Adsorption of Methyl Blue by Maize Waste Based Biochar: Adsorption Kinetics and Isotherms

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Abstract. Different residue sections from maize were used as the raw materials to prepare biochars through pyrolysis under oxygen-limited conditions, and their adsorption efficiencies were compared. The results showed that leaf derived biochar had a best adsorption performance for methyl blue (MB) than other residues. It is interesting that the correlation coefficient ($R^2$) was affected by the dosage of leaf biochar, indicating that the adsorption mechanism was influenced by the dosage of adsorbent. Elovich model was suitable for the low dose of adsorbent while the pseudo-second-order kinetic model was more appropriate for the high dose of adsorbent. The adsorption of MB onto biochar was governed by chemisorption as well as bulk and surface diffusion, and was mainly controlled through the chemisorption. The adsorption kinetics of MB from leaf derived biochar could be well described by a first-order, two-compartment model under the dose of leaf derived biochar. MB adsorption onto leaf derived biochar was likely controlled by heterogeneous processes with multilayer adsorption, and the process could be governed by multiple mechanisms through the analysis of adsorption isotherms.

1. Introduction
At present, a large number of synthetic dyes are produced worldwide every year, and extensively used in textile, printing, paper, leather, cosmetic, and other industries. Due to their large production and wide application, a large amount of dye-containing wastewater has been released into the aquatic environment, resulting in ubiquitous presence of dyes in natural waters. The presence of these pollutants in water has caused serious concerns with regards to strong visual impact and adverse effects on human and aquatic organisms due to their carcinogenic, mutagenic and teratogenic properties [1]. Thus, it is necessary to find effective approaches to purify dye-containing effluents. Various methods have been investigated for the dye-containing effluents including physical, chemical, biological methods. Among the above-mentioned methods, adsorption is considered to be an attractive method for the treatment of organic pollutants wastewaters due to its low cost and energy requirement, simplicity of design, ease of operation, wide range of adsorbents availability, and insensitivity to toxic pollutants [2]. Many materials, such as sludge [3], and wastes from agriculture [1, 2], have been applied for such processes. Recently, Biomass has been used to prepare biochar for removing different kinds of pollutants from water due to its low cost and wide availability.

Maize is one of the main crops grown worldwide with annual production of over $1.06 \times 10^9$ tons from the Food and Agriculture Organization of the United Nations. China is the second largest producer of maize in the world, and the annual maize residues yield reaches over $2.7 \times 10^8$ tons according to data from National Bureau of Statistics of People’s Republic of China in 2016 [4]. Maize
residues are not only a representative of agricultural wastes but also one of significant renewable biomass resource with abundantly available lignocellulosic. However, these residues are often treated as waste and burned without being used effectively in the field, thus not only wasting of energy but causing severe environmental problem. It is urgent to make good use of maize residues and transformed into high value-added by-products in a sustainable and environment-friendly way. The maize residues have a great potential to be used as feedstock of biochar [5], and biogas production [4]. Among these applications, biochar is produced from the incomplete burning of carbon-rich biomass in a closed container with limited oxygen which could be used for removing environmental pollutants [5, 6]. However, the whole maize residues are often used as raw material for biochar and other products in previous reports although they contain a number of parts including tassel, stalk, leaf, root, silk, ear, and corncob, which has different composition/structure and thus probably leads to distinct properties of the obtained products.

In current study, biochars are prepared from different maize residue sections through pyrolysis under oxygen-limited conditions, respectively, and their adsorption performances for MB are compared. The primary objective is to elucidate the differences and related mechanisms of MB adsorption on biochars derived from various maize residue sections.

2. Materials and Methods

2.1 Preparation and Characterization of Biochar Samples
Maize residues were selected as the precursor material for biochar production, which were collected from a local farmland in Jinzhou, Liaoning province, China. The maize residues were first divided into seven sections (tassel, leaf, stalk, root, silk, ear, corncob), and then washed with water several times to remove dusts and air-dried for 2 d, respectively, and subsequently oven-dried overnight at 80 °C. The maize residues were crushed into powder by a disintegrator and passed through the sieve for use. Biochars were produced by pyrolyzing the maize residue powders under oxygen-limited conditions as described in a previous report. A certain amount of the powders (including tassel, leaf, stalk, root, silk, ear, corncob, respectively) were tightly packed in porcelain crucibles with lids, and pyrolyzed in a muffle furnace at 500 °C for 6 h with a 5 °C/min heating rate.

2.2 Batch Adsorption Experiment
Adsorption experiments were conducted to determine and compare the adsorption potential of biochars derived from different residue sections of maize. An aliquot of biochar was added into 300 mL solution with certain MB concentration. The adsorption studies were carried out at different intervals in an incubator shaker at 150 rpm. The detail experimental conditions could be found in figure caption. After adsorption, the suspensions were centrifuged at 8000 rpm for 5 min, and the supernatant was collected by decantation. The remaining concentration of MB in the solution was measured at 665 nm using a UV-visible spectrophotometer. All experiments were carried out in triplicate. The adsorption capacity and adsorption efficiency (AE) was calculated using Eq. (1), Eq. (2) and Eq. (3).

\[ Q_t = \frac{(C_0 - C_t)V}{m} \]  \hspace{1cm} (1)

\[ Q_e = \frac{(C_0 - C_e)V}{m} \]  \hspace{1cm} (2)

\[ AE(\%) = \frac{(C_0 - C_e)}{C_0} \times 100 \]  \hspace{1cm} (3)

where \( C_0, C_t, C_e (\times 10^{-2} \text{ mmol/L}) \) are the initial, \( t \) min and equilibrium concentrations, respectively. \( V (\text{L}) \) is the volume of MB solution. \( m (\text{g}) \) is the mass of biochar adsorbent.
3. Results and Discussions

3.1 Adsorption Efficiency

The biochars derived from different residue sections of maize on the adsorption efficiency of MB from aqueous solution. The mixture of MB solution and biochar (0.4 g/L) was shaken at a rate of 150 rpm (25±1 °C) with a MB concentration of $1.5 \times 10^{-5}$ mol/L. In general, the adsorption efficiency of pollutants increased with increasing contact time. The adsorption efficiency of MB for all samples increased quickly at the beginning and then slowly increased at longer contact time to reach an equilibrium at 1440 min. The adsorption efficiency of MB of tassel, leaf, stalk, root, silk, ear, and corncob based biochar was 59.08, 89.32, 23.32, 41.12, 20.94, 44.13, and 33.38%, respectively. The adsorption performance of maize leaf derived biochar was better than any other samples under the same experimental condition. The results indicated that the adsorption of MB on biochars might be mainly due to the differences in specific surface area, pore structure, and surface functional groups of biochar from seven raw materials.

3.2 Adsorption Kinetics

In order to elucidate the adsorption mechanism and potential rate-controlling steps, Pseudo-first-order, Pseudo-second-order, Elovich, Intraparticle diffusion kinetic models were used to fit the experimental data (Figure 1a and Table 1).

Table 1. The four adsorption kinetic fitting parameters of leaf derived biochar

| Biochar (g/L) | $Q_{\text{exp}}$ (×10$^2$ mmol/kg) | Pseudo first order kinetic equation | Pseudo second order kinetic equation |
|---------------|---------------------------------|-----------------------------------|-------------------------------------|
|               | $Q_{\text{cal}}$ (×10$^2$ mmol/kg) | $k_1$ (min$^{-1}$) | $R^2$ | $Q_{\text{cal}}$ (×10$^2$ mmol/kg) | $k_2$ (kg/(×10$^2$ mmol/kg) min$^{-1}$) | $h$ (×10$^{-3}$ mmol/kg) | $R^2$ |
| 0.2 g/L       | 3.6450                          | 2.6425/1.0091                  | 0.0455 | 0.6389 | 2.8934/0.7604 | 0.0190 | 0.1594 | 0.7832 |
| 0.4 g/L       | 3.3158                          | 2.6448/1.0068                  | 0.0284 | 0.7559 | 2.8627/0.7908 | 0.0149 | 0.1221 | 0.8760 |
| 0.6 g/L       | 2.3789                          | 2.1028/1.5465                  | 0.0312 | 0.7562 | 2.2455/1.4042 | 0.0230 | 0.1162 | 0.8799 |
| 0.8 g/L       | 1.7646                          | 1.6171/2.0312                  | 0.0600 | 0.8284 | 1.7159/1.9326 | 0.0602 | 0.1772 | 0.9317 |
| 1.0 g/L       | 1.3993                          | 1.3541/2.2938                  | 0.2319 | 0.9394 | 1.3912/2.2568 | 0.3062 | 0.5925 | 0.9812 |
| 1.5 g/L       | 0.9418                          | 0.9427/2.7048                  | 0.3215 | 0.9731 | 0.9591/2.6884 | 0.7947 | 0.7309 | 0.9912 |

| Biochar (g/L) | Eluvich model | Intraparticle diffusion model |
|---------------|---------------|-------------------------------|
|               | $\alpha$     | $\beta$                      | $R^2$ | $C$ | $k_e$ (mmol/(g min$^{0.5}$)) | $R^2$ |
| 0.2 g/L       | 1.1021        | 2.5237                        | 0.9210 | 1.3575 | 0.0650 | 0.9824 |
| 0.4 g/L       | 0.7467        | 2.4614                        | 0.9806 | 1.2794 | 0.0629 | 0.9236 |
| 0.6 g/L       | 0.9198        | 3.3424                        | 0.9542 | 1.1243 | 0.0445 | 0.8210 |
| 0.8 g/L       | 3.4714        | 5.2712                        | 0.9419 | 1.0823 | 0.0261 | 0.6852 |
| 1.0 g/L       | 3.5268×10$^4$ | 13.6240                      | 0.8914 | 1.1759 | 0.0094 | 0.5521 |
| 1.5 g/L       | 6.7546×10$^3$ | 33.4225                      | 0.7030 | 0.8763 | 0.0034 | 0.3267 |

As shown in Figure 1b, the correlation coefficient ($R^2$) of the Pseudo-second-order kinetic model (0.7832-0.9912) was larger than that of the Pseudo-first-order kinetic model (0.6389-0.9731) under the same dose of leaf derived biochar. Meanwhile, the goodness of fit between experimental ($Q_{\text{exp}}$) and calculated ($Q_{\text{cal}}$) equilibrium adsorption capacity was evaluated by the sum of squared errors (SSE, (Eq. (4)) [5]. According to SSE, the adsorption capacity calculated from pseudo-second-order equation
(SSE = 0.7604-2.6884) was more consistent with the experimental values than calculated from the pseudo-first-order equation (SSE = 1.0091-2.7048). Therefore, the Pseudo-second-order kinetic model was more suitable for describing the adsorption behaviors of MB onto leaf derived biochar, indicating that the adsorption rate-controlling step of the MB was dominated by chemisorption involving valence forces by sharing or exchange of electrons between adsorbate and adsorbent [6]. In addition, the initial adsorption rate (\(h\)) showed a tendency of increase as the dose of leaf derived biochar increased.

\[
SSE = \sqrt{\frac{\sum (Q_{\text{exp}} - Q_{\text{cal}})^2}{N}}
\]  

(4)

Different processes, including bulk and surface diffusion as well as chemisorption, could be described by an Elovich model. Compared with \(R^2\) value, kinetics data fitted through Elovich model for the adsorption of MB from aqueous solution were better than Intraparticle diffusion model which assumed that pore diffusion was the main rate-controlling step. The above analysis demonstrated chemisorption occurred between MB and leaf derived biochar on highly heterogeneous adsorbents, and the pore diffusion might not be the sole rate-controlling step in adsorption processes. As shown in Table 1, \(\alpha\) and \(\beta\) value varied as a function of the dose of leaf derived biochar. The \(\alpha\) value was tremendous increased as the dose of leaf derived biochar increased from 0.2 to 1.5 g/L, and the \(\beta\) value showed a similar trend, implying that initial adsorption rate increased with the added dose of leaf derived biochar which was consistent with the conclusion from pseudo-second-order kinetic model.

It is interesting that the correlation coefficient \((R^2)\) of Pseudo-first-order and Pseudo-second-order kinetic models increased as the dose of leaf derived biochar increased from 0.2 to 1.5 g/L; while the correlation coefficients \((R^2)\) of Elovich and Intraparticle diffusion kinetic models showed opposite trend. It was apparent that the \(R^2\) was affected by the dosage of leaf biochar, indicating the adsorption mechanism was influenced by the dosage of adsorbent. Elovich model was suitable for the low dose of adsorbent while the pseudo-second-order kinetic model was more appropriate for the high dose of adsorbent. This was to say, the adsorption of MB onto biochar was governed by physisorption as well as bulk and surface diffusion, and was mainly controlled through the chemisorption.
Figure 1. Four kinetics of MB adsorption onto leaf derived biochars. (300 mL 1.5 × 10⁻⁵ mol/L MB solution, 0.06, 0.12, 0.18, 0.24, 0.30, 0.45 g biochar, 25 °C); The first-order, two-compartment model of MB adsorption onto leaf derived biochars. (300 mL 1.5 × 10⁻⁵ mol/L MB solution, 0.06, 0.12, 0.18, 0.24, 0.30, 0.45 g biochar, 25 °C); The adsorption isotherms of MB adsorption onto maize derived biochars. (300 mL 1.0, 1.5, 2.0, 2.5, 3.0, 4.0, 5.0 × 10⁻⁵ mol/L MB solution, 0.12 g biochar, 25 °C).

In addition, the adsorption kinetics of MB from leaf derived biochar was also described by a first-order, two-compartment model (Figure 1c). It could be seen that the adsorption of MB by leaf derived biochar could be divided into fast and slow processes by first order, two compartment model. As shown in Table 2, the first-order, two-compartment model well fitted the kinetics data of MB adsorption on leaf derived biochar with $R^2$ ranged in 0.9898-0.9983 with the different doses of biochar. The $F_{\text{fast}}$ values of leaf derived biochar were smaller than those of $F_{\text{slow}}$ when the dose of leaf derived biochar was from 0.2 to 0.6 g/L, indicating that the slow adsorption stage was predominant during MB adsorption process. While the $F_{\text{fast}}$ values of leaf derived biochar were larger than those of $F_{\text{slow}}$ when the dose of leaf derived biochar was over 0.6 g/L, suggesting that the fast sorption stage was prominent. It was commonly considered that large amount of vacant adsorption sites with functional groups binding of MB as well as the π-π electron-donor-acceptor interactions on the surface of biochar might cause the rapid adsorption at initial stage through the binding of MB, nevertheless, as adsorption continued, the surface sites were close to saturation and hardly occupied [5].

Table 2 The first-order, two-compartment model fitting parameters of leaf derived biochar

| Biochar | $F_{\text{fast}}$ | $F_{\text{slow}}$ | $k_{\text{fast}}$ | $k_{\text{slow}}$ | $R^2$ |
|---------|-----------------|-----------------|-----------------|-----------------|-------|
| 0.2 g/L | 0.5559          | 0.4280          | 326.8380        | 2.0098          | 0.9912|
| 0.4 g/L | 0.5424          | 0.4410          | 247.3572        | 3.4157          | 0.9924|
| 0.6 g/L | 0.5686          | 0.4306          | 121.7508        | 1.5173          | 0.9975|
| 0.8 g/L | 0.4976          | 0.4918          | 68.2552         | 1.3171          | 0.9983|
| 1.0 g/L | 0.4868          | 0.4857          | 6.6176          | 0.0925          | 0.9495|
| 1.5 g/L | 0.4971          | 0.4977          | 3.6343          | 0.0080          | 0.9775|
3.3 Adsorption Isotherms

The adsorption isotherms were used to investigate how the adsorbates distribute on the adsorbents under the adsorption/desorption equilibrium. Adsorption isotherm data was investigated by Langmuir, Freundlich, Sips (Langmuir-Freundlich), Redlich-Peterson, and Dubinin-Radushkevich models which were employed to interpret the adsorption mechanism, and the figure and fitting parameters of these models were presented in Figure 1d and Table 3, respectively.

**Table 3** The isotherm constants for MB adsorption onto maize derived biochars

| Adsorption isotherm     | Parameter 1       | Parameter 2       | Parameter 3       | $R^2$  |
|------------------------|-------------------|-------------------|-------------------|--------|
| Langmuir               | $Q_0$ = 3.8307    | $K_L$ = 21.05     | $R_L$ = 0.0094    | 0.9238 |
| Freundlich             | —                 | $K_F$ = 3.50      | $n$ = 8.80        | 0.9252 |
| Sips                   | $Q_0$ = 4.3563    | $K_{LF}$ = 4.70   | $n$ = 0.4972      | 0.9523 |
| Redlich-Peterson       | $Q_0$ = 3.6700    | $K_{RP}$ = 39.79  | $n$ = 0.9396      | 0.9686 |
| Dubinin-Radushkevich   | $Q_0$ = 3.7844    | $K_{DR} = 1.07 \times 10^{-8}$ | —                | 0.9161 |

The Langmuir constant $K_L$ is often applied to calculating the dimensionless separation factor $R_L$ (Eq. 5), which can evaluate the feasibility and favorable of adsorption. When $R_L = 0$, adsorption is irreversible; $0 < R_L < 1$ indicates favorable adsorption; $R_L = 1$ indicates linear adsorption; and $R_L > 1$ indicates unfavorable adsorption. The $R_L$ results for this study were summarized in Table 3, and all the values (0.0094-0.0454) were between 0 and 1, indicating the adsorption of MB onto the biochar was favorable.

$$R_L = \frac{1}{1 + K_L C_0} \quad (5)$$

The Freundlich isotherm model was proposed as an empirical equation that was often used to describe chemisorption and treat multilayer adsorption on heterogeneous surfaces [7], and the adsorption capacity of biochar for MB infinitely increased with increasing initial concentration to maximum adsorption. The values of $1/n$ (0.1136, 0 < $1/n < 1$) revealed a higher likelihood of chemisorption and multilayer adsorption of MB on the active heterogeneous sites of leaf derived biochar. The degree of heterogeneity of adsorbent surface increased when the value of $1/n$ approached zero.

This finding could be further established by the coverage factor ($\theta$) for MB adsorption, as reported in a previous research, which could be estimated from the amount of dye adsorbed and the surface area occupied by one dye molecule using the following equation:

$$\theta = \frac{Q_{\text{max}} N \sigma \times 10^{-20}}{S_{\text{BET}}} \quad (6)$$

where $Q_{\text{max}}$ (3.83 × 10⁻⁵ mol/g) is the maximum adsorption capacity at saturation, as obtained from the Langmuir model; $N$ is the Avogadro’s number ($6.022 \times 10^{23}$ molecules/mol); $S_{\text{BET}}$ is the specific surface area of the adsorbent (2.72 m²/g), the surface areas were measured using the Brunauer–Emmett–Teller (BET) method by Surface Area and Porosimetry Analyzer (ASAP 2010, Micromeritics Instrument Corporation, USA); $\sigma$ (79.40 Å²/molecule) is the surface area occupied by one molecule, and is estimated from the following empirical relationship:

$$\sigma \left( \text{Å}^2/\text{molecule} \right) = 1.091 \times 10^{16} \left( \frac{\text{MW}}{\rho N} \right)^{2/3} \quad (7)$$
where MW is the molar mass of the adsorbed MB molecule (373.90 g/mol); \( \rho \) is the adsorbate density (1.00 g/cm\(^3\)). The value of coverage factor (\( \theta \)) was calculated equal to \( \theta = 6.726 \) more than 1, further indicating that the formation of a complete molecular layer (\( \theta = 1 \)) was achieved for the MB and all the biochar surface had been occupied [8].

The Sips isotherm model could be considered as a combination of Langmuir and Freundlich isotherm models. If the value of \( C_v \) or \( K_{LF} \) approached to 0, Sips equation reduced the Freundlich isotherm model, while the value of \( n \) equal or close to 1, the Sips equation reduced to the Langmuir isotherm model. Thus, the sips binding isotherm model was regarded as a model both homogeneous and heterogeneous binding surfaces [9]. The higher \( R^2 \) (0.9523) for the Sips isotherm model indicated that the data could be fitted this model satisfactorily, implying the interaction between MB and leaf derived biochar could be better governed by both the Langmuir and Freundlich, that is to say, multiple mechanisms [10].

The Redlich-Peterson isotherm model provided the best fit \( (R^2 = 0.9698) \) to the data of MB equilibrium adsorption onto leaf derived biochar. The value of \( n_{RP} \) (0.9396) was close to 1, indicating that MB adsorption onto leaf derived biochar was favorable. The value of \( n_{RP} \) was also closer to \([1-(1/n_{RP})]=(0.8864)\), implying that the adsorption of MB onto leaf derived biochar is more heterogeneous in nature [11].

Dubinin-Radushkevich isotherm was chosen to estimate the apparent free energy as well as the characteristics of adsorption. The isotherm was similar to the Langmuir model but it was more general due to determining the occurrence of adsorption on both homogeneous and heterogeneous surfaces [12]. In the case of \( E_d \) less than 8 kJ/mol, the adsorption process is of a physical nature. The adsorption process occurs by ion exchange when \( E_d \) is between 8 and 16 kJ/mol. Its value in the range of 16-40 kJ/mol is indicative of chemisorption [3]. The obtained values of the \( E_d \) (6.84 kJ/mol) indicated that the interaction between MB and leaf derived biochar existed a weak physisorption.

According to the Langmuir, Sips, Redlich-Peterson, and Dubinin-Radushkevich models, the maximum adsorption capacities for MB were achieved: 3.83, 4.36, 3.67, and 3.78 mmol/g, respectively. With respect to \( Q_m \): Sips > Langmuir > Dubinin-Radushkevich > Redlich-Peterson. However, when \( R^2 \) was considered, the order was: Redlich-Peterson (0.9686) > Sips (0.9523) > Freundlich (0.9252) > Langmuir (0.9238) > Dubinin-Radushkevich (0.9161). The above results revealed that MB adsorption onto leaf derived biochar was likely controlled by heterogeneous processes with multilayer adsorption, and the process could be governed by multiple mechanisms.

4. Conclusions

The adsorption efficiency (89.32\%) of maize leaf derived biochar was better than any other samples (The tassel, leaf, stalk, root, silk, ear, and corncob based biochar were 59.08, 23.32, 41.12, 20.94, 44.13, and 33.38\%, respectively) under the same experimental condition. The correlation coefficient \( (R^2) \) from kinetic fitting was affected by the dosage of leaf biochar, suggesting the adsorption mechanism was influenced by the dosage of adsorbent. Elovich model was suitable for the low dose of adsorbent while the pseudo-second-order kinetic model was more appropriate for the high dose of adsorbent. The adsorption of MB onto biochar was governed by chemisorption as well as bulk and surface diffusion, and was mainly controlled through the chemisorption. The adsorption kinetics of MB from leaf derived biochar could be well described by a first-order, two-compartment model under the dose of leaf derived biochar. Redlich-Peterson presented a perfect fitting for MB adsorption, indicating that the adsorption of MB onto leaf derived biochar is more heterogeneous in nature. Langmuir and Redlich-Peterson showed that MB adsorption onto leaf derived biochar was favorable. The fitting results of adsorption isotherms indicated that MB adsorption onto leaf derived biochar was likely controlled by heterogeneous processes with multilayer adsorption, and the process could be governed by multiple mechanisms.

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