



ORIGINAL RESEARCH ARTICLE

EVALUATION OF PYROLYZED OIL PALM FROND IN THE ADSORPTION
OF RESIDUAL PHENOLIC COMPOUNDS IN FACULTATIVELY TREATED
PALM OIL MILL EFFLUENT

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ABSTRACT

Aerobic digestion is the final stage of biological treatment systems employed for treating Palm Oil Mill Effluent (POME). The presence of phenolic compounds in POME inhibits the performance of aerobic microbes. To mitigate the inhibitory effect, removing phenolic compounds at the early stage of aerobic treatment could be a workable solution. In this study, the performance of biochar from oil palm frond (OPF-BC) prepared using steam pyrolysis was investigated for the efficient removal of phenolic compounds. Adsorption kinetics and equilibrium tests were performed to assess the time required to reach adsorption equilibrium and uptake capacity for phenol, catechol and gallic acid spiked in water and facultatively treated POME. Adsorption of the phenols onto OPF-BC attained equilibrium within 2 h irrespective of the water matrix. In the water maximum adsorption capacities of 38.7, 28.2 and 26.2 mg/g were obtained for phenol, catechol, and gallic acid, respectively. The presence of background organic matter in facultatively treated POME reduced the adsorption capacities by 3-folds. The simplified equivalent background model estimated dosage to achieve 80% removal ranged between 14.4 and 18.8 g/L. The OPF-BC demonstrated efficient phenolic compounds removal performance, making it a promising and economical adsorbent for the hybrid adsorption-aerobic treatment system.

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1.0 Introduction

Palm Oil Mill Effluent (POME) is an aqueous mixture of organic acids, phenolic compounds, and particulate matter derived from palm oil fruit and cell wall fragments. The concentration of the organic matter in POME is expressed in chemical oxygen demand (COD), which ranges between 16,000 and 100,000 mg/L, and when compared to domestic sewage it is 2 folds more polluting (Alhaji *et al.*, 2016; Wu *et al.*, 2010). A series of biological degradation processes such as anaerobic, facultative and aerobic treatment systems are typically employed to reduce the concentration of the organic matter in POME before discharge to surface water (Hassan and

Abd-Aziz, 2012). Nevertheless, this treatment is ineffective for the complete removal of COD mainly due to the inhibitory effect of a high concentration of phenolic compounds on microorganisms (Chantho *et al.*, 2016; Muñoz-Palazon *et al.*, 2019), the bio-recalcitrant nature of phenolic compounds at low concentrations (Hussein *et al.*, 2001), and microbial degradation of organic acids into micropollutants such as phenolic compounds (Ghosh *et al.*, 2007; Max *et al.*, 2012). Incomplete degradation of COD may indirectly lead to the discharge of treated POME with a substantial amount of organic micropollutants into the aquatic environment.

In an effort to reduce the concentration of COD in POME to a safe discharge limit, numerous advanced treatment techniques have been investigated, mainly focusing on the feasibility of upgrading the conventional biological treatment system (Bashir *et al.*, 2019; Othman *et al.*, 2014; Tan *et al.*, 2014). Accordingly, identifying a technically and economically feasible treatment technique that can be integrated into the conventional biological treatment system is gaining tremendous research attention. Based on these criteria, the adsorption technique is generally considered to be a preferred option due to its simplicity of operation, non-generation of toxic pollutants, efficient removal of recalcitrant and organic compounds, and cost-effectiveness (De Gisi *et al.*, 2016).

Adsorptive removal of contaminants by adsorbents, mainly with activated carbon, has been widely used for wastewater treatment. Though activated carbon is considered the most effective adsorbent due to its higher surface area, the activation of its precursor and regeneration after use are associated with excessive energy consumption, and its precursor, which is mostly coal, is non-renewable. Accordingly, applying low-cost adsorbents for wastewater treatment has been the main research focus in recent years. Biochar derived from oil palm frond is an environmentally friendly and low-cost adsorbent produced from a renewable bioresource. Nevertheless, little attention is given to the adsorption of micropollutants on biochar derived from oil palm fronds (OPF-BC). In addition, limited studies are focusing on the removal of phenolic compounds from facultatively treated POME. Therefore, the objective of this study was to evaluate the effectiveness of OPF-BC in adsorbing phenol, catechol, and gallic acid from facultatively treated POME.

I. Materials and Methods

1.1 Materials

Oil palm fronds were harvested in January 2021 from the oil palm plantation of Universiti Putra Malaysia in Selangor, Malaysia. The fronds were cut into approximately 5 cm and dried in an oven (Memmert, Germany) for about 48 h at 100 °C. The dry fronds were stored at room temperature in an airtight plastic bag before biochar production (Figure 1). Effluent from facultatively treated POME was collected in February 2021 from FELDA Palm Oil Mill, Pasoh. The wastewater sample collected was filtered to remove suspended solids using Whatman's filter (0.45 µm) (Figure 1). The initial contents of 1001.57 ± 51.4 mg/L for COD, 42.19 ± 0.62 mg/L for total phenolic compounds, and 8.47 ± 0.03 for solution pH were recorded. Subsample from the filtered facultatively treated POME was diluted to 1:1 with ultrapure water (Arium mini Lab Water System). A mM phosphate-buffered ultrapure water with 8.0 ± 0.2 was prepared by dissolving the required amounts of K_2HPO_4 and KH_2PO_4 in a known quantity of ultrapure water. Phenol, catechol and gallic acid used in this study as adsorbates were selected based on their residual concentration in facultatively treated POME. All chemicals used were of analytical grade. Sodium carbonate, dipotassium phosphate, monopotassium phosphate and Folin-Ciocalceou phenol reagent were purchased from Merck, gallic acid was purchased from R & M, while phenol and catechol were purchased from Scharlau and TCI respectively.



Figure 1. Feedstocks: (a) oil palm fronds, (b) facultatively treated POME

1.2 Methods

1.2.1 Production of biochar

The OPF-BC was produced by steam pyrolysis in a locally fabricated steam carbonization system. A detailed description of the system was described elsewhere (Lawal et al., 2020a; Lawal et al., 2020b). The dry oil palm fronds weighing about 250 g were loaded into a reactor and placed in the vertical tubular furnace of the carbonization unit. About 100% saturated steam was allowed to flow at 200 cm³/min into the furnace to remove air in the reactor and to maintain a steam atmosphere during pyrolysis. The dry frond sample was heated to 600 ± 5 °C at a 10 °C/min heating rate and held for 2 h and finally cooled to room temperature.

1.2.2 Adsorption kinetic test

Adsorption kinetic experiments were carried out by adding a fixed dose (4 g/L) of OPF-BC into 100 mL of water matrix of a known concentration of a phenolic compound contained in a 250 mL Erlenmeyer flask. The Erlenmeyer flask was covered with aluminium foil and transferred into a shaker incubator (TECH-LAB TSI-285, Malaysia) and shaken at 150 rpm and in a controlled temperature atmosphere of 30 °C. About 10 to 12 samples of approximately 1 mL were collected at different time points from the mixture and analyzed for residual concentration.

1.2.3 Equilibrium isotherm test

A bottle point experiment with variable biochar doses (1 to 50 g/L) was also used to evaluate the adsorption capacity of phenol, catechol, and gallic acid spiked in phosphate-buffered ultrapure water onto OPF-BC. The effect of background organic matter on the adsorption of individual phenolic compounds spiked in samples of the facultatively treated POME water matrix was also investigated. For each water matrix, a 25 mL sample was added to a known amount of OPF-BC in a 60 mL glass bottle vial and sealed with a rubber stopper. Glass vials without OPF-BC are designated as control. The sealed vials were vigorously shaken by hand and transferred into a shaker incubator (TECH-LAB TSI-285, Malaysia) and shaken at 150 rpm for 2 h at 30 °C. After equilibrium was attained, the water matrix from each vessel was filtered through a 0.22 µm syringe filter to remove the added OPF-BC and analyzed for residual concentrations of an adsorbate. Both kinetic and equilibrium isotherm experiments were repeated three times under similar conditions, and average values with their standard deviations were reported.

1.3 Analysis

1.3.1 Water matrices

Concentrations of phenol, catechol and gallic acid in the filtered water matrices were quantified using the modified Folin-Ciocalceau method (Ergül et al., 2009). Herein, 500 µL of

Folin-Ciocalceau phenol reagent diluted 1:3 with ultrapure water was added to a 100 μL water matrix. After 5 minutes, 500 μL sodium carbonate solution (2000 mg/L) was added and the mixture was allowed for the reaction to complete for 30 minutes at room temperature. Absorbance was measured by a UV-vis spectrophotometer (GENESYS 20, ThermoFisher Scientific, US) at the wavelength of 725 nm for gallic acid and total phenolic compounds and 760 nm for phenol and catechol against blank. The measured absorbance was used to calculate the concentration of an analyte based on its calibrated curve developed using 10 concentration points spread over the concentration range of 5 to 50 mg/L. The coefficient of determination of the curves was > 0.998 . The concentration of COD in facultatively treated POME was determined based on APHA (American Public Health Association) standard method 8000. This involved adding 2 mL of filtered water matrix into a COD vial (20 to 1500 mg/L) and heated to 150 $^{\circ}\text{C}$ in a HACH reactor (HACH, USA) for 2 h. After cooling, COD concentration was measured using the HACH DR 2800 spectrophotometer (HACH, Germany).

1.4 Modeling

Data obtained from the equilibrium isotherm experiments in phosphate-buffered ultrapure water were simulated using the Freundlich model

$$q_e = K_F(C_e)^n \quad (1)$$

where q_e (mg/L) is the solid-phase concentration of adsorbate at equilibrium, C_e (mg/L) is the aqueous-phase concentration of adsorbate, and K_F [(mg/g)(mg/L) n] and n are Freundlich parameters for capacity affinity and linearity respectively.

In a situation where the isotherm shifts of different adsorbates on an adsorbent are relatively non-parallel (n values varied significantly), the concentration-specific distribution ratio expressed as

$$K_{csd} = \frac{q_e}{C_e} \quad (2)$$

is more appropriate for affinity comparison than K_F (Lattao *et al.*, 2014).

Since the Freundlich model can not adequately predict the competitive adsorption process, the simplified equivalent background compound model (SEBCM) was found to be suitable to describe the adsorption of a target compound in the presence of a higher concentration of background organic matter in the wastewaters (Qi *et al.*, 2007). The SEBCM relates the residual concentrations of the targeted compound to the adsorbent dose in a 2-parameter model:

$$\ln\left(\frac{C_{1,0}}{C_1} - 1\right) = \frac{1}{n_1} \ln \frac{m_A}{V} - \ln A \quad (3)$$

where $C_{1,0}$ and C_1 are the initial and equilibrium concentrations of adsorbate, n_1 is the Freundlich linearity parameter, m_A mass of adsorbent, V volume of solution and A equation parameter. The parameters n_1 and A obtained by fitting experimental data in eq. (3) are important parameters required in eq. (4) to determine the adsorbent dose necessary for the removal of a certain percentage of target adsorbate (Bonvin *et al.*, 2016; Qi *et al.*, 2007):

$$\frac{m_A}{V} = A^{n_1} \left(\frac{C_{1,0}}{C_1} - 1\right)^{\frac{1}{n_1}} \quad (4)$$

2. Results and Discussion

2.1 Adsorption Kinetics

Adsorption kinetic test was carried out to estimate the time required to reach equilibrium. The experimental results showed that adsorption of phenol, catechol and gallic acid in the

phosphate-buffered ultrapure water and facultatively treated POME onto OPF-BC attained equilibrium within 2 h (Figure 2). In phosphate-buffered ultrapure water, phenol recorded the highest initial percentage removal of 80% and attained closed to adsorption equilibrium after 40 min, followed by catechol, with a similar trend but at a slightly lower percentage removal of 77%, and a higher adsorption equilibrium time of 90 min. Gallic acid exhibited the lowest initial percentage removal of 68% and reached adsorption equilibrium after 2 h. However, in facultatively treated POME, the percentage removal was drastically reduced to 46% for phenol, 55% for catechol, and 33% for gallic acid. The adsorption equilibrium for phenol was delayed to 90 min, while that for gallic acid was achieved in only 60 min, indicating possible adsorption competition and/or interference from background organic matters in the facultatively treated POME. These results are in agreement with previous studies showing an equilibrium time of less than 2 h for phenol and catechol (Alam et al., 2009; Vunain et al., 2018). Early and fast removal of phenolic compounds during aerobic treatment is an operational requirement that can benefit the entire treatment process by reducing the inhibitory effect of the phenolics on microorganisms and reducing bio-recalcitrant micropollutants.

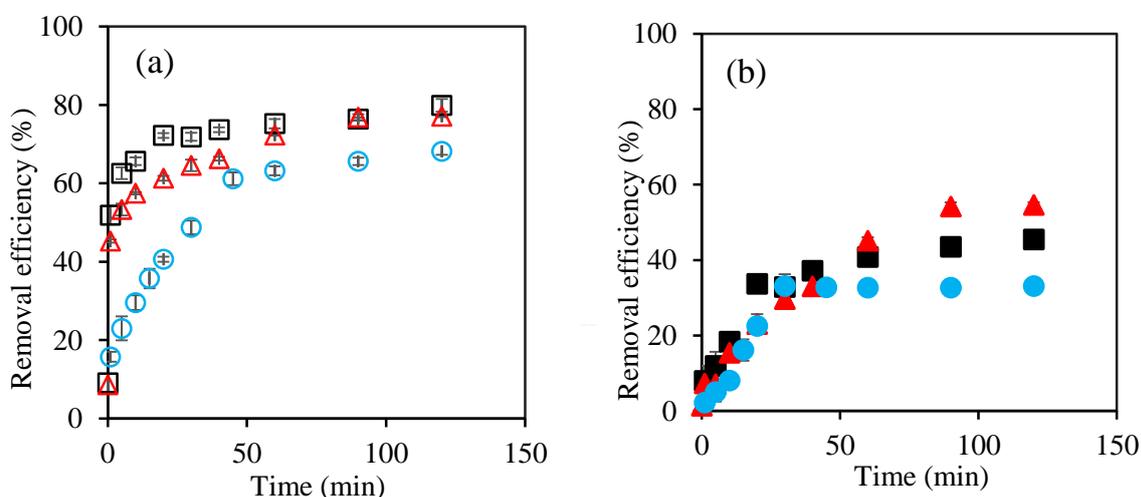


Figure 2. Adsorption kinetics of OPF-BC at 4 g/L dosages for phenol (□, ■), catechol (△, ▲) and gallic acid (○, ●) spiked in (a) phosphate-buffered ultrapure water and (b) facultatively treated POME.

2.2 Adsorption Isotherm

2.2.1 Adsorption equilibrium in water

The isotherms for the adsorption of phenol, catechol, and gallic acid from phosphate-buffered ultrapure water onto OPF-BC are presented in Figure 3a, and their respective Freundlich parameters are listed in Table I. It can be seen from Figure 3a, that the isotherms of phenol and catechol are quite similar, both showing a pore-filling mechanism at low carbon dosage. The isotherm trend for gallic acid differed considerably. Its adsorption capacity at both high and low biochar dosages decreased by more than half that of phenol and catechol indicating low OPF-BC affinity for gallic acid. The maximum experimental adsorption capacities of 38.7, 28.2, and 26.2 mg/g were obtained for phenol, catechol, and gallic acid, respectively. The values of the n parameter are highly nonlinear (<0.5) indicating a predominantly adsorption/pore-filling mechanism. The estimated capacity parameter of Freundlich, K_F will not provide a reliable basis for comparison among the phenolic compounds due to the relative non-parallel shift of the isotherms (unequal n values of the adsorbates). For a reliable comparison of estimated capacity parameters, K_{csd} values calculated at three different specific liquid-phase

concentrations were evaluated (Figure 3b). At low concentration, OPF-BC showed high affinity for the phenols in the decreasing order of catechol > phenol > gallic acid. As liquid-phase concentration increased, the affinity of OPF-BC for the phenolic compounds decreased indicating near saturation of its adsorption sites.

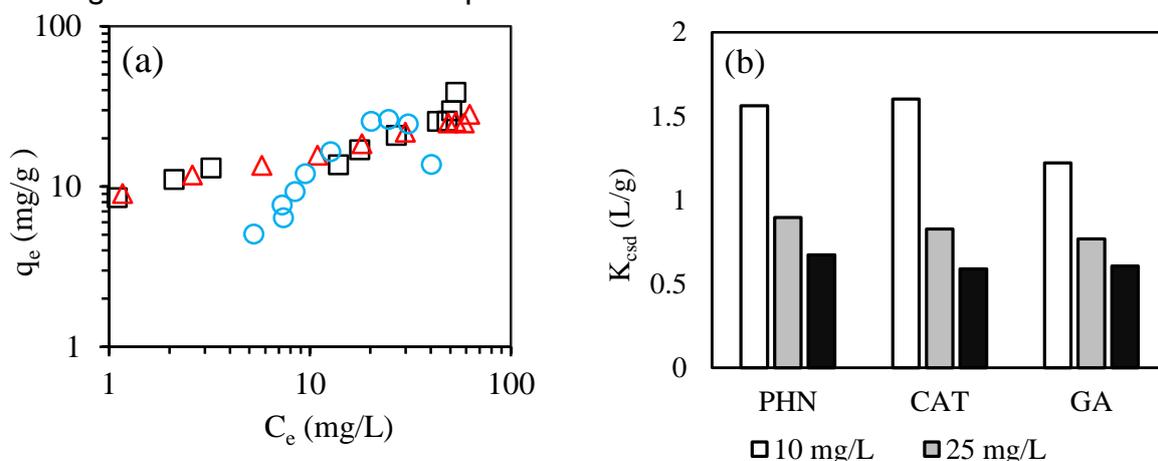


Figure 3. (a) Adsorption isotherms for phenol (□), catechol (Δ) and gallic acid (○) in phosphate-buffered ultrapure water, and (b) their respective concentration-specific distribution coefficient at 10, 25 and 40 mg/L.

Table I. Freundlich Parameters and Coefficient of Determination for Phenol, Catechol, and Gallic Acid Adsorption onto OPF-BC.

Adsorbate	Freundlich parameters		
	K_F	N	R^2
Phenol	6.333	0.3921	0.9356
Catechol	8.447	0.2779	0.9999
Gallic acid	3.9231	0.4937	0.4207

2.2.2 Adsorption equilibrium in facultatively treated POME

To study the effect of background organic matter on the adsorption capacity of phenolic compounds on OPF-BC, the adsorption isotherms for phenol, catechol, and gallic acid were determined in phosphate-buffered ultrapure water containing no organic compounds and in facultatively treated POME, containing background organic matter (Figure 4). For adsorption of the phenolics in facultatively treated POME approximately three-fold lower adsorption capacities were observed for phenol, catechol, and gallic acid, reflecting the negative effect of competing for organic matter. It is widely known that background organic matter in wastewater affects the adsorption of target micropollutants on porous carbon materials via direct competition for adsorption sites or size exclusion effect or both (Bonvin *et al.*, 2016; Zoschke *et al.*, 2011). In comparison, OPF-BC was able to adsorb approximately 1 and 3 mg/g more of catechol than phenol and gallic acid across all dosages respectively in the presence of background organic matter (Figure 3d). Due to their small size and molecular weight, phenol and catechol competed favourably well with other micropollutants in facultatively treated POME for adsorption sites located in the micropore region of OPF-BC as compared to gallic acid.

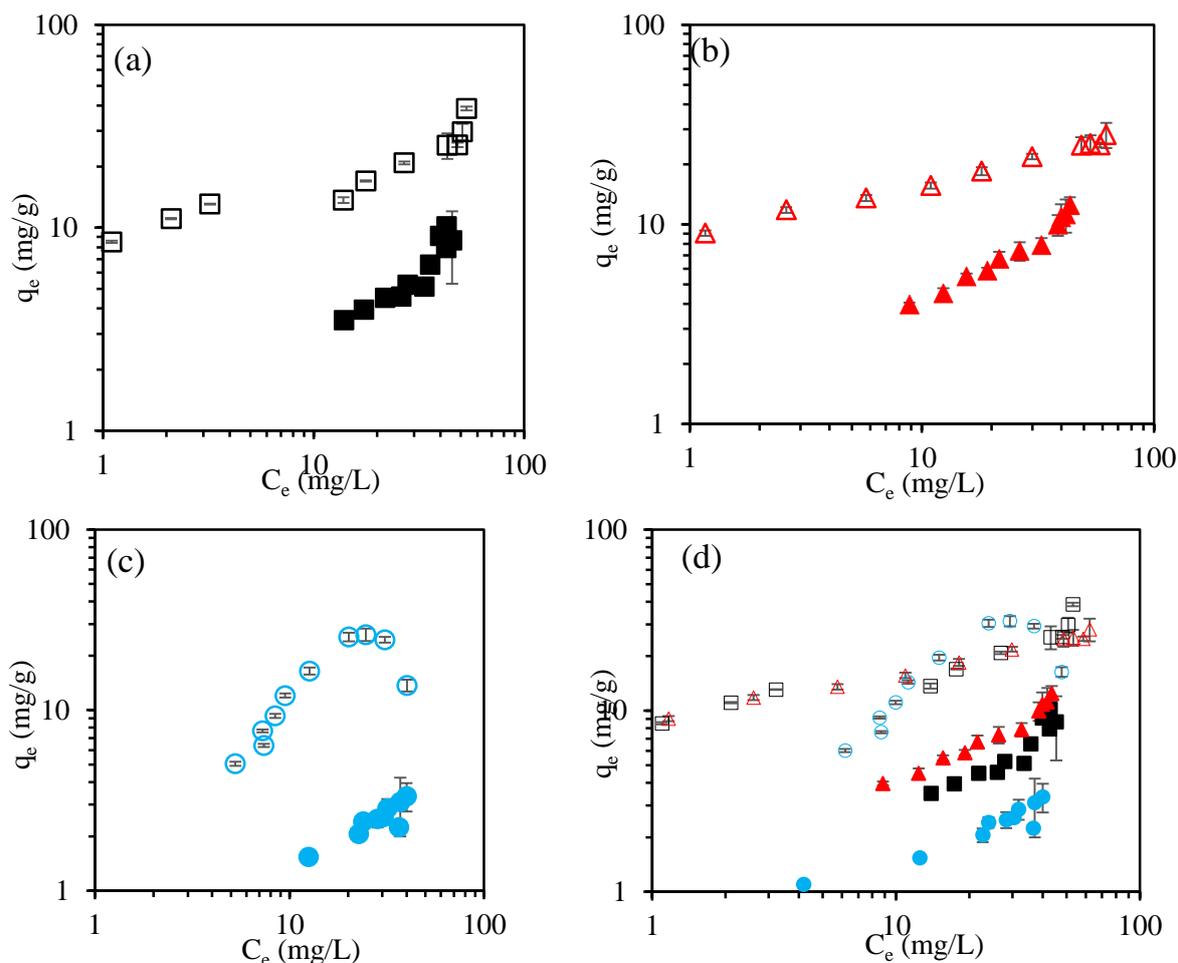


Figure 4. Comparison between adsorption isotherms of (a) phenol, (b) catechol, (c) gallic acid (d) group isotherms in phosphate-buffered ultrapure water (empty symbols) and facultatively treated POME (filled symbols).

2.3 Effect of Background Organic Matter on Removal Efficiency of Phenolic Compound

The dependence of the removal efficiency of phenol, catechol and gallic acid from phosphate-buffered ultrapure water and facultatively treated POME water matrices on OPF-BC dosage ranging from 0.4 to 10 g/L is presented in Figure 5a. At the same biochar dosage, an OPF-BC dose of 6 g/L was required to remove more than 97% phenol and catechol, and 84% gallic acid from phosphate-buffered ultrapure water, whereas the presence of background organic matter in facultatively treated POME drastically reduced their removal efficiency to 63%, 67%, and 44%, respectively. The percentage removal of COD in facultatively treated POME was comparatively lower than that of phenolic compounds (Figure 5b). To compare the effect of background organic matter on the removal efficiency of OPF-BC for the phenolic compounds, their removals were examined in both the facultatively treated POME and its half-diluted sample at three biochar doses. Figure 6 shows the competitive effect of increasing COD concentration on the removal efficiency of the phenols at three different doses of OPF-BC. For all doses of OPF-BC, an increase in COD concentration reduced the removal efficiency of the phenolic compounds. This is in line with the findings of Bonvin *et al.* (2016) on the removal of micropollutants in varying concentrations of background organic matter. Dilution reduces the concentration of background organic matters which compete for adsorption sites or block pores and thus reflecting an increase in available adsorption sites. Alternatively, increasing adsorbent dosage is expected to reduce the difference between removal efficiencies

of adsorbate in wastewater with different concentrations of background organic matter because of the additional adsorption sites provided. This trend was observed with the removal of catechol where the difference in its removal efficiency in facultatively treated POME and half-diluted facultatively treated POME decreased at high dosage (Figure 6c). However, this is not the case with gallic acid where the difference in removal efficiency increases at high dosage indicating its low affinity for OPF-BC.

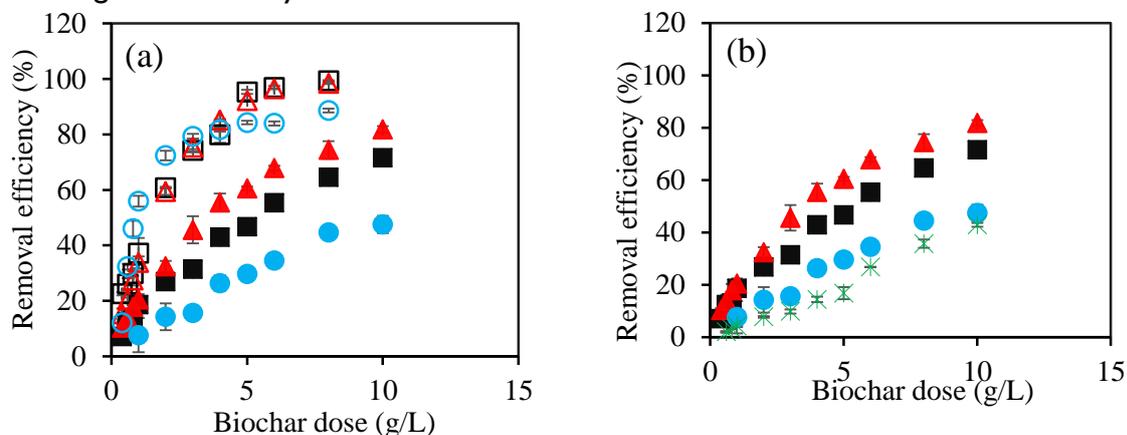


Figure 5. Comparison of the dependence of removal efficiency on OPF-BC dose: (a) among phenol (\square , \blacksquare), catechol (Δ , \blacktriangle) and gallic acid (\circ , \bullet) spiked in phosphate-buffered ultrapure water and facultatively treated POME, (b) among phenol, catechol and gallic acid spiked in facultatively treated POME and its COD (\ast) content.

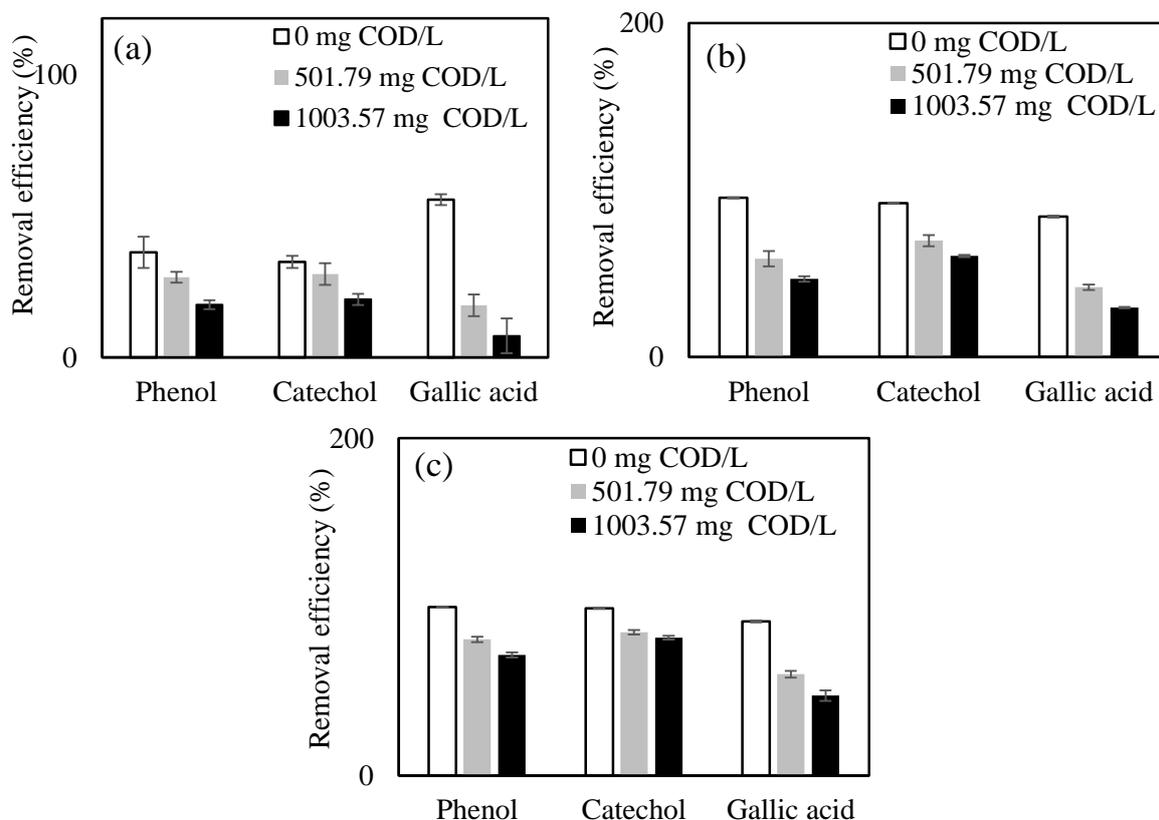


Figure 6. Effect of COD concentration on the removal efficiency of phenol, catechol and gallic acid at: (a) OPF-BC dose (1 g/L); (b) OPF-BC dose (5 g/L); and (c) OPF-BC dose (10 g/L).

2.4 Biochar Dose for Efficient Removal of Phenolic Compound in Facultatively Treated POME

To accurately estimate the quantity of OPF-BC dose required for efficient removal of the phenolic compounds spiked in facultatively treated POME, SEBCM was first employed to simulate the relative removal of experimental data (Figure 7). The removal plots predicted by SEBCM fit well with the experimental data as shown in Figure 7, indicating the validity of extending the application of SEBCM for wastewater containing a high concentration level of background organic matter. The parameters obtained from fitting the experimental data were then utilized to quantitatively estimate the OPF-BC dose required for 80% removal using eq. (4), and the results are presented in Table 2. The OPF-BC dose required to remove 80% ranges from 14 to 19 g/L with an average dose of 15.87 g/L to remove all compounds combined. Though the estimated quantity of OPF-BC seems large (14 -19 g/L) compared to activated carbon doses (15 - 21 mg/L) reported for the 80% removal of benzotriazole, mecoprop, and sulfamethoxazole from wastewater (Altmann et al., 2014; Bonvin et al., 2016), the performances of the adsorbents are comparable considering the concentration levels of the respective target pollutants and background organic matter. In this study, concentration levels are three-fold larger (mg/L against $\mu\text{g/L}$), corresponding to the three-fold higher dosage. The result indicates that efficient removal of phenolic compounds to below inhibitory level can be achieved with a low OPF-BC dose and this underscores the possibility of the practical application of the adsorption process in large scale aerobic treatment system.

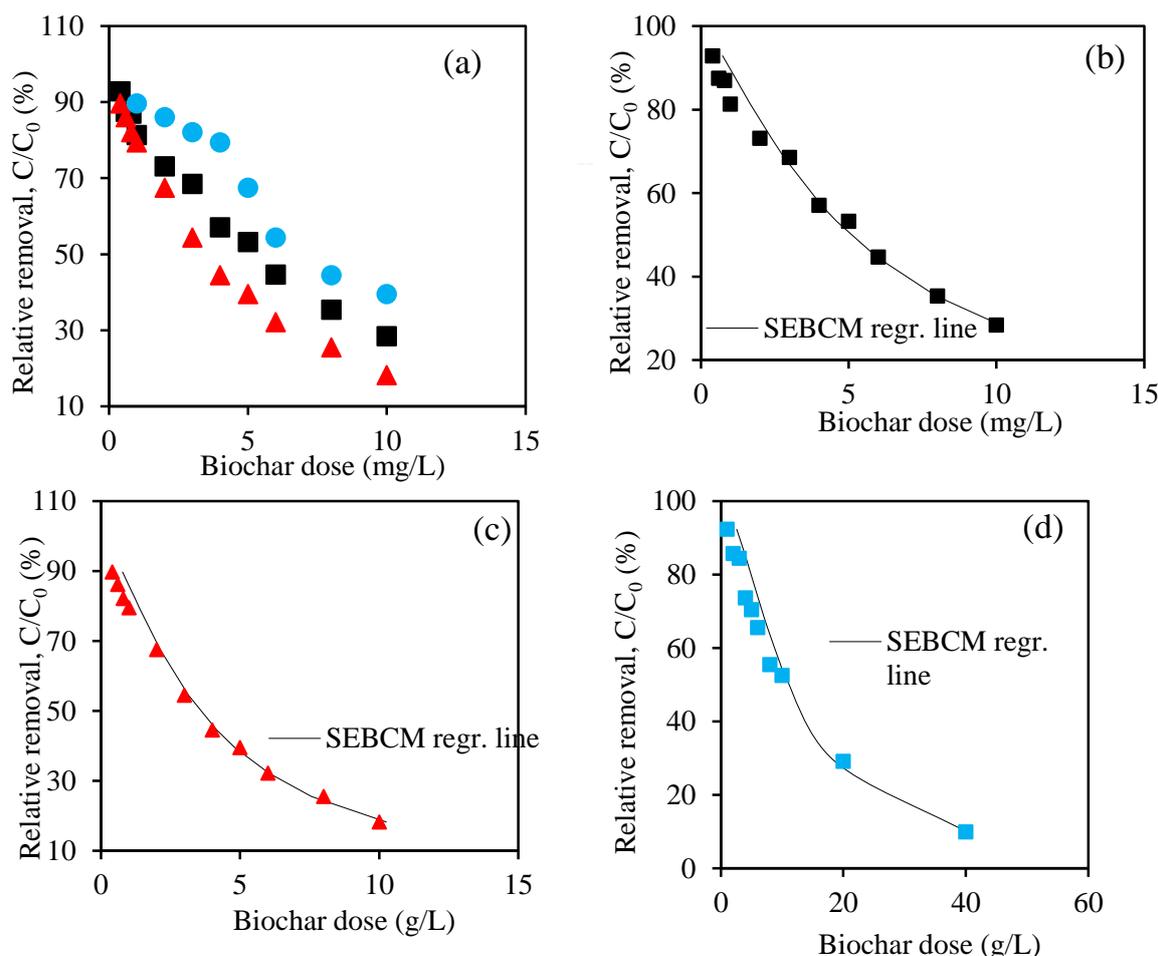


Figure 7. (a) Group plots of experimental data for the relative removal of phenol, catechol and gallic acid in facultatively treated POME, and SEBCM simulation of relative removal of (b) phenol, (c) catechol and (d) gallic acid in facultatively treated POME.

Table 3. SEBCM Parameters and Goodness of Fit for Phenol, Catechol and Gallic Acid, and their Estimated 80% Removal.

Adsorbate	SEBCM parameter			Estimated OPF-BC dose for 80% removal (g/L)
	A	n_i	R^2	
Phenol	8.6478	1.3298	0.9434	14.36
Catechol	6.0761	1.4199	0.9603	14.48
Gallic acid	62.35	1.717	0.969	18.81

3. Conclusion

The fast adsorption kinetics of OPF-BC for phenolic compounds will prevent the initial inhibition effect on the aerobic microbes, which will lead to a potential reduction of hydraulic retention time of the aerobic process. For instance, an aerobic process usually takes 2 to 10 days to degrade phenolic compounds, whereas complete removal will be achieved within 2 h using OPF-BC. Therefore, the initial removal of phenolic compounds using OPF-BC during aerobic treatment will benefit the entire process in terms of reducing the time for treatment. Another benefit arising from adsorbing more than 43% COD in facultatively treated POME will be a plus to the primary objective of removing phenolic compounds. The removal of close to half the concentration of COD in facultatively treated POME reduces the organic loading during aerobic treatment, which could translate to a reduction in the size of the aerobic tanks.

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