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Adsorption of 2,4-dichlorophenoxyacetic acid by mesoporous activated carbon prepared from H_3PO_4-activated langsat empty fruit bunch

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ABSTRACT

The removal of toxic herbicide from wastewater is challenging due to the availability of suitable adsorbents. The Langsat empty fruit bunch is an agricultural waste and was used in this study as a cheap precursor to produce activated carbon for the adsorption of herbicide 2,4-dichlorophenoxyacetic acid (2,4-D) at different initial concentrations ranging from 50 to 400 mg/L. The produced Langsat empty fruit bunch activated carbon (LEFBAC) was mesoporous and had high surface area of 1065.65 m^2/g with different active functional groups. The effect of shaking time, temperature and pH on 2,4-D removal were investigated using the batch technique. The adsorption capacity of 2,4-D by LEFBAC was decreased with increase in pH of solution whereas adsorption capacity increased with temperature. The adsorption data was well described by Langmuir isotherm followed by removal capacity of 261.2 mg/g at 30 °C. The results from this work showed that LEFBAC can be used as outstanding material for anionic herbicide uptake from wastewater.

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1. Introduction

2,4-Dichlorophenoxyacetic acid (2,4-D) is a popular, selective and systematic herbicide, as well as a plant growth regulator, that is extensively used worldwide on broadleaf plants. The soil/water adsorption coefficient (K_d) measures the strength of pesticide binding with soil particles, where K_d greater than 5 indicates strong adsorption onto soil and organic matter and less probability of leaching. The K_d of 2,4-D is 1.24, which indicates non-persistence in soil, and may therefore persist in aquatic environment. In addition, 2,4-D is non-volatile and highly soluble in water. Exposure of surface and ground water to 2,4-D leads to contamination, which consequently poses serious threat to humans (as carcinogen, reproduction development effects, etc.) and more conspicuously, to benthic marine organisms (PPDB, 2006). The potential toxicity effects of 2,4-D and its transformation products, such as 2,4-dichlorophenol (2,4-DCP), has been well documented by several researchers (Njoku et al., 2013; NRC, 1982; Xi et al., 2010).

The versatility of adsorption using activated carbon in water and wastewater purification has been well established (Loredo-Cancino et al., 2013). However, its high cost limits the use of activated carbon for adsorption methods (Han et al., 2013). Therefore, preparation of activated carbon from locally available waste materials with little or no economic importance is a welcome development to alleviate the problem (Njoku et al., 2014).

Over the past several years, considerable efforts have been made to develop activated carbon or similar porous materials from different sources for 2,4-D removal from wastewater. Jung et al. (2013) studied the metal–organic framework (Cr-benzenedicarboxylate) as an adsorbent for 2,4-D removal from contaminated water and obtained an adsorption capacity of 556 mg/g. Koner et al. (2012) used a surface-modified silica gel from factory waste as an adsorbent to remove 2,4-D from agricultural runoff of a tea industry. In a recently published study, cotton and filter paper were used to produce carbonaceous nano-materials, for 2,4-D adsorption (Khoshnood and Azizian, 2012). Oil palm frond activated carbon was successfully prepared for the removal of 2,4-D from aqueous solution through batch and fixed-bed adsorption process (Salman

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et al., 2011a). The adsorption of 2,4-D from aqueous solutions using a calcined Zn–Al layered double hydroxide incorporated with Zr^{4+} were studied by Chaparadza and Hossenlopp (2011). 2,4-D was also removed by pumpkin seed hull activated carbon (Njoku et al., 2013), corncob activated carbon (Njoku and Hameed, 2011), and palm date pits activated carbon (Salman and Abid, 2013). The use of granular-activated carbon had also been highlighted recently in literature (Pirsaheb et al., 2013).

Langsat (*Lansium domesticum*) is a tropical multipurpose tree species with an erect and short trunk, reaching 10–15 m in height, and represented by the *Meliaceae* family. The tree is indigenous to Southeast Asia and widely cultivated in southern peninsular Malaysia (Lim, 2012). Other than its value as food, various medicinal properties have been attributed to its leaves, bark, seed, and other parts (Morton, 1987). Langsat empty fruit bunch has no known economic importance, and reports have indicated that more than 10,264 metric tons of empty fruit bunch waste are produced in Malaysia per year (Foo and Hameed, 2012). Therefore, langsat empty fruit bunch could be an alternative precursor for activated carbon preparation. However, no information is available in the literature regarding Langsat activated carbon, which undergoes chemical activation by H_3PO_4. Therefore, this study aims to demonstrate the suitability and applicability, of Langsat empty fruit bunch activated carbon (LEFBAC) for 2,4-D removal from aqueous solutions. The adsorption mechanisms and thermodynamics under different conditions were also discussed.

2. Materials and methods

2.1. Chemicals

2,4-D (CAS. No. = 94–75–7, molecular weight = 221.04, molecular formula = $C_8H_6Cl_2O_3$, pKa = 2.87, log Kow = −0.82) with 97% purity level was acquired from Sigma–Aldrich (M) Sdn. Bhd., Malaysia. The various concentrations of appropriate solutions were made from standard stock solution (500 mg/L). The solubility of 2,4-D is 900 mg/L in water and higher solubility in different organic solvents. The pH of the solution was prepared in a range 2–12 by adding either 0.1 M HCl or 0.1 M NaOH. All dilutions were carried out by using distilled water. The 2,4-D chemical structure is demonstrated in Fig. 1a.

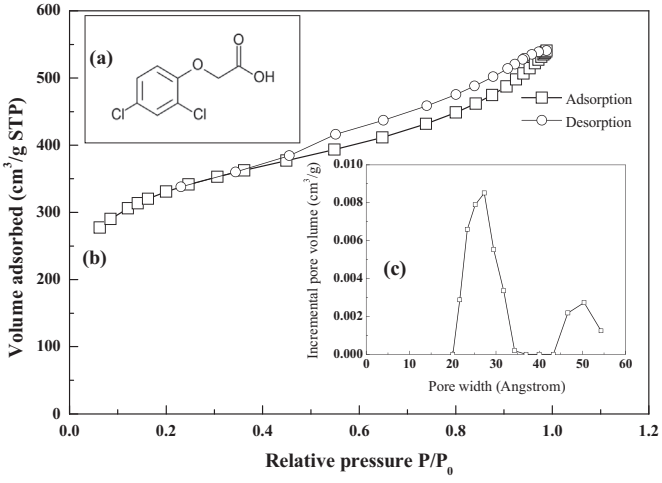

Fig. 1. (a) Chemical structure of 2,4-D, (b) N_2 adsorption–desorption isotherms and (c) pore size distribution.

2.2. Preparation of activated carbons

The raw material langsat empty fruit bunch was collected from the local market. It was then cleaned with distilled water and air-dried for 3 days. Afterward, dry samples were cut into small pieces as well as grinded to particle sizes from 0.50 to 0.71 mm and put in the oven for 24 h at 80 °C in order to remove all free water from the samples. Then, oven dried samples were impregnated with orthophosphoric acid (H_3PO_4) for 24 h. The impregnation ratio of samples to H_3PO_4 was maintained at 1:1 (w/w) and then put into the oven overnight. The activation of acid impregnated sample was done by electric vertical kiln at 500 °C for 2 h under continuous N_2 (purity, 99.995%) and flow rate of 150 cm^3/min. The heating rate was set to 10 °C/min. After certain period of time, the langsat empty fruit bunch activated carbon (LEFBAC) was collected and washed several times with hot distilled and subsequently with normal distilled water until the solution pH reached 6–7. Finally, these wet activated carbon were consigned in an oven for complete drying at 105 °C and poured into a polyethylene jar for subsequent use.

2.3. Specific characterization of LEFBAC

The surface area and pore size distribution of LEFBAC were evaluated by nitrogen (N_2) adsorption–desorption at −196 °C (77 K) by a Micromeritics, Model ASAP 2020, USA, programmed gas sorption system. The micropore volume of LEFBAC was evaluated by *t*-plot method (Lippens and deBoer, 1965) whereas the average pore width was measured according to Barrett–Joyner–Halenda method (Barrett et al., 1951).

The available active functional groups on the LEFBAC surface were monitored by Perkin Elmer, Model 2000, USA, Fourier transform infrared (FTIR) spectrophotometer.

Scanning electron microscope (SEM) was taken to visualize the microstructure of the produced activated carbon by using Zeiss Supra 35 VP, Germany.

2.4. Adsorption experiments

The batch adsorption study was executed in a horizontal water bath shaker with varying preselected temperatures (30–50 °C). The different initial concentrations (50–400 mg/L) of 200 mL 2,4-D solution were prepared from the stock solution and then dispensed into some 250 mL stopper Erlenmeyer flasks that contained 0.2 g of LEFBAC. All Erlenmeyer flasks loaded with adsorbate solutions and adsorbent were shaken in the water bath shaker at 120 rpm for a predefined time interval. The supernatant was collected using a syringe and the 2,4-D concentration was measured by using UV–Vis spectrophotometer at λ = 283 nm.

2.5. Effect of solution pH

The pH effect of 2,4-D uptake by LEFBAC was examined at different pH ranges from 2 to 12, at a fixed LEFBAC dosage of 0.2 g, fixed initial 2,4-D concentration of 200 mg/L and temperature of 30 °C for 24 h contact time.

2.6. Adsorption kinetic studies

Kinetic studies were performed to observe the time-dependence of LEFBAC by quantification at varying time intervals until equilibrium was identified. These studies were conducted according to the method of batch equilibrium studies outlined in Section 2.4. Two kinetic models were applied to analyze the kinetic data obtained in this study. The Lagergren pseudo-first-order model (Lagergren, 1898) is represented by Eq. (1).

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