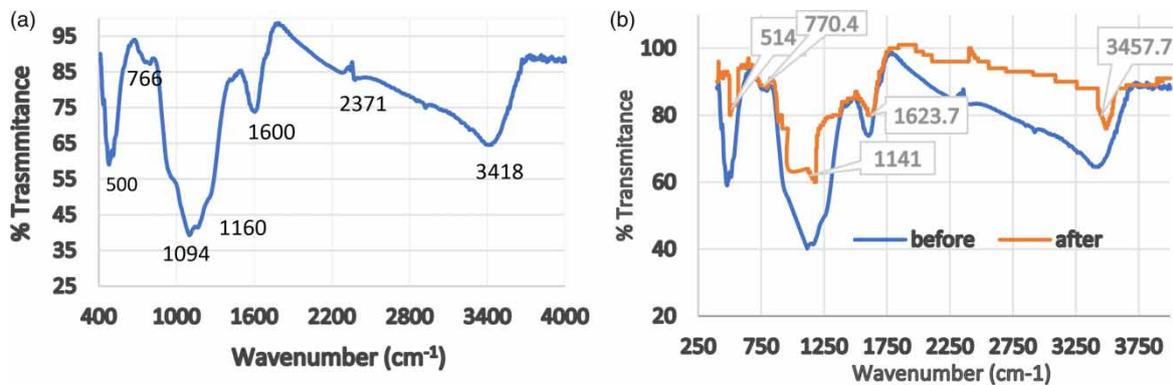

Figure 2 | FT-IR spectra of AWH before (a) and after (b) adsorption of AY-17 dye.

Scanning electron microscopy (SEM)

SEM is widely used to study the morphological feature and surface characteristics of the adsorbent material (Elkady *et al.* 2015). In this study, the surface structures of AWH root before and after activation were analyzed using SEM as shown in Figure 3(a) and 3(b), respectively. The scanning electron micrographs enable the direct observation of the surface microstructures of the adsorbent (Nwankwo *et al.* 2018). The micrographs of the AWH showed more ridged pores than the non-activated water hyacinth. This might be the micropore formation via the insertion of P-O-P from H_3PO_4 used for activation into the C lattice in water hyacinth root (Zhao *et al.* 2017).
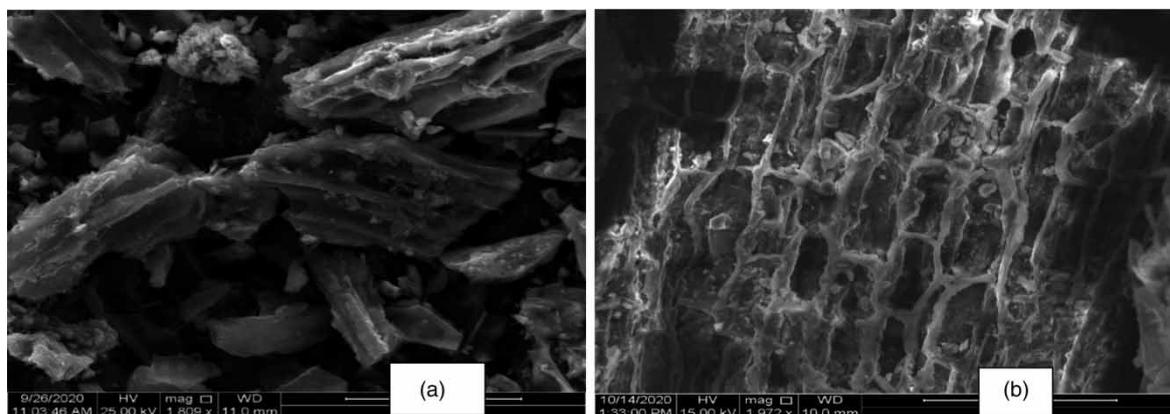

Figure 3 | SEM image of water hyacinth root before activation (a); SEM image of activated water hyacinth root (b).

Thus, it indicates a better possibility of the acid yellow dye to be adsorbed onto the surface AWH (Figure 3(a)) than the non-activated biomass of water hyacinth root (Figure 3(b)).

Effects of pH

The pH of the system can affect the adsorptive uptake of adsorbate molecules most probably due to its influence on the surface properties of the adsorbent and ionization or dissociation of the adsorbate molecule (Santhi *et al.* 2010). The effect of pH on the adsorption of AY-17 dye on AWH is shown in Figure 4. It was observed that pH influences the AWH surface dye-binding sites and the dye chemistry in water. The highest dye removal efficiency was observed at pH 2 (92%). The figure shows the adsorption of AY-17 dye on AWH root was decreased to 91.2%, with increasing pH to 11. This value is in agreement with the effect of pH on the removal of Congo red dye on the surface of halloysite-magnetite-based composite (Ferrarini *et al.* 2016). This indicates the effect of pH on the change of surface property is low. The removal of AY-17 dye may be based on the reactions of different functional groups on the dye (-OH, $-N = $N-, two anionic parts) with the different functional groups ($-C \equiv C$-H, $-C = O$, -C-O, -OH) on the AWH. The presence of ligands on the dye helps to react with the surface of AWH both in acidic and basic conditions forming a complex that helps for the removal of the dye in aqueous solution.
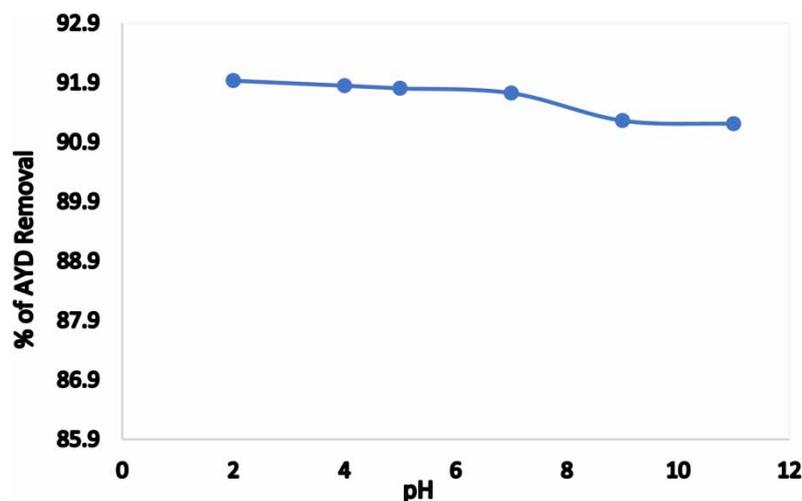

Figure 4 | Effect of pH on removal of AY-17 dye on AWH (Initial concentrations: 50 mg/l; adsorbent dose: 20 g/L; contact time: 120 min).

Effects of adsorbent dosage

The effect of adsorbent dose on adsorption was studied using different adsorbent doses in the range of 1–6 g. An increase of adsorbent dose from 1 to 4 g in 100 mL of dye solution, % of removal of AY-17 dye on AWH was increased from 75.9% to 90.37%. The reason behind increasing the adsorption with increasing adsorption dose from 10 to 60 g/L is the greater availability of adsorption sites for the AY-17 dye solution. Figure 5 shows that the dye removal percentage was very low at the beginning. But this increased with increasing adsorbent dose. The maximum removal efficiency of AY-17 dye onto the surface AWH was 94.2%. After this dose, the increment of dosage doesn't show any significant change in the percentage of removal of the dye.

Effects of contact time on dye adsorption

The results from experimental data indicated that the percentage of removal of AY-17 dye increased with the contact time. The effect of contact time on the percentage of AY-17 dye removal from synthetic solution is shown in Figure 6. The observed contact time was in the range between 10 and 150 minutes. In the first 30 min of contact time the rate of reaction was fast or the percentage removal of the AY-17 dye was rapid and thereafter it proceeds slowly and finally attains saturation at 120 min.

Effect of initial dye concentration

The effect of initial AY-17 dye concentration in the range 50–300 mg/L onto the AWH adsorbent was studied. As it can be seen from Figure 7, the percentage of dye removal decreased with an increase in an initial dye concentration. This might be due to the saturation of the available active sites on the AWH adsorbents. At a pH of 2, the
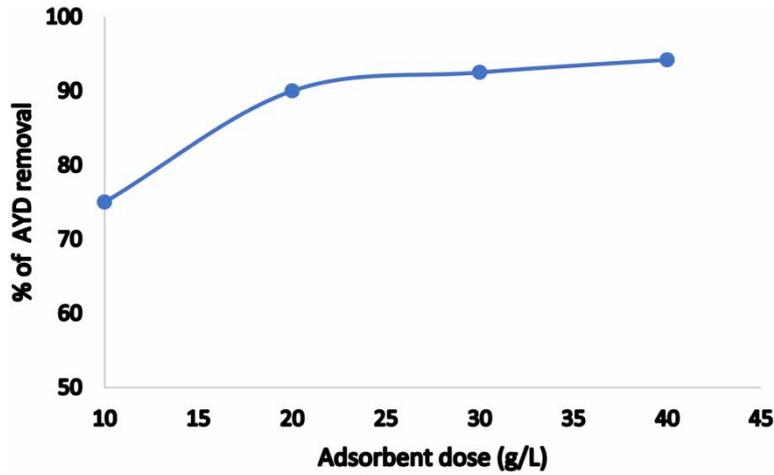
Figure 5 | Effects of adsorbent dose on the removal efficiency of acid yellow dye on AWH (Initial dye concentrations 50 mg/L; contact time 120 min and pH 2).
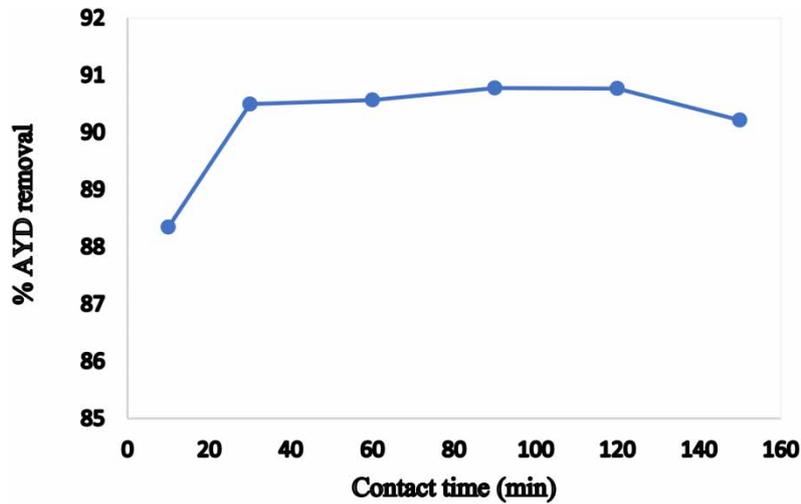

Figure 6 | Effect of contact time for AWH on AY-17 dye removal. (Initial concentrations: 50 mg/L; adsorbent dose: 20 g/L and pH 2).
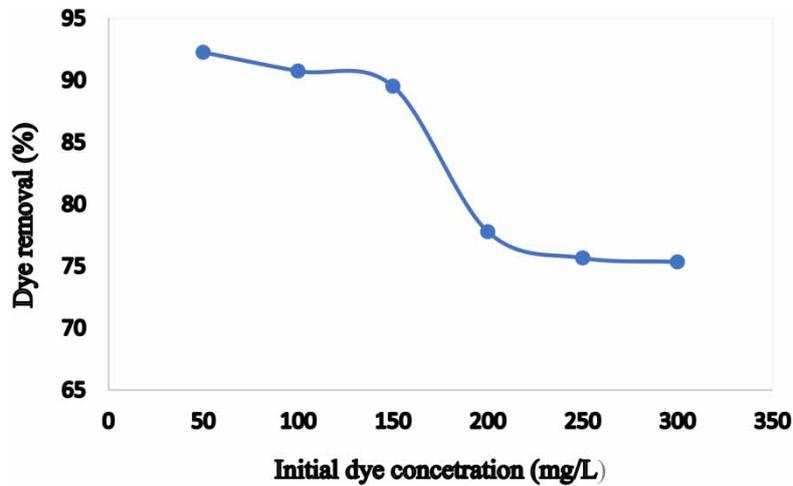

Figure 7 | Effect of initial dye concentration on the removal of acid yellow dye with AWH (adsorbent dose 2 g/L; contact time 120 min, and pH 2).

percentage of AY-17 dye removal decreased from 92.26 to 75.34% as the initial dye concentration increased from 50 to 300 mg/L.

Adsorption isotherm studies

The relationship between the amount of a substance adsorbed at constant temperature and its concentration in the equilibrium solution is termed as the adsorption isotherm. To optimize the design of dye adsorption system, it is important to establish the most appropriate correlations of the equilibrium data of each system. There are two most commonly used isotherm models to analyze equilibrium data of solute between adsorbent and solution. These are the Langmuir and the Freundlich isotherm models. The parameters obtained from models provide important information on the adsorption mechanisms and the surface properties and affinities of the adsorbent.

The Langmuir model is a monolayer adsorbed surface that assumes all sites have identical affinity towards the adsobate. Each adsorbed molecule on the surface of the adsorbent has the same adsorption activation energy. The Langmuir isotherm equation (Langmuir 1916) is given by:

$$\frac{C_e}{q_e} = \frac{1}{K_L\, q_{max}} + \frac{C_e}{q_{max}} \tag{4}$$

where K_L is the Langmuir adsorption equilibrium constant and q_{max} is the maximum adsorption capacity at saturation of the monolayer. The plot of C_e/q_e versus C_e enables the determination of isotherm constants.

The Langmuir isotherm is also used to calculate the dimensionless constant known as separation constant or equilibrium parameter (R_L), which gives information about the favorability of adsorption of the adsorbate on the adsorbent.

$$R_L = \frac{1}{K_L C_o + 1} \tag{5}$$

where, C_o (mg/L) is the initial concentration of the AY-17 dye. The R_L values indicate the type of adsorption: $0 < R_L < 1$, favorable; $R_L = 1$, linear; $R_L > 1$ unfavorable and $R_L = 0$, irreversible (Malik 2003).

The correlation coefficient (R^2) from the linear Langmuir isotherm model is 0.953 (Figure 8(a)). This shows that the Langmuir isotherm fits the experimental data very well and confirms the monolayer coverage of the dye onto the AWH ($q_{max} = 11.8$ mg/g) and also the homogeneous distribution of active sites on the adsorbent.
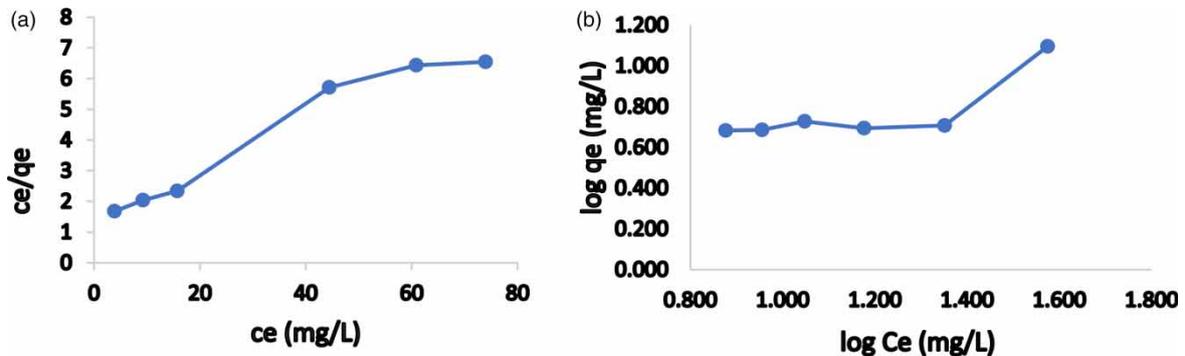

Figure 8 | Langmuir (a) and Freundlich (b) adsorption isotherms for adsorption of AY-17 dye onto AWH (pH 2; adsorbent dose 20 g/L; contact time 120 min; and initial dye concentration 50 mg/L).

The Freundlich isotherm model describes the adsorption of the solutes from a liquid to a solid surface. The Freundlich equation expresses adsorption on a heterogeneous surface using the following equation (Freundlich 1906).

$$q_e = K_F\, C_e^{\frac{1}{n}} \tag{6}$$

By taking the logarithmic function of the Equation (6), the linear equation of Freundlich isotherm is represented as:

$$log q_e = log K_F + 1/n\, log C e \tag{7}$$

where K_F is the Freundlich isotherm constant (mg/g) related to adsorption capacity and $1/n$ is Freundlich exponent related to adsorption intensity.

The Freundlich model was used to evaluate the adsorption of AY-17 dye onto AWH. The linear plot of the Freundlich model log q_e vs. log C_e showed a correlation coefficient (R^2) of 0.6266 (Figure 8(b)). The KF and $1/n$ values are obtained from Equation (7) were 1.563 and 0.491, respectively. This indicated AWH produced a favorable condition for adsorption of AY-17 dye on its surface (Mallampati & Valiyaveettil 2013).

Comparing the two isotherm models, it was found that the data were better fitted with Langmuir isotherm model ($R^2 = 0.953$) than the Freundlich isotherm model ($R^2 = 0.6266$) (Table 1). The R_L value of this study was 0.038 which is between $0 < R_L < 1$. This indicates that the adsorption process of AY-17 dye onto AWH root powder is favorable.

Table 1 | Langmuir and Freundlich isotherm model constants for adsorption of AY-17 dye onto AWH

Adsorbent	Langmuir isotherm				Freundlich isotherm		
	R^2	q_m (mg/g)	K_L (L/mg)	R_L	K_F (mg/g)	$1/n$	R^2
AWH	0.953	11.79	0.085	0.038	1.563	0.491	0.6266

Adsorption kinetics

Adsorption kinetic models are used to investigate the mechanism of sorption and potential rate-controlling steps to select optimum operating conditions for the full-scale batch process. The kinetic parameters, which are helpful for the prediction of adsorption rate, give important information for designing and modeling the adsorption processes (Santhi *et al.* 2010).

Determination of best-fit kinetic models is the most common way to predict the optimum adsorption kinetic expression (Kumar & Sivanesan 2006). Pseudo-first-order and pseudo-second-order kinetic models were used to investigate our study.

Mathematically the pseudo-first-order model is expressed as:

$$\frac{dq_t}{dt} = K_1(q_e - q_t) \tag{8}$$

The linear form of Equation (8) is :

$$log(q_e - q_t) = \log q_e - \frac{K_1 t}{2.303} \tag{9}$$

where q_t is the amounts of adsorbate adsorbed (mg/g) at equilibrium and at any time t, and K_1 (min^{-1}) is the pseudo-first-order rate constant, which can be evaluated from the plot of $log(q_e - q_t)$ versus t.

Pseudo-second-order kinetic model is expressed mathematically as:

$$\frac{dq_t}{dt} = K_2(q_e - q_t)^2 \tag{10}$$

The linear equation form of Equation 10 is:

$$\frac{t}{q_t} = \frac{1}{K_2 q_e^2} + \frac{t}{q_e} \tag{11}$$

where K_2 is a pseudo-second-order rate constant (g/mg h) and obtained from the plot t/q_t versus t.

The kinetics of AY-17 dye adsorption onto AWH adsorbent were analyzed using pseudo-first-order and pseudo-second-order kinetic models in the study. The correlation coefficient (R^2) of the pseudo-first-order and pseudo-second-order kinetic models in this study are 0.6219 and 1.00, respectively (Table 2; Figure 9(a) and 9(b)).

Table 2 | Pseudo-first- and second-order parameters for the adsorption of AY-17 dye onto AWH

	Pseudo-first-order			Pseudo-second-order		
C_o (mg/ L)	K_1 (1/min)	q_e (mg/g)	R^2	K_2 (kg/mg. min)	q_e (mg/g)	R^2
50	0.057	2.269	0.6219	0.381	9.46	1.00
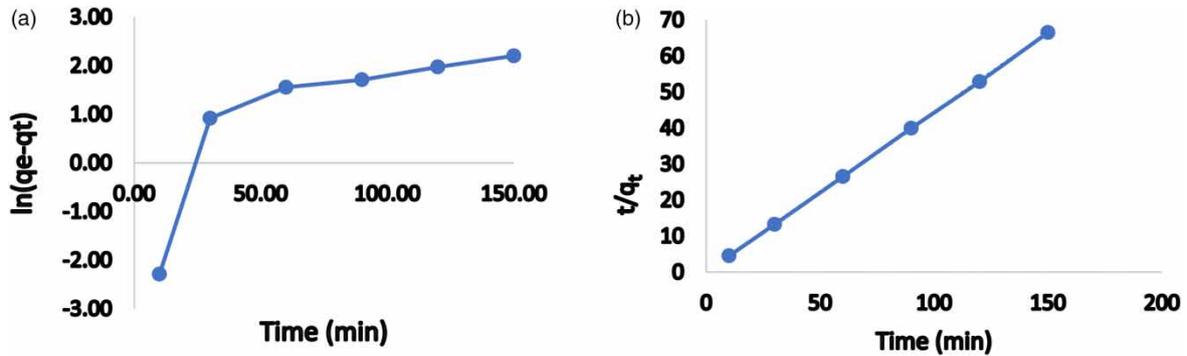
Figure 9 | The pseudo-first-order (a) and pseudo-second-order (b) plots of adsorption of AY-17 dye with AWH.

The calculated q_e value of pseudo-second-order kinetic model agreed well with the experimental data obtained in the lab. This implies that the pseudo-second-order kinetic model is best to describe the adsorption kinetics of AY-17 dye onto AWH.

The removal of AY-17 dye using AWH was compared with other adsorbents reported in literature. The removal capacities with their experimental settings are shown in Table 3. The AWH indicated good removing potential of AY-17 dye in aqueous solution. The availability of WH in tropical lakes and simple preparation might be important for both the treatment of AY-17 contaminated wastewater and management of invasive WH species on aquatic bodies.

Table 3 | Comparison of adsorption capacity of AY-17 dye onto AWH with other adsorbents including studied isotherm and kinetic models

Adsorbent	C_o (mg/ L)	pH	q_m (mg/ g)	Isotherm models	Kinetics model	References
Mesoporous carbon	30– 310	–	35–53	LM	PSO	Jedynak *et al.* (2019)
Persea Americana seed	300	2	42.7	LM	PSO	Munagapati *et al.* (2021)
Solanum melongena biomas waste activated carbon	15	5	93.545	LM	PSO	Kannaujiya *et al.* (2021)
Euterpe oleracea activated carbon	50	2	47.9	FM	PSO	de Oliveira Lopes *et al.* (2022)
Nephelium lappaceum peel activated carbon	50	2	49	LM	PSO	Njoku *et al.* (2014)
Granular sludge	40	2	13	TM	PSO	Gao *et al.* (2010)
AWH root	50	2	11.8	LM	PSO	This study

LM, Langmuir model; FM, Freundlich model; PSO, pseudo-second-order; TM, Temkin model.

CONCLUSION

This study was conducted to analyze the removal efficiency of the AWH adsorbent for the removal of AY-17 dye from textile wastewater using aqueous solutions. Batch studies were carried out under some limited operating parameters including pH, contact time, adsorbent dosage, and initial dye concentration. At optimum conditions,

AWH root showed a removal efficiency of 92.26% AY-17 dye from aqueous solution. The results obtained from this study indicated high removal efficiency of AY-17 dye in aqueous solutions. The material used for adsorption is obtained from the collected water hyacinth root around lakes, which is cheap and locally available. Therefore, the water hyacinth root activated carbon can be used as a potential sorbent to AY-17 dye in the textile wastewater to reduce its burden on the environment.

ACKNOWLEDGEMENTS

DATA AVAILABILITY STATEMENT

All relevant data are included in the paper or its Supplementary Information.

CONFLICTS OF INTEREST STATEMENT

The authors declare there is no conflict.

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