Effectiveness of Oil Palm Frond Activated Carbon for Removing COD, Color and Fe from Landfill Leachate

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Highlights:

- OPF-AC as a new adsorbent in leachate treatment
- High surface area and micropores developed on OPF-AC surface
- Potential of COD, color and Fe removal up to 70%

Abstract. This study examined the applicability of oil palm frond-activated carbon (OPF-AC) as adsorbent in the treatment of landfill leachate. OPF-AC was prepared by mixing granular OPF with an aqueous solution composed of distilled water and zinc chloride ($ZnCl_2$) at different ratios. Batch experiment studies were carried out to investigate the optimum OPF-AC dosage as well as the optimum shaking time. The optimum condition was achieved at 1.2 g of OPF-AC dosage and 50 minutes of shaking time. The highest removals of COD, color and Fe (77%, 75% and 69%, respectively) were obtained at the optimum condition. Meanwhile, the adsorption capacities were 146.93 mg/g (COD), 56.43 mg/g (color) and 0.11 mg/g (Fe). The increase in adsorbent dosage also resulted in a larger free surface area and exchangeable binding sites. However, the unsaturation of adsorption sites during the adsorption process resulted in a slight drop in removal efficiency of COD, color, and Fe. Furthermore, due to the aggregation and overcrowding of adsorbent particles, the use of a greater amount of adsorbent dosage imposed particle interactions.

1 Introduction

Leachate is a by-product resulting from solid waste due to its physical, chemical and biological transformation. It can be produced with high toxicity in landfills, incineration plants and transfer stations [1]. It is often characterized by high-

resistance wastewater with high pH, chemical oxygen demand (COD), biochemical demand for oxygen (BOD_5), inorganic salts and toxicity [2,3]. The characteristics of leachate can vary within a landfill because of several conditions, such as the surrounding environment, the composition of the landfill, and the compaction of solid waste [4].

In general, the leachate produced in the primary stages of waste decomposition includes a high concentration of BOD_5 and high amounts of biodegradable and non-biodegradable materials [3,5]. On the other hand, leachate produced from old landfills also contains non-biodegradable organic compounds such as fulvic and humic-like substances, which are usually measured as COD, as well as high ammonia-nitrogen (NH_4-N) concentrations [6,7]. Numerous treatment methods have been reported, such as physical, chemical, biological treatment, and integrated methods using the combination of two or more methods [4,8].

Activated carbon (AC) is the physical/chemical treatment that is the most widely employed in leachate treatment. A significant trend in the production of AC as adsorbent was promulgated because of its superior capacity in adsorbing a wide range of organic and inorganic contaminants dissolved in aqueous media due to its large specific surface area, controllable porous structure, low acid/base reactivity, and thermo-stability [9]. However, commercial AC is usually costly. The use of agricultural by-products as raw materials for AC production could potentially be cheaper since these raw materials are renewable and considered waste [10-12].

A previous study by Poblete, *et al.* [12] showed that the use of coffee-based activated carbon to mitigate the color and COD from landfill leachate was effective in the treatment of landfill leachate. Subsequently, Abdul Halim, *et al.* [13] applied a fixed-bed column using AC by allowing leachate samples to pass through until saturation for ammonia removal. They found that this method was effective in removing ammonia up to 87%. In a study conducted by Ferraz and Yuan [5], a granular activated carbon was prepared using chemical activation of oat hulls to treat landfill leachate. The proportional relationship was established between the removal efficiency of color and COD and the adsorbent dosage and contact time, with maximum removals of color and COD of 91% and 76% respectively, using 20 g/L at pH 4.

The current study was aimed at assessing the effectiveness of AC produced from oil palm frond (OPF) activated with $ZnCl_2$ for the treatment of landfill leachate. The advantage of using agricultural by-products as raw materials for AC production is that they are renewable and potentially less expensive to produce.

2   Materials and Methods

2.1   Landfill Leachate Sampling

Samples of leachate were obtained from the Pulau Burung Sanitary Landfill (PBSL) located in the Byram reserve in Penang, Malaysia with coordinates 5°24′ N and 100°24′ E. The total site area is 62.40 ha, where currently 33.00 ha are operated [4]. The site annually receives around 1800 tons of municipal and industrial solid wastes. The composition of the PBSL site mainly consists of marine clay liner, since the landfill is located near the seashore. From the 1980s to 1990, the site lacked proper management and leachate control; thus, the waste was disposed without a suitable plan. In 1991, PBSL began operating as a semi-aerobic system that met the requirements of a level-II sanitary landfill by introducing a controlled tipping method. In 2001, the PBLS level was elevated to level III by applying controlled tipping with recirculation of the leachate. In 2012, PBSL's operations and management were transferred and awarded to a private company to ensure smooth and efficient day-to-day service and landfill management [2,14].

Sample collection was performed directly from the pond using a bucket. All samples were filled into HDPE bottles and kept at 4 °C. The preserved samples were allowed to condition at room temperature (25 °C) before starting the experiment. The main characteristics of leachate, such as pH, turbidity, suspended solids (SS), COD, BOD_5, color, and iron (Fe), were measured. All analytical procedures were carried out following the Standard Methods for Examination of Water and Wastewater (APHA, 2005) [15]. A pH test was performed using a portable pH meter while a DR/2800 spectrophotometer was used to measure turbidity, suspended solids (SS), COD, and color. The BOD_5, COD and Fe were assessed using standard methods APHA 5210B, 5220D and 3111B, respectively.

2.2   Preparation of OPF-AC

Dried oil palm frond (OPF) was obtained, blended, and sieved to remove impurities and dust. A total of five beakers were prepared and 10 g of OPF was inserted into each beaker. The OPFs were impregnated with $ZnCl_2$ in varying weight ratios from 1:1 to 1:5 (1:1, 1:2, 1:3, 1:4 and 1:5). Then, distilled water was added up to 100 ml of the beaker and continuously stirred for one hour. Thereafter, the mixture was filtered and then placed inside a crucible with a lid before heating in an oven for 5 hours. The crucible was then removed from the oven and placed in a muffle furnace for 1 hour at different temperatures (300 °C, 400 °C and 500 °C). Then, the boiled distilled water was used to wash the activated OPF until the pH reached 7-8. It was then dried again in the oven for 24

hours, where the end product obtained was OPF-AC. The OPF-AC produced was in granular form and was then blended into powder to be used to treat the leachate through batch treatment. This method has previously been carried out by Sahin and Saka [16] to produce impregnated acorn shell samples of AC from $ZnCl_2$ using a chemical activation method with a temperature range of 300 to 600 °C.

After preparing the OPF-AC, an iodine test was carried out at different activation temperatures of 300 °C, 400 °C, and 500 °C. A total of 15 samples were tested in order to identify the maximum iodine number to indicate the AC's porosity. The theory behind this test is based on the three-point adsorption isotherms. The iodine number can be identified at a residual iodine concentration of 0.02 N based on the amount of iodine adsorbed (mg/g carbon) [17]. Then, the general characteristics of OPF-AC were investigated. The OPF-AC surface morphology and elemental composition were examined with a scanning electron microscope (SEM) and energy-dispersive X-ray (EDX) spectroscopy. Fourier transform-infrared (FTIR) spectroscopy was used to analyze the functional groups of the OPF-AC and Brunauer–Emmett–Teller (BET) was employed to investigate the porosity of the OPF-AC [11,12,18].

2.3 Batch Studies

Experiments were conducted using different OPF-AC dosages (1.0, 1.2 and 1.4 g) and shaking times (10, 20, 30, 40, 50 and 60 minutes), respectively. The OPF-AC was prepared in powder state; the dosage range was chosen based on a previous study [18]. Fifty milliliter of leachate was mixed with the different OPF-AC dosages in a conical flask and shaken with different shaking times using a digital orbital shaker at 200 rpm. After the samples were appropriately shaken, settling was allowed for 90 minutes before filtering using Whatman 2V filter paper prior to analysis for COD, color and Fe removal. A graph was plotted to obtain the optimum dosage of OPF-AC and the maximum removal of pollutants.

3 Results and Discussions

3.1 Leachate Characteristics

Table 1 summarizes the characteristics of PBSL. The range of COD (2245-2253 mg/L) was higher than the Malaysian Environmental Quality Act 1974 standard discharge limit (400 mg/L) [19]. The high content of COD suggests that the leachate sample contained a high volume of oxidizable organic material, which decreases the levels of dissolved oxygen (DO) until it is released into water bodies [20]. A reduced DO level can lead to anaerobic conditions that are dangerous to higher forms of aquatic life.

The leachate had a pH level of 8.10 to 8.15, which is within the standard discharge limit of Malaysia Environmental Quality Act 1974 (pH 6 to 9) [18]. The data complied with the pH level of old leachate (landfill age > 10 years), pH > 7.5 [3]. Besides, the SS was also higher than the accepted range for leachate disposal (SS = 137 to 141 mg/L), where the accepted range for SS disposal is 50 mg/L.

Meanwhile, the turbidity was in the range of 45 to 50 NTU. The amount of SS is strongly correlated to the amount of turbidity, where a high SS results in a high turbidity value [4]. This is due to particles suspended in the polluted water, mainly from adsorbed organic compounds and minerals. The high color pollution (3920 to 3926 PtCo) present in the leachate results from the presence of organic-metal compounds, humic acids, and microbiological matter [5,7].

Table 1 Leachate characteristics.

Parameter	Unit	Minimum values	Maximum values	*Standard
pH	-	8.10	8.15	6.9
BOD_5	mg/l	10.00	12.00	20
COD	mg/l	2245.00	2253.00	400
Color	Pt-Co	3920.00	3926.00	100
Suspended Solids (SS)	mg/l	137.00	141.00	50
Turbidity	NTU	45.00	50.00	-

*Standard Based on Environmental Quality (Control of Pollution from Solid Waste Transfer Station and Landfill) Regulations 2009 (PU(A) 433) [18]

3.2   Iodine Test

An iodine test was performed to assess the porosity of the OPF-AC surface. The iodine numbers were obtained for varying ratios of OPF to $ZnCl_2$ at different carbonization temperatures (300 °C, 400 °C, and 500 °C), as shown in Figure 1.
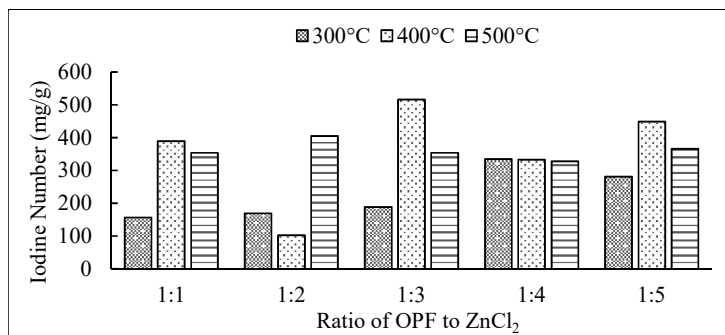

Figure 1   Iodine test results for the OPF-AC at carbonization temperatures of 300 °C, 400 °C, and 500 °C.

The highest iodine adsorption of 516 mg/g was obtained at a ratio of 1:3 and a carbonization temperature of 400 °C. At a carbonization temperature of 300 °C, a ratio of 1:4 yielded the highest iodine number at 335 mg/g. Meanwhile, the maximum iodine number at 405 mg/g was obtained with a ratio of 1:2 and a temperature of 500 °C. According to Hijazi, *et al.* [17], the amount of iodine may be represented as milligrams of iodine adsorbed by 1.0 g of carbon when the filtrate iodine concentration is 0.02 N (0.02 mol/L).

The optimum ratio obtained at 1:3 presents a sufficient amount of $ZnCl_2$ in OPF for the highest performance in iodine absorption, which is related to the porous structures developed on the OPF-AC surface [9]. However, it can be seen that an increase in impregnation ratio does not increase the activated carbon iodine number due to the destruction of pores by the excess activating agent [10]. The highest iodine number was not achieved at the highest activation temperature, i.e. 500 °C, which could be due to over-burning, which eventually affects the number of micro-pores and may influence the adsorption capacity [16]. Besides, overheating (> 400 °C), decreases the surface area of OPF-AC due to the shrinkage of the carbon [11,21,22].

3.3 OPF-AC Characteristics

The optimum condition of OPFA-AC was identified at a 1:3 ratio of OPF to $ZnCl_2$ with a carbonization temperature of 400 °C prior to the BET analysis. Figure 2 shows the N_2 adsorption isotherms of the raw OPF and the OPF-AC. The graph presents OPF-AC categorized as a type 1 exclusively microporous material, which is a common characteristic of AC [9].
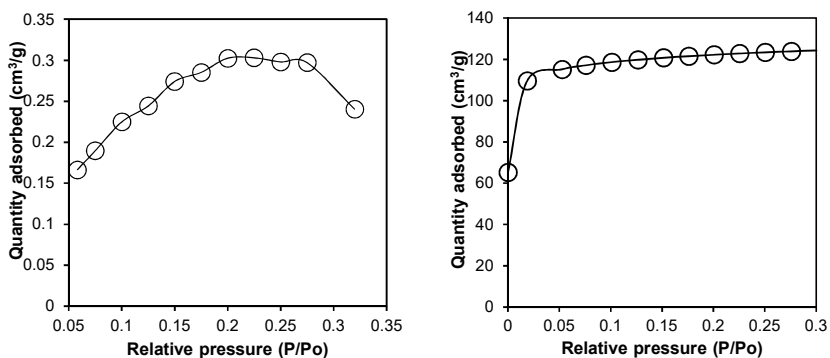

Figure 2 N_2 adsorption isotherm at temperature 295 K for (a) raw OPF, and (b) OPF-AC.

A previous work by Under, *et al.* [23] concluded that watermelon rind with $ZnCl_2$ activation classified as isotherm type I and IV at carbonization temperatures of 600 °C and 700 °C. They concluded that the activation by $ZnCl_2$ on AC tend to create mesoporous and microporous structures. In contrast, the isotherm behavior of raw OPF indicated a non-porous material. It had no micropores present on its surface, as shown in Table 2 [24]. The BET surface area was found to be 0.86 m^2/g, and 388.98 m^2/g for the raw OPF and the OPF-AC, respectively. Meanwhile, the pore volume for the OPF-AC was 0.16 cm^3/g, while 0.000001 cm^3/g was attributed to the raw OPF.

Spagnoli, *et al.* [22] found nutshell impregnated with $ZnCl_2$ at a ratio of 2:1, contributed to a BET surface area of 857 m^2/g, while cherry stone activated with $ZnCl_2$ (1:3) displayed a higher surface area (1071 m^2/g) [21]. Brito, *et al.* [11] have reported that yellow mombin stone activated with phosphoric acid (409 m^2/g) had a higher surface area compared to when activated by potassium hydroxide (222 m^2/g) at a temperature of 500 °C. Remarkably, the surface area of an adsorbent does not only depend on the type and ratio of activating agent, but is also influenced by the lignocellulose properties of the precursor employed [25,26].

Table 2 Summary of BET analysis.

Material	Raw OPF	OPF-AC
BET surface area (m^2/g)	0.8556	388.97
Micropore surface area (m^2/g)	-	320.47
External surface area (m^2/g)	0.9846	68.51
Pore volume (cm^3/g)	0.000001	0.16

The surface morphology of the raw OPF and the OPF-AC were investigated by SEM analysis, as shown in Figure 3. Significant differences were observed on both surfaces. The rough OPF-AC surface obtained was an effect of $ZnCl_2$ activation [22]. Ahmed, *et al.* [10] observed the same features on slash pinewood activated with $ZnCl_2$. They concluded that the presence of the flake-like structure on the AC surface was due to the development of new active sites. Meanwhile, the smooth surface shown by the raw OPF was identical to that in an SEM image of raw foxnut shell without activation [9]. An increase of the pore size leads to an increase of the AC adsorption capacity [16,21].

Table 3 illustrates the elemental composition from the EDX analysis of the OPF-AC. As shown in Table 3, it can be seen that the OPF-AC contained a high amount of carbon in atomic percentage (71.60%), which is a relatively good characteristic for an adsorbent. It also has a direct influence on the carbon porosity [11,13].

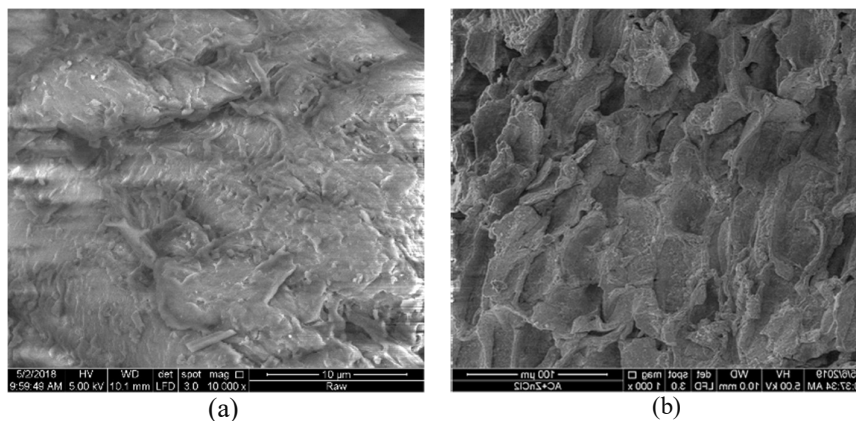(a)   (b)

Figure 3   Surface morphology of (a) raw OPF, and (b) OPF-AC.

Table 3   Elemental composition from EDX analysis of the OPF-AC.

Element	Weight (%)	Atomic (%)
C K	61.45	71.60
O K	29.32	25.65
Al K	0.36	0.18
Cl K	2.91	1.15
K K	0.92	0.33
Zn K	5.05	1.08
Totals	100.00	100.00

Figure 4 shows the FTIR spectra of the raw OPF and the OPF-AC. The atoms in the molecules were not static. However, even in a solid state they vibrate about their equilibrium positions. Each atom vibrates at a frequency depending on its weight and the length and strength of its bonds [25]. More peaks were observed in the raw OPF compared to the OPC-AC. This occurs due to burning off during activation of $ZnCl_2$, where the carbonization process results in the elimination of peaks [11]. The broad band between 3388.93 cm^{-1} and 3363.86 cm^{-1} indicates O-H stretching of hydroxyl groups and that adsorbed water has been assigned [13].

C=C aromatic ring stretching vibration was assigned based on the high band of 1600 cm^{-1} to 1800 cm^{-1}, enhanced by polar functional groups identified by the bands at 1712.79 cm^{-1}, 1618.28 cm^{-1} and 1589.34 cm^{-1} [5]. The bands at 1369.46 cm^{-1} and 1352.10 cm^{-1} indicate C-O stretching in the aromatic ring [20]. The C-H in alkane and alcoholic C-O stretching vibrations are indicated by the bands at 1249.87 cm^{-1} and 1033.85 cm^{-1}, respectively. Meanwhile, the bands at 887.26 cm^{-1}, 781.17 cm^{-1} and 599.86 cm^{-1} indicate C-H wagging [12]. These functional groups feature OPF-AC as an aromatic polymer; they act as active sites, which

tend to increase the interactions between pollutants and the adsorbent, thus increasing the removal of pollutants [5,22].
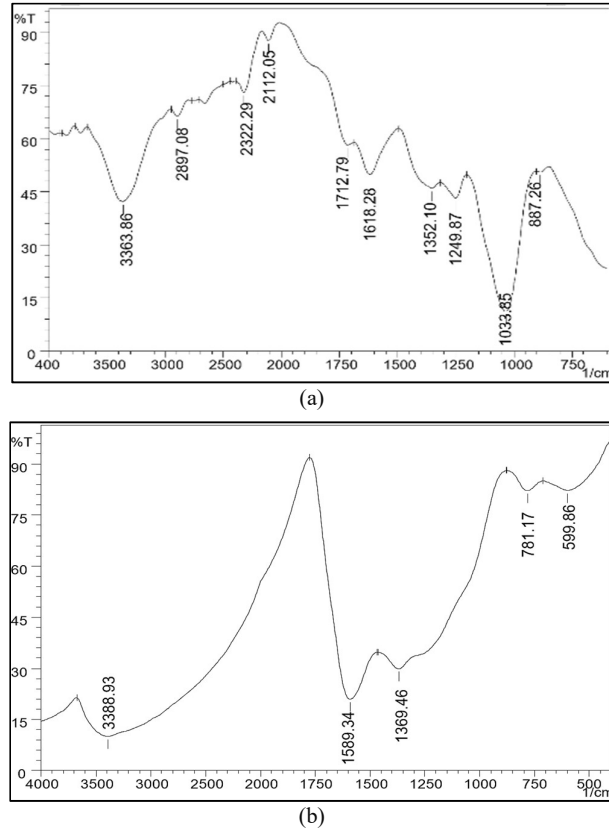

(a)

(b)

Figure 4 FTIR spectra of (a) raw OPF and (b) OPF-AC.

3.4   Batch Experiments Studies

Figure 5 shows the removal of COD, color, and Fe using different OPF-AC dosages of 1.0 g, 1.2 g and 1.4 g at different shaking times (40 to 60 min). The efficiency of COD, color and Fe removal consistently increased from 0 to 40 minutes of shaking time since a significant amount of organic matter can be adsorbed when the treatment continues for a more extended period. This is due to the higher surface area and active sites on the OPF-AC surface in the initial stage of the experiment. On the other hand, a shaking time of 10, 20, and 30 minutes was not enough for the OPF-AC to adsorb the pollutants [21,25].
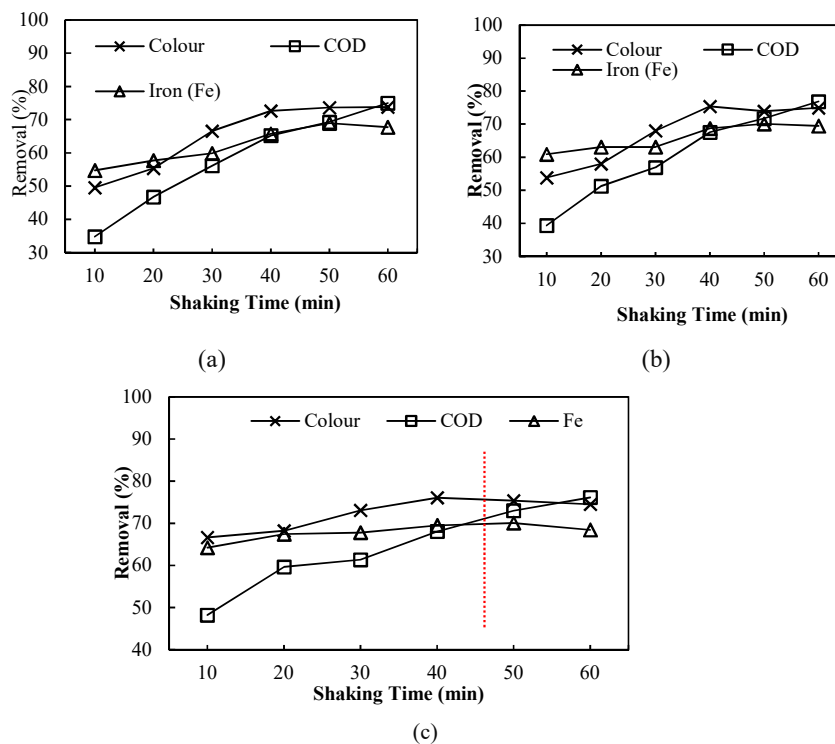
Figure 5 Removal efficiency of COD, color, and Fe (%) versus shaking time (min) using OPF-AC dosages of (a) 1.0 g, (b) 1.2 g, and (c) 1.4 g.

However, after 40 minutes the trend of removal of color, and Fe was slightly constant owing to the attachment of pollutants to active sites on the OPF-AC [10,26]. Meanwhile, the COD was removed continuously until 60 minutes of shaking time due to the availability of interaction between pollutants (COD) and the OPF-AC surface was persistent [9,27]. According to Jain, *et al.* [25], the removal efficiencies and adsorption capacity increase with the increase of shaking time. Thus, the maximum removal indicates the maximum adsorption capacity of the adsorbent [23].

In adsorption treatment, the adsorbent dosage plays a vital role because it affects the cost of the pollutants to be treated [12]. If less AC can achieve a higher removal percentage of pollutants, then it is considered an effective adsorbent. This study found that the optimum condition for all parameters was inconsistent. The maximum color removal of 76% was obtained at 1.4 g of OPF-AC and 40 minutes of shaking time. Meanwhile, the highest removal of COD (77%) and Fe

(70%) was achieved at 1.2 g and 60 minutes, and at 1.2 g and 50 minutes of OPF-AC dosage and shaking time, respectively.

Among all parameters, COD is seen as the main indicator for leachate quality since it provides biodegradability of the leachate [5,20,28]. Therefore, the highest removal of COD (77%) was assigned as the optimum condition, i.e. 1.2 g and 60 minutes of OPF-AC dosage and shaking time. At optimum condition, the maximum removals of COD, color and Fe were 7%, 75% and 69%, respectively. Meanwhile, the maximum adsorption capacity was 146.93 mg/g, 86.43 mg/g and 0.11 mg/g for COD, color and Fe, respectively. This study found that the result obtained is consistent with the findings in previous works that used a similar chemical agent for AC production in leachate treatment. According to Azmi *et al.* [28], COD and color could be removed up to 78% and 87% by using sugarcane bagasse AC. Ghani, *et al.* [20] found that banana pseudo stem AC could remove 83% and 92% of COD and color from leachate. Moreover, the removal of Fe has been carried out by Rajabzahed, *et al.* [29] and Md. Isa *et al.* [30]. They found that the removal of Fe was 68% via employing an alumina and composite adsorbent.

This result means that a low dosage of OPF-AC is necessary at a longer shaking time for the adsorbent to adsorb the pollutant particles. A lower dosage of adsorbent results in a lower removal rate. Thus, fewer pollutant particles are adsorbed. It can therefore be inferred that if the dosage of activated carbon increases, the percentage of removal of pollutants in the leachate also increases, because a large amount of AC provides a higher surface area for adsorption [21,22]. In addition, increasing the adsorbent dosage offers a higher surface area and exchangeable binding sites [31].

However, a further increment of the OPF-AC dosage beyond 1.2 g using a shaking time of 60 minutes (Figure 5(c)) resulted in a slight drop in the removal efficiency of COD, color, and Fe as a consequence of the saturation of adsorption sites during the adsorption process [10,20]. Moreover, the higher OPF-AC dosage may impose interactions between adsorbent particles and lead to the reduction of removal of pollutants (COD, color and Fe) due to the aggregation and overcrowding of OPF-AC particles [5,26].

4   Conclusions

OPF activated with $ZnCl_2$ (OPF-AC) has potential as an adsorbent in treating leachate with a high removal efficiency and adsorption capacity of COD (77%; 146.93 mg/g), color (75%; 86.43 mg/g) and Fe (69%; 0.11 mg/g) at a dosage of 1.2 g and a shaking time of 60 minutes, respectively. Chemical and physical characteristics showed a high surface area, a microporous structure and various

functional groups on the OPF-AC surface. These properties increase the adsorption capacity by surface interaction between the OPF-AC and pollutants via chemical and physical adsorption. However, the performance of AC depends on the properties of the precursor, type of activation and activating agent applied, which control the characteristics of the AC produced. Besides, the shaking time and the OPF-AC dosage also contribute to the removal trend.

A higher shaking time promotes higher interaction between the adsorbate and the OPF-AC surface, while more active sites are present by increasing the dosage. However, the surface of the OPF-AC becomes saturated at sufficient shaking time. Also, agglomeration of OPF-AC particles is observed when an extremely high dosage is provided. Notably, an extended shaking time and a high dosage will lead to a decrease in removal. Overall, it is concluded that OPF-AC can be used as an effective adsorbent for the treatment of landfill leachate.

Acknowledgement

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