Treatment of Tropical stabilized landfill leachate by Adsorption using Powdered Activated Carbon: Isothermal and Kinetic Studies

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Abstract. This study investigates the adsorption removal capacity of chemical oxygen demand (COD), color, and ammoniacal nitrogen (NH₃-N) from Sahom stabilized landfill in Kampar, Malaysia by powdered activated carbon (PAC). The effects of shaking speed, contact time, dosage of activated carbon and pH level on the adsorption performance were tested in a batch equilibrium study. Equilibrium data was favorably described by Langmuir isotherm model. The maximum monolayer adsorption capacity for COD, color and NH₃-N were 27.7 mg/g, 172.4 PtCo/g and 4.76 mg/g, respectively and achieved at the optimum conditions of: shaking speed 250 rpm, contact time 4 hours, PAC dosage 4 g/100 ml leachate, and at pH=9. Effluent’s average removal efficiency was found to be 66.00, 87.63 and 25.89% for COD, color, and NH₃-N, respectively. Based on the kinetic data, the adsorption process was controlled by chemisorption as it agreed satisfactorily with the Pseudo-second order model. The regeneration of activated carbon was done thermally via microwave heating. The recovery efficiency for COD, color and NH₃-N were found to be 85.47%, 92.65% and 59.53%, respectively. The results revealed the feasibility of PAC adsorbent for the adsorptive treatment of landfill leachate.

Keywords: Adsorption isotherm; Kinetics; Activated Carbon; Stabilized Landfill Leachate; Waste Management.

1. Introduction
Landfilling is the most commonly used method for solid waste disposal in Malaysia. Unfortunately, because of the lack of technology and investment, open-dumping landfills are the most public in Malaysia which are not equipped with proper facilities such as leachate collection and treatment systems; and gas collection system. As a result, the untreated leachate can cause various environmental hazards like release of toxic gases to open environment and altering natural soil, surface and ground water chemistry [1–2].

Landfill leachates vary in composition and are usually categorized based on its age into three categories, i.e., young, intermediate, and stabilized leachate. With time, as the leachate moves from young to stabilized condition, the biodegradable compounds decomposed into more stable non-biodegradable organic components like humic and fulvic-like fractions [1]. Usually, stabilized leachate is produced from a landfill site which is at least 10 years old and also has high ammoniacal nitrogen and...
low BOD₅/COD ratio [1,4]. So, for stabilized leachate, biological treatment method is no longer efficient to treat the leachate and reduce its negative impacts on the environment and aquatic life [9,10]. Because of the stable nature of stabilized leachate, it is comparatively easy to develop physiochemical treatment process in which adsorption is the most prominent technology [5,6].

In adsorption, an adsorbent plays a key role in removing the desired pollutants from the wastewater. In this regard, activated carbon has been thoroughly studied as an adsorbent mainly because of its high surface area and it can be modified in a number of ways to increase its adsorption capacity [7]. The carbon in the activated carbon is used to remove dissolved organic matter via adsorption [8]. Different kinds of activated carbons have been tried for wastewater with properties similar to that of landfill leachate. It has shown high removal efficiency of chemical oxygen demand (COD), total suspended solids, turbidity, heavy metals, ammonium nitrogen (NH₃-N) and colour in landfill leachate [8]. There are many types of activated carbon with different uses, sizes, and preparation methods. Activated carbon such as powdered activated carbon (PAC), granular activated carbon (GAC), extruded activated carbon (EAC) and bead activated carbon (BAC) are the ones mostly used. Generally, activated carbon is used in gas purification, gold extraction, water purification, medicine, sewage treatment and many other applications [9]. Bashir et al. [6] found that the powder activated carbon (PAC) adsorption is one of the most attractive methods for the removal of recalitrant compounds from leachate because of its large surface area, microporous structure, and surface reactivity. The adsorption of pollutants onto PAC results in great reduction in COD levels; whatever the initial organic matter concentration is [6]. This study aims to investigate the treatability of stabilized Sahom landfill leachate using activated carbon to remove chemical oxygen demand (COD), colour, and ammoniacal nitrogen (NH₃-N). Batch adsorption studies were conducted to evaluate the effect of shaking speed, contact time, adsorbent dosage, and pH level. Furthermore, the equilibrium isotherm as well as the kinetic study have been investigated for the adsorption treatment process of stabilized leachate using PAC as adsorbent. The study also covers the effects of thermal regeneration of the activated carbon via microwave heating.

2. Materials and Methods

2.1. Sample Preparation
Stabilized landfill leachate samples were collected from Sahom landfill (4° 23’ 25” N, 101° 10’ 57” E), located at Jalan Sahom, Kampar district, Perak, Malaysia. The sample was immediately transported to Environmental Laboratory (EV-Lab), Faculty of Engineering and Green Technology, UTAR, Kampar and stored at 4°C to minimize any chemical reactions and biodegradation of the leachate sample. Activated carbon used in this study was procured from R&M Chemicals (Essex, United Kingdoms). The activated carbon was charcoal based and consisted of Chloride, Sulphate, Sulfide, Calcium, Copper, Iron, Lead and Zinc. The density of this PAC was 1.8 – 2.1 kg/m³ and the pH level was 4 – 7.

2.2. Experimental Set-up
The original SLL characteristics were measured firstly in the lab, and treatment of SLL was done by adsorption using PAC as an adsorbent. A leachate sample of 100 mL in volume was transferred to a 250 mL conical flask, followed by addition of PAC into the leachate sample where the parametric effects of the PAC dosage, shaking speed and contact time were investigated. The sample was gently mixed and fixed to the Orbital Shaker, NB-101M, Korea. Later, as the shaking process was stopped, PAC was filtered out of the SLL and the characteristics of the treated leachate were analyzed. The percentage of removal of pollutant in the aqueous solution was calculated using equation 1.

\[
\text{Percentage of pollutant removal (\%)} = \left(\frac{C_0 - C_e}{C_0}\right) \times 100
\]

where

\(C_0 = \text{initial concentration of the pollutant}\)

\(C_e = \text{final concentration of the pollutant}\)
Batch experiments were carried out to investigate the effect of parameters on the PAC treated leachate sample, including contact time, shaking speed and PAC dosage. The contact time of the adsorption process was manipulated at 0, 15, 30, 60, 120, 180, 240, 360, and 480 min, with the same shaking speed and PAC dosage of 250 rpm and 2 g/100 mL SLL, respectively. To determine the effect of shaking speed, the adsorption process was carried out under different shaking speed at 0, 10, 30, 50, 100, 200, and 250 rpm. All of the adsorption process for all of the samples were carried out with the constant PAC dosage of 2 g/100 mL SLL and optimum contact time obtained.

2.3. Leachate Characteristics
The initial characteristics of leachate such as total dissolved solids (TDS) and temperature were measured in situ. COD was measured by using USEPA Reactor Digestion Method-Method 8000 according to the standard methods (APHA, 2008). Suspended solid was measured by using Photometric Method with Spectrophotometer. Turbidity was evaluated by TurbiCheck (Lovibond). Colour was measured by using Platinum-Cobalt (PtCo) standard method (Standard Method of the Determination of Wastewater and water, the method used was method 8025). NH\textsubscript{3}-N test was conducted using Nessler Method.

2.4. Isothermal Studies
Equilibrium isotherms of the adsorption characteristics including the type of surface, capacity, intensity, and energy of the adsorbent (PAC) on the adsorbate (SLL) were studied by Langmuir isotherm and Freundlich isotherm models. Langmuir isotherm describes the efficiency of the adsorbent in the adsorption mechanism with a plot of amount of impurity adsorbed by adsorbent (PAC) against the amount of impurities remaining in the adsorbate (SLL). Langmuir model assumes that the adsorbent surface has fixed numbers of accessible sites which are homogeneously available on the monolayer surface where each site can accommodate only one molecule. There is a fixed area for each site of adsorbent that is quantified by the geometry of the surface, with the same adsorption energy at all the sites. According to Metcalf & Eddy, the linear form equation of Langmuir isotherm is shown in eq. 2.

\[
\frac{1}{q_e} = \frac{1}{Q K_L C_e} + \frac{1}{Q}
\]

(2)

where

- \( q_e \) = mass of adsorbate adsorbed per unit mass of adsorbent, (mg/g)
- \( Q \) = the maximum monolayer adsorption capacity, (mg/g)
- \( K_L \) = Langmuir constant, (L/mg)
- \( C_e \) = equilibrium concentration of adsorbate in solution after adsorption, (mg/L)

In Freundlich isotherm model, it is assumed that heterogeneous surfaces favor the adsorption process at multilayer which is composed of various classes of adsorption sites. According to Metcalf & Eddy, the linear form equation of Freundlich isotherm is shown in equation 3, where \( K_f \) and \( \frac{1}{n} \) are represented by the intercept and slope of log \( q_e \) against log \( C_e \) plot, respectively.

\[
\log q_e = \log K_f + \frac{1}{n} \log C_e
\]

(3)

where

- \( C_e \) = equilibrium concentration of adsorbate in solution after adsorption, (mg/L)
- \( q_e \) = mass of adsorbate adsorbed per unit mass of adsorbent, (mg/g)
- \( K_f \) = Freundlich constant, adsorption capacity indicator, (mg/g) (mg/L)
- \( \frac{1}{n} \) = Freundlich constant, adsorption intensity indicator
2.5. Kinetic Studies

According to Bashir et al. [10], kinetic modelling is commonly used to investigate the adsorption mechanism and the potential rate of the controlling process such as mass transfer and chemical adsorption. Among the kinetic models available for adsorption study, the pseudo-first order and pseudo-second order models are the most commonly used kinetic models in water and wastewater treatment [11]. Pseudo-first order kinetic model is used to describe a reversible adsorption reaction, namely physisorption which is established with an equilibrium relation between liquid and solid phase. This model suggests that the rate of the reaction is proportional to the concentration of the contaminant. The pseudo-first order equation based on Aziz et al. [12] is shown in equation 4.

$$\log \left( q_e - q_t \right) = \log \left( q_e \right) + \frac{K_1}{2.303} t$$

where

$q_e = \text{total adsorbed pollutants at equilibrium, (mg/g for COD and PtCo/g for colour)}$
$q_t = \text{amount of adsorbate adsorbed at time, } t \ (\text{mg/g})$
$K_1 = \text{equilibrium rate constant of pseudo-first order kinetic model, (min}^{-1})$

Pseudo-second order kinetic model suggests that the surface adsorption is controlled by chemisorption where the removal occurs due to the physicochemical interaction between the solids which are normally adsorbate and liquids which are normally the adsorbtent [13]. The pseudo-second order equation based on Aziz et al. [12] is shown in equation 5.

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

where

$q_e = \text{total adsorbed pollutants at equilibrium, (mg/g for COD and PtCo/g for colour)}$
$q_t = \text{amount of adsorbate adsorbed at time, } t \ (\text{mg/g})$
$K_2 = \text{equilibrium rate constant of pseudo-second order kinetic model, (g/mg min)}$

3. Results and Discussion

3.1. Leachate Characteristics

Table 1 presents the characteristics of leachate collected from Sahom landfill. Characteristics of a leachate can be represented by several parameters including BOD$_5$, COD, colour, NH$_3$-$N$, turbidity, and pH. Based on the results shown in Table 1, Sahom landfill leachate is categorized as stabilized landfill leachate as the BOD$_5$/COD ratio is less than 0.1 [14, 15]. Other factors indicating that the SLL is in the methanogenic phase include relatively low COD concentration and pH >8.

| Parameters                | Units | Values (Range) | Values (Average) | Malaysia Discharge Standards |
|---------------------------|-------|----------------|------------------|-----------------------------|
| BOD$_5$                   | mg/L  | 55 - 122       | 88.5             | -                           |
| COD                       | mg/L  | 1360-1545      | 1452.5           | 400                         |
| BOD$_5$/COD               |       | 0.065 - 0.080  | 0.073            | -                           |
| Total Suspended Solids (TSS) | mg/L  | 38-46          | 42               | 50                          |
| Total Dissolved Solids (TDS) | ppm  | 3.637-4.328   | 3.9825           | -                           |
| Ammonia (NH$_3$-N)        | mg/L  | 1690-1845      | 1767.5           | 5                           |
| Temperature               | °C    | 27.6-29.3      | 28.45            | -                           |
| pH                        |       | 8.84-8.94      | 8.89             | 6.0 – 9.0                   |
| Colour                    | PtCo  | 1475-1575      | 1525             | 100                         |
| Turbidity                 | ntu   | 34.7-36.4      | 35.55            | -                           |
Based on the given data on Table 1, the average values of COD, colour and NH$_3$-N, which are 1452.5 mg/L, 1525 PtCo and 1767.5 mg/L, respectively, do not meet and in fact are much greater than the corresponding Malaysian discharge standards’ values which are 400 mg/L, 100 PtCo and 5 mg/L, respectively.

3.2. Batch Adsorption Studies

3.2.1. Effect of Shaking Speed. The powdered activated carbon was added to the leachate and then were shaken together at different shaking speeds. The constant parameters were the adsorbent dosage which was 2 g of PAC added to 100 ml of leachate and the contact time was 3 hours. A graph of removal efficiency and effluent concentration in respect to different shaking speeds was plotted, as shown in Figure 1. It can be observed from Figure 1 that the reduction of the effluent’s concentrations of COD, colour and NH$_3$-N were relatively steady but slowly increasing. Note that the removal efficiencies of COD, colour and NH$_3$-N increased as the shaking speed increased since the shaking process provided mixing with higher probability of contact between the adsorbate (SLL) and adsorbent (PAC) which improved the treatment efficiency. Also, according to Chabani et al. [15], the resistance of the boundary layer surrounding the adsorbate weakens at strong agitation rates. Thus, the adsorption rate becomes faster at higher shaking speeds. The optimum shaking speed was chosen to be 250 rpm. The removal efficiency of COD, colour and NH$_3$-N were 75.4%, 91.75% and 40.65%, respectively.

![Figure 1. The effluent removal (%) of COD, colour, and NH$_3$-N at different shaking speed (rpm).](image)

3.2.2. Effect of Contact Time. To study the effect of contact time, a set of experiments were carried out by using different contact times. At this stage, the shaking speed used was the optimum speed (250 rpm) and the activated carbon dosage was 2 g/100 ml leachate. A graph of removal efficiency and effluent concentration in respect to different contact times is plotted in Figure 2. The parameters being concerned were chemical oxygen demand (COD), colour, and ammonia. As a result, the optimum contact time was chosen to be 4 hours. The removal efficiency of COD, colour, and ammonia were 76.7%, 91.75% and 38.75%, respectively. It is clear from Figure 2 that the adsorption process increased sharply at the initial stage indicating the availability of readily accessible sites. At the beginning, the solute molecules were adsorbed onto the exterior surface of PAC. When the adsorption on the exterior surface reached saturation, the molecules needed to diffuse into the interior surface of PAC [14].
3.2.3. Effect of Activated Carbon Dosage. The effect of adsorbent dosage on the effluent removal efficiency of COD, colour and NH$_3$-N are demonstrated in Figure 3. A set of experiments were carried out by using different activated carbon dosages (e.g. 0, 0.1, 0.3, 0.5, 0.8, 1.0, 1.5, 2.0, 3.0, 4.0, 5.0 and 6.0 grams). The optimum of both shaking speed (250 rpm) and contact time (4 hrs) were used. The parameters being concerned were COD, colour and ammoniacal nitrogen. As a result, the optimum activated carbon dosage was chosen to be 4 g/100 ml leachate as no more removal efficiency could be achieved with the increase of PAC dosage up to 6 g. The effluent concentration of COD, colour, and NH$_3$-N were 340 mg/L, 95 PtCo and 1025 mg/L, respectively. The removal efficiency of COD, colour, and NH$_3$-N were 77.99%, 93.97% and 44.44%, respectively. The activated carbon dosage helped in the study of quantitative uptake of pollutants by investigating the retention time in response to the activated carbon dosage.

3.2.4. Effect of pH. A set of experiment was carried out by using different pH levels (e.g. 3, 5, 7, 9 and 11). The pH level was adjusted by 0.1 M hydrochloride acid (HCl) and 0.1 M sodium hydroxide.
(NaOH). A graph of removal efficiency and effluent concentration in respect to different pH level was plotted, as shown in Figure 4. As a result, the optimum pH level was found to be 9. The removal efficiency of COD, colour, and ammoniacal nitrogen were 78.68%, 95.93% and 47.34%, respectively. Solution pH was a predominant factor affecting the degree of ionization of adsorbate and the surface charge of a solid adsorbent. Generally, a stabilized leachate contains high amount of polar and nonpolar aggregated organic constituents, in the form of proteins, carbohydrates, organic compounds, and usually expressed in the term COD [13]. The removal of these organic compounds may occur in two mechanisms, physical adsorption (non-polar attractions) and ion exchange (polar attractions). Ion exchange involves counterion displacement and electrostatic [16,17].

![Figure 4. The removal efficiency (%) of COD, colour, and NH₃-N at different pH.](image)

3.3. Isotherm Study

Data obtained from the experimental work was tested with the Langmuir and Freundlich models. Linear regression is frequently used to determine the best-fitting isotherm and the applicability of isotherm equations was compared by judging the correlation coefficients as shown in Table 2. In Langmuir isotherm, the value of adsorption capacities for COD, colour and ammoniacal nitrogen were 27.7 mg/g, 172.4 PtCo/g and 4.76 mg/g, respectively. The R² values obtained for COD, colour and NH₃-N were 0.9668, 0.9707 and 0.949, respectively. In Freundlich isotherm, the value of 1/n for COD, colour, and NH₃-N were 2.9703, 0.5402 and 2.7678, respectively. The R² values obtained for COD, colour, and NH₃-N were 0.9739, 0.9521 and 0.9324, respectively. When comparing the average R² value throughout these two models, the average R² value in Langmuir Isotherm is higher than that in Freundlich Isotherm and closer to 1. As a result, Langmuir model is more fitted to the data and it was selected to represent them. Langmuir isotherm model indicates that the adsorption process was done by monolayer from the leachate pollutants attached on a homogenous surface of adsorbent (PAC) [16,17].

| Parameter | Langmuir isotherm coefficient | Freundlich isotherm coefficient |
|-----------|-------------------------------|---------------------------------|
|           | Q (mg/g) | b (L/mg) | R² | K (mg/g) | 1/n | R² |
| COD       | 27.1     | 0.00157224 | 0.9668 | 0.00000086 | 2.9703 | 0.9739 |
| Colour    | 172.4    | 0.00454047 | 0.9707 | 4.41265527 | 0.5402 | 0.9521 |
| NH₃-N     | 4.76     | 0.00041503 | 0.949  | 0.00000014 | 2.7678 | 0.9324 |
3.4. Kinetic Study
As shown in Table 3, the $R^2$ values obtained in pseudo-first order for COD, colour, and NH$_3$-N were 0.8938, 0.9936 and 0.9642, respectively. The $R^2$ values obtained in pseudo-second order for COD, colour, and NH$_3$-N were 0.9979, 0.9998 and 0.99, respectively. When comparing the $R^2$ value throughout these two models, the $R^2$ value in pseudo-second order is the closest to 1. As a result, pseudo-second order was found to be the most accurate and was thus selected. When the correlation coefficient, $R^2$ is high, that means the adsorption kinetics of the parameter is high [18]. In addition, the calculated equilibrium sorption capacities ($q_{e,\text{cal}}$) of pseudo-second order kinetic model also agreed with experimental data ($q_{e,\text{exp}}$). Thus, the adsorption process for COD, colour, and NH$_3$-N were controlled by chemisorption [17].

Table 3. Kinetic Model of Pseudo-first, Pseudo-second and Intra Particle Model

| Parameter | Exp | Pseudo-First Order Model | Pseudo-Second Order Model |
|-----------|-----|--------------------------|---------------------------|
|           | $q_{e,\text{exp}}$ (µg/g) | $K_1$ (min$^{-1}$) | $q_{e,\text{cal}}$ (µg/g) | $R^2$ | $q_{e,\text{cal}}$ (µg) | $K_2$ (g/µg min$^{-1}$) | $R^2$ |
| COD       | 607 | 0.0103635                | 10926.0                    | 0.89 | 5E-06 | 5000 | 0.99 |
|           | 50  |                          | 972                        | 38   |       | 0    | 98   |
| Colour    | 7375| 0.0158907                | 58735.0                    | 0.99 | 2.5E-07 | 1000 | 0.99 |
| NH$_3$-N  | 410 | 0.0094423                | 3405.6                     | 0.96 | 2.9E-07 | 5000 | 0.99 |
|           | 0   |                          | 944                        | 42   |       | 0    | 0    |

3.5. Regeneration of Spent Activated Carbon
After the spent activated carbon was washed with distilled water, the weights of the two samples were 3.5 g and 3.49 g, respectively. The colour removal efficiency was found to be the best among all which at 87.8% and 87.46%, the COD removal efficiency was 66.18 % and 65.81%, while the NH-$N$ removal efficiency was found to be 25.74% and 26.04%. Figure 5 shows the average removal as well as the recovery efficiencies for COD, colour, and NH$_3$-N. Comparing the original result with the result of regeneration, it can be shown that the recovery efficiency for COD, colour, and NH-$N$ were 85.47%, 92.65% and 59.53%, respectively. The NH-$N$ recovery efficiency was found to be 59.53%, while the colour recovery efficiency was found to be 92.65%, indicating that PAC has high adsorption capacity in organic compounds but low in NH$_3$-N [6].
4. Conclusion
From the result obtained, the optimum shaking speed, contact time, activated carbon dosage and pH level of operation condition for landfill leachate treatment were 250 rpm, 4 hours, 4 grams/100 ml leachate and pH 9, respectively. When referring to the leachate standard, the COD and colour had met the discharge standard. Unfortunately, NH₃-N level did not meet the discharge standards. Moreover, the most suitable isotherm model is Langmuir isotherm model with $R^2$ value 0.9668, 0.9707 and 0.949 for COD, colour, and NH₃-N, respectively. The adsorption capacity, $Q$ for COD, colour and NH₃-N were 27.714 mg/g, 172.414 PtCo/g and 4.762 mg/g, respectively. In the kinetics study, the most suitable model was found to be Pseudo-Second order model with $R^2$ value 0.9998, 0.9979 and 0.990 for COD, colour, and NH₃-N, respectively. In the regeneration of the spent activated carbon process, all the COD, colour, and NH₃-N values failed to meet the discharge standards. Moreover, in the activated carbon section, the recovery efficiency for COD, colour, and NH₃-N were 85.47%, 92.65% and 59.53%, respectively. In conclusion, activated carbon has a great potential in landfill leachate treatment with high removal efficiency of COD, colour, and some removal of NH₃-N via adsorption process.

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