

Pyrolysis Biochar from Cellulosic Municipal Solid Waste as Adsorbent for Azo Dye Removal: Equilibrium Isotherms and Kinetics Analysis

Manu Agarwal, James Tardio, and S. Venkata Mohan

Abstract—Municipal solid waste (MSW) can have a negative impact on the environment and / or human health, if not properly managed. Though landfill is the most common method used to manage MSW, treating MSW using waste-to-energy (WtE) technologies is an option that is receiving increasing interest. Pyrolysis is a WtE technology that is capable of converting waste into products such as bio-gas, bio-oil and bio-char. Bio-char, which varies in composition based on the raw material pyrolysed, has the potential to be used as a fuel and as an applied material for catalysis or adsorption. In this work the removal of azo dye was investigated using bio-char from pyrolysis of a cellulosic municipal solid waste. The bio-char was characterised using Scanning Electron Microscopy and Surface Area Analysis. The influence of pH on the dye removal was investigated over the range 4-7. The experimental data was analysed using four non-linear kinetic models (Pseudo first-order, pseudo second-order, Weber intra-particle diffusion and Elovich equation) and four non-linear isotherm models (Langmuir, Freundlich, Temkin and Sips), and ranked based on the statistical analysis tools in origin software. The dye removal kinetics most closely followed pseudo-first order kinetics whilst the adsorption isotherms were most closely fitted by the Temkin model over the pH range studied.

Index Terms—Azo dye, bio-char, municipal solid waste, water treatment.

I. INTRODUCTION

Management of municipal solid waste (MSW) is a challenge for all societies. Though landfill is the most commonly practiced way of managing MSW, treating MSW to produce valuable products [1] is receiving increased interest. Of the treatment options available pyrolysis has been identified as having significant potential as it produces lower emissions than combustion and gasification [2]. One of the known value added products from pyrolysis of waste containing organic matter is bio-char. Bio-char has a wide range of applications such as: a soil conditioner [3]-[5], adsorbent [6], electric double layer capacitor [7] and boiler

fuel for steam [8]. The similarities between bio-char and the raw products that are used to produce activated carbon indicate that bio-char also has potential for use as a source for activated carbon. One of the main uses of activated carbon is in the removal of pollutants from waste water. Azo dyes, which are used in the textile industries, are a common pollutant found in waste water [9], [10]. Azo dyes are known carcinogens which have a mutagenic effect on both aquatic life and humans. This property of dye forces the need of its removal from the wastewater before being discharged into water bodies. Colour removal from textile effluents is therefore an area of significant interest. Adsorption processes have shown promise as a practical and economic process; especially for colour removal [11]. Textile dye colour removal by adsorption onto activated carbon has proven to be highly efficient and reliable. However although colour removal by adsorption onto activated carbon is highly efficient, the overlying cost of the activated carbon and associated problems of regeneration and reuse warrants search for alternative low cost adsorbents [12]. Various materials derived from industrial waste materials, agricultural wastes, minerals, coal, soil, etc. have been widely investigated in this direction [11]-[15]. In the present work activated carbon was prepared using bio-char generated from pyrolysis of cellulose based municipal solid waste (CSW). The focal objective of this research was to investigate the potential of CSW as a raw material for producing an adsorbent that can be used for the removal of azo dye from aqueous solutions.

II. MATERIALS AND METHODS

A. Materials

The CSW was collected from a canteen in Hyderabad, India. The waste was dried under direct sunlight for three days to remove the unbound moisture and then shredded. C.I. Acid Black 10B (4-amino-5-hydroxy-3-[(4-nitrophenyl)azo]-6-(phenylazo)-2,7-naphthalene disulfonic acid disodium salt; $C_{22}H_{14}N_6O_9S_2Na_2$; MW, 616.49; CAS No. 1064-48-8), an azo dye (Fig. 1) belonging to acid application class was used for the dye removal tests. KOH and HCl where laboratory reagents procured from AVRA Industries Pvt. Ltd., India and Rankem Fine Chemicals Ltd., India respectively.

B. Pyrolysis and Preparation of Adsorbent

1) Preparation of bio-char

Pyrolysis of CSW was conducted in a custom designed packed bed reactor [16] at 673 K – 1073 K for three hours.

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A fixed amount of sample was packed in the reactor and placed inside the heater. The reactor was sparged with 99.99% nitrogen gas to maintain inert atmosphere. The reactor was heated at a controlled rate (12°/min) to the specified temperature and maintained for three hours.

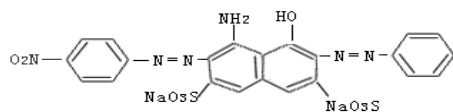


Fig. 1. Acid Black 10B azo dye.

2) Preparation of adsorbent

Bio-char was mixed with KOH in the ratio 1:4 by weight. The aforementioned mixture was placed in the reactor and fixed inside the heater. Pure nitrogen gas (99.99%) was passed through the reactor to maintain an inert atmosphere. The reactor was heated at a controlled rate of 10°/min to 1073 K and maintained for 5 hours thereafter. At the end of the process the reactor was allowed to cool and the resultant material was neutralized with 10% HCl. The material was washed with distilled water until the pH of the filtrate was 7.0. Finally, the prepared material (activated carbon (CSW-AC)) was dried overnight at 333 K.

C. Adsorption Experiments

The dye adsorption experiments were carried out in a batch reactor under the following conditions: T = 303 K; pH 4-7; activated carbon = 200 mg / 100 mL; acid black 10 B dye concentration solution 10 mg/l. The reaction vessel agitated using a shaking incubator (Labtech) at 120 rpm for one hour.

D. Analysis

SEM was conducted using a FEI Quanta SEM 200. Surface area was analysed using a Micromeritics ASAP 2010. Dye concentration was determined using a UV-VIS spectrophotometer (GE Genequant 1300).

III. RESULTS AND DISCUSSION

A. Characterization of Prepared Activated Carbon

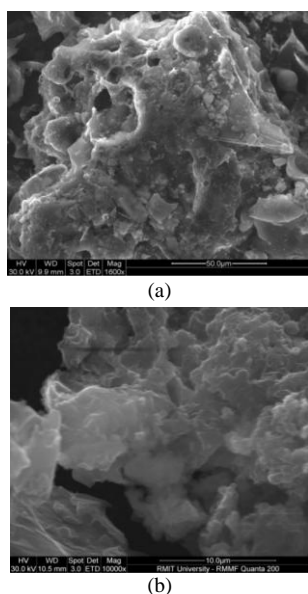


Fig. 2. SEM images of (a) Bio-char (b) Activated carbon.

The bio-chars produced at medium and high temperature were selected for studying the dye adsorption capacity. It was observed that the high temperature bio-char produced elemental sodium after treatment and so was not suitable for adsorption study. Hence, the bio-char produced from CSW at medium temperature was further studied. After the acid treatment (10% HCl) of the bio-char from CSW it was observed that the CSW-AC yield was 93.55% by wt. The SEM images of bio-char and activated carbon were recorded (Fig. 2) and micropore surface area of the samples were 150.67 and 1237.73 m²/g, respectively.

B. Investigations on the Removal of Azo Dye (Acid Black 10B)

Removal of Acid black-10B (azo dye) using activated carbon (prepared from bio char generated from kitchen vegetable waste) was investigated over a period of 60 minutes at four pH values (4, 5, 6 and 7). The adsorption profiles obtained for these tests are shown in Fig. 3. The adsorption rate was relatively high up to 20 min (pH 4: 0.093 mg/g/min; pH 5: 0.1 mg/g/min; pH 6: 0.125 mg/g/min; pH 7: 0.1 mg/g/min) and subsequently reduced with time (rates from 20 – 60 minutes were as follows: pH 4: 0.0781 mg/g/min; pH 5: 0.075 mg/g/min; pH 6: 0.0625 mg/g/min; pH 7: 0.075 mg/g/min). For the tests conducted in the acidic pH range 100% adsorption capacity was achieved in 60 min, whereas at neutral pH this was achieved in 50 min. The higher adsorption rate initially could be attributed to the participation of active sites/functional group present on the surface while the adsorption rate later decreased due to more and more occupancy of these active sites.

1) Sorption kinetics

Linear forms of different kinetic models (intra-particle diffusion model [17], [18], the pseudo-first-order kinetic model, the pseudo-second-order kinetic model [19]-[21] and the Elovich equation) based on the aqueous phase concentration of sorbate were used to evaluate the sorption mechanism at the pH values investigated.

a) Kinetic models

Sorption rate can be estimated based on the pseudo-first-order (Lagergren's equation) and pseudo-second order kinetic equations by assuming the dye-CSW-AC sorption process as a pseudo-chemical reaction [16], [21], [22] represented by equations 1 and 2, respectively.

$$dq_t/dt = K_1(q_e - q_t) \quad (1)$$

$$dq_t/dt = K_2(q_e - q_t)^2 \quad (2)$$

where, q_e (mg/g) and q_t (mg/g) are adsorption capacities at equilibrium and time t , respectively and K_1 (min⁻¹) and K_2 (g/mg/min) are the first and second order rate constants respectively. Pseudo first order and pseudo second order plots for CSW-AC showed a reasonably good fit using the experimental data obtained, with the 1st order fits being slightly better (Fig. 4). The first and second order rate constants (Table I) for the sorption of azo dye from the aqueous phase by CSW-AC were obtained by the slope of

the respective curves.

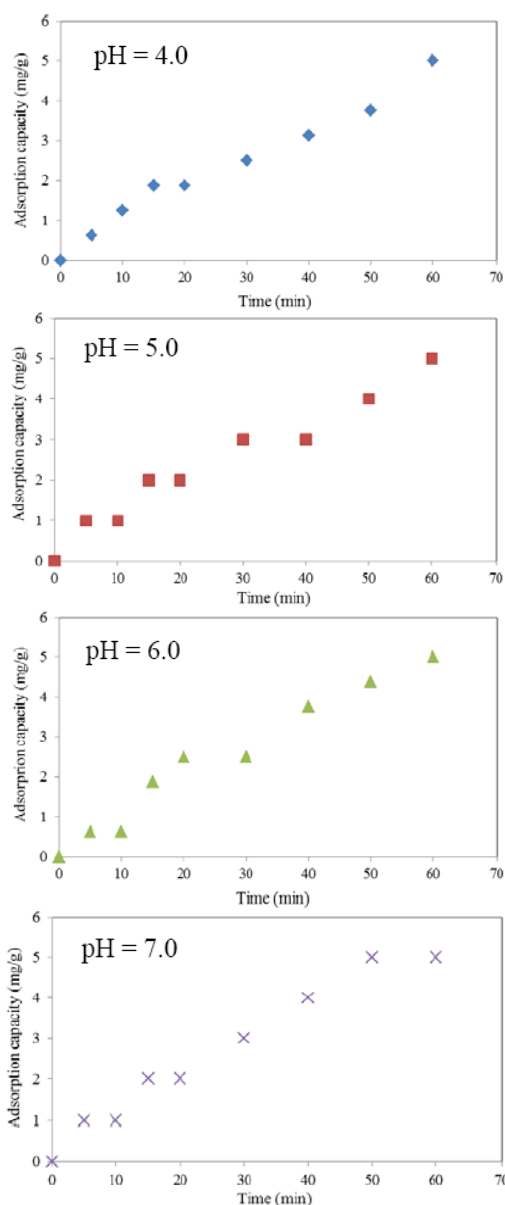


Fig. 3. Acid black 10 B dye sorption with CSW-AC as sorbent.

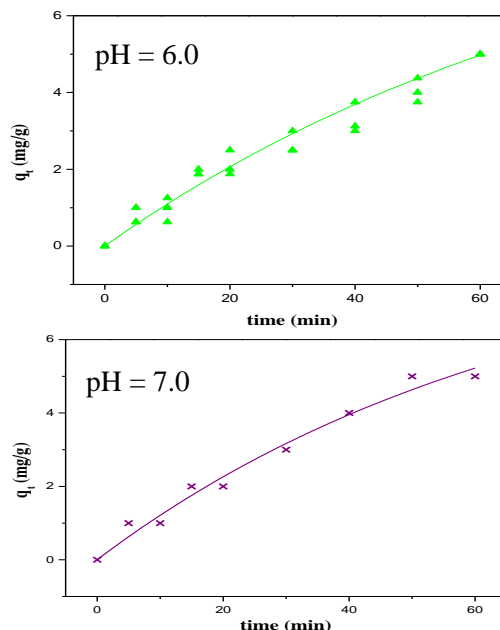
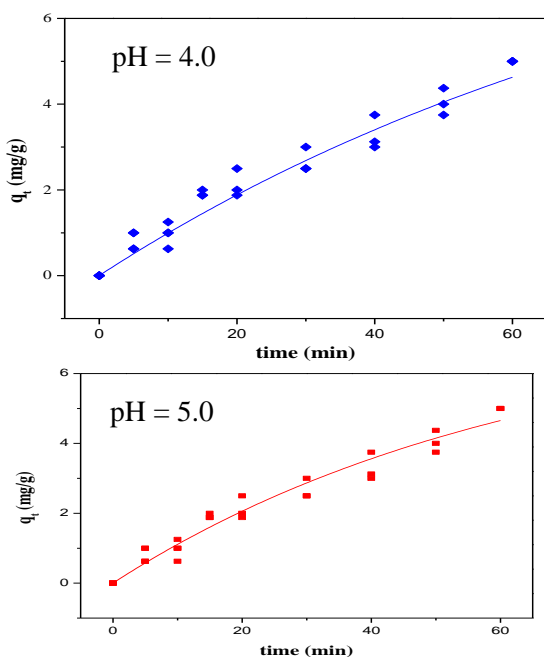


Fig. 4. Pseudo first order kinetic model.

b) Weber's intra-particle diffusion model

The intra-particle diffusion model is a single-resistance model derived from Fick's second law which can be expressed mathematically by equation 3 [11], [23].

$$q_t = K_p t^{0.5} \quad (3)$$

where, K_p ($\text{mg/g} \cdot \text{min}^{0.5}$) is the intra-particle diffusion rate constant. To fit the intra-particle diffusion model over the data the amounts of azo dye adsorbed per unit mass (q_t) was plotted against the square root of contact time ($t^{0.5}$). The intra-particle diffusion plot was observed to fit successfully for the systems at different pH, and an increase in the intra-particle diffusion rate constant (Table I) was observed with increasing pH. A straight-line plot that did not pass through the origin was noticed for the azo dye sorption system. This indicates some degree of boundary layer control which further shows that intra-particle diffusion is not the only rate-controlling step, but also other processes may control the rate of adsorption [19]-[23]. The intra-particle diffusion plot of azo dye sorption system indicates the complex nature involving both boundary layer and intra-particle diffusions with respect to initial azo dye concentration. The intra-particle diffusion model assumes that the diffusion (internal surface and pore diffusion) of sorbate molecules inside the adsorbent is rate limiting and the film diffusion is considered negligible [23], [24].

c) Elovich equation

The Elovich equation (Eq. 4) is a model used to describe irreversible chemisorption [25].

$$dq_t/dt = ae^{-bq_t} \quad (4)$$

where, a ($\text{mg/g} \cdot \text{min}$) is the initial adsorption rate. The Elovich equation parameters a , b were calculated from q_t vs $\ln t$ plots (Table I). The constants a , b are related to the rate of chemisorption and the surface coverage, respectively [26]. Therefore, increase in the concentration of dye solution

and mass of sorbent will increase the rate of adsorption. Also an increase in dye concentration or decrease in the mass of CSW-AC will reduce the available adsorption surface. The constant a (related to the rate of chemisorption) was observed to be highest at neutral pH supporting maximum sorption reached in 50 min, which was 60 min with other pH conditions.

d) Comparison of adsorption kinetics

The parameters calculated by application of kinetic models over the experimental data were used to predict the effect of time on dye-CSW-AC adsorption system. Considering all the above results, the kinetics of dye adsorption over CSW-AC can be described best by pseudo-first order.

TABLE I: COMPARISON OF ADSORPTION KINETIC CONSTANTS

	pH = 4.0	pH = 5.0	pH = 6.0	pH = 7.0
Pseudo-first order				
q_e (mg/g)	9.744	7.608	9.735	9.030
K_1 (min ⁻¹)	1.07E-2	1.57E-2	1.19E-2	1.44E-2
R^2	0.965	0.952	0.968	0.977
Pseudo-second order				
q_e (mg/g)	8.771	4.566	9.708	5.291
K_2 (g/mg.min)	1.7E-3	1.04E-2	1.2E-3	7.4E-3
R^2	0.988	0.768	0.763	0.768
Weber's Intra-particle diffusion model				
K_p (mg/g.min ^{0.5})	0.612	0.613	0.685	0.709
R^2	0.936	0.938	0.927	0.940
SSE	1.157	1.124	1.658	1.468
Elovich equation				
a (mg/g.min)	0.352	0.386	0.356	0.402
b (g/mg)	0.641	0.651	0.549	0.551
R^2	0.884	0.867	0.911	0.890

TABLE II: COMPARISON OF ADSORPTION ISOTHERM MODEL PARAMETERS

	pH = 4.0	pH = 5.0	pH = 6.0	pH = 7.0
Langmuir isotherm				
q_{ml} (mg/g)	0.794	0.903	0.887	1.035
b_L (mg/l)	0.414	0.531	0.783	0.581
R_L	0.194	0.158	0.113	0.146
R^2	0.432	0.355	0.337	0.533
Freundlich isotherm				
K_F ((m/g)/(mg/l) ^N)	6.896	6.566	6.302	5.731
N	-1.230	-1.303	-1.206	-1.406
R^2	0.781	0.844	0.784	0.885
Temkin isotherm				
b (J/mol)	-942.439	-1027.382	-1202.454	-1228.250
K_T (l/mg)	0.086	0.081	0.076	0.073
R^2	0.956	0.941	0.919	0.949
Sips isotherm				
q_s (mg/g)	1.575E-3	1.557E-3	3.98E-4	1.19E-3
K_s ((l/mg) ^{1/m})	-0.998	-0.999	-0.999	-1.000
$1/m$	1.28E-3	8.36E-4	2.57E-4	4.48E-4
R^2	0.372	0.554	0.339	0.496

2) Adsorption isotherms

Equilibrium isotherm models express the relationship between the quantity of sorbate absorbed and the concentration in solution at equilibrium. The parameters derived from these equations and the underlying thermodynamic assumptions of these equilibrium isotherm models provide insight into the sorption mechanism and the surface properties and affinity of the sorbent. Equilibrium sorption models were employed to estimate sorption capacities of azo dye with CSW-AC as sorbent.

a) Langmuir model

The Langmuir isotherm is one of the most commonly

used models describing the equilibrium concentration at the adsorbent surface (q) and sorbate concentration in solution (C) at a constant temperature. The Langmuir model [27], [28] can be represented by equation 5. The essential characteristic of the Langmuir isotherm can be expressed in terms of a dimensionless constant, separation factor R_L (equation 6), which describes the type of isotherm. The values of R_L indicate the type of isotherm to be unfavourable ($R_L > 1$), linear ($R_L = 1$), favourable ($0 < R_L < 1$) and irreversible ($R_L = 0$).

$$q = q_{ml} b_L C / (1 + b_L C) \quad (5)$$

$$R_L = (1 + b_L C_0)^{-1} \quad (6)$$

where, q_{mL} ($\mu\text{g/g}$) and b_L are the Langmuir parameters that represent the maximum sorption capacity and the site energy factor, respectively. A non-linear regression of sorption data for the azo dye-CSW-AC system was applied and the resulting Langmuir parameters for sorption capacity (q_{mL}) calculated from the slope and intercept of the plot respectively (Table II). The Langmuir model for CSW-AC sorption system showed a poor fit. The values of R_L for CSW-AC sorbent system represented that the azo dye sorption was favourable over the pH range.

b) Freundlich model

The Freundlich model, which is given in equation 7, is very similar to the Langmuir model [28], [29].

$$q = K_F C^N \quad (7)$$

where, C (mg/l) is the aqueous phase concentration, q_t (mg/g) is the solid phase concentration, and K_F [$(\text{mg/g})/(\text{mg/l})^N$] and N are the Freundlich sorption coefficient and the Freundlich exponent, respectively. The Freundlich sorption coefficient (K_F) for azo dye-CSW-AC system was calculated by fitting the sorption data using equation 7. The Freundlich model showed a good fit with the CSW-AC sorption system. It was observed that the K_F (Table II) decreased with increasing pH while the Freundlich exponent was almost constant.

c) Temkin model

The Temkin isotherm assumes that the decrease in the heat of adsorption is linear and the adsorption is characterized by a uniform distribution of binding energies. The Tempkin model can be explained using equation 8. [30].

$$q_t = (RT/b) \ln(K_T C) \quad (8)$$

where, C (mg/l) is the aqueous phase concentration, q_t (mg/g) is the solid phase concentration, K_T (l/mg) is equilibrium binding constant, b (J/mol) is related to heat of adsorption, R is the gas constant (8.314 J/mol/K) and T (K) is the absolute temperature. The equilibrium binding constant (K_T) for azo dye-CSW-AC system were calculated by fitting the sorption data in equation 8 (Fig. 5). It was observed that the K_T value reduced while the b value increased with pH. The values of constant ' b ' indicated that the heat of adsorption increased with increase in pH and hence the adsorption can possibly be improved by decreasing the temperature.

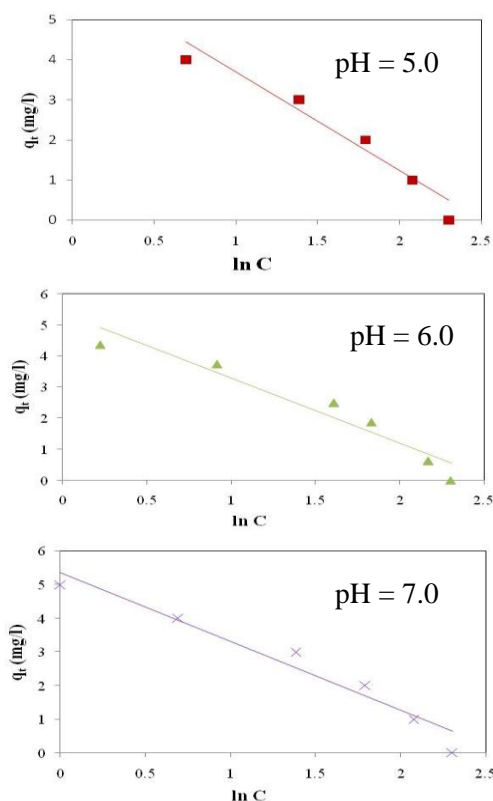
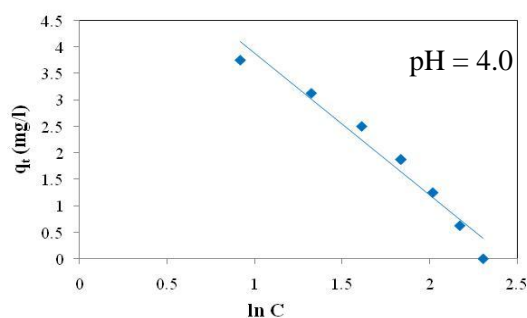


Fig. 5. Temkin adsorption equilibrium model.

d) Sips model

The Sips model (equation 9) [31] is a combination of the Langmuir and Freundlich models. At low sorbate concentrations it reduces to the Freundlich isotherm and at high concentrations it becomes the Langmuir model.

$$q_t = q_s K_s C^{m^{-1}} / (1 + K_s C^{m^{-1}}) \quad (9)$$

where, q_s (mg/g) is monolayer adsorption capacity and K_s is Sips constant related to energy of adsorption. A power fit over the sorption data for the dye-CSW-AC system was applied to calculate the parameters shown in Table II. The exponent m^{-1} (Table II) is close to zero, indicating the dye-CSW-AC system is more closely explained by the Freundlich model compared to the Langmuir model.

e) Comparison of adsorption isotherms

The constants derived by fitting the isotherms over the experimental data were used express relationship between the quantity of sorbate absorbed and the concentration in solution at equilibrium. Considering all the above results, the adsorption isotherm of dye-CSW-AC system can be best described by Temkin isotherm over the pH range.

IV. CONCLUSIONS

Adsorption of acid black dye using activated carbon derived from the cellulose fraction of municipal solid waste was investigated. Four non-linear kinetic models and four non-linear isotherm models were used to investigate the experimental data and ranked based on the statistical analysis tools in origin software. The dye-CSW-AC system is best described by pseudo-first order. Also the equilibrium data fit the Temkin model well at all aqueous solution pH.

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