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Application of Soot in the Removal of 2, 5-Dichlorophenol in Aqueous Medium

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Abstract: The study aims to use, soot as adsorbent for the removal of 2, 5-dichlorophenol (2, 5-DCP) in aqueous medium. The adsorption kinetic and isotherm were studied using a variety of adsorption models. Soot, obtain from the exhaust pipe of vehicles, was a poor adsorbent with a very limited surface area. As such, the adsorption of 2, 5-DCP had to be conducted for 7 h in order to achieve equilibrium which was between the 240 and 360th min. The highest percentage of removal of 2, 5-DCP achieve was 43.9%. This showed that soot was a weak adsorbent. The experimental data showed that the adsorption kinetics and adsorption isotherm of 2, 5-DCP by soot obeyed the pseudo-second order kinetic model (R^2 = 0.9985) and Freundlich isotherm (R^2 = 0.9825), respectively indicating that both physisorption and chemisorptions were instrumental in the adsorption process. Characterization of the soot recovered from the exhaust pipe showed that the ash content and moisture content in soot were quite high at 4.62 and 2.50%, respectively. The SEM indicated that the soot was nanoparticle in size (>100 nm) with non-visible pores. The soot had spherical shape and tend to cluster together forming aggregates. The FTIR showed that the functional groups such as O-H, C-H and C = O groups, may have contributed to the adsorption process. The BET isotherm indicated that soot obey the type III isotherm without the presence of hysteresis loop. Although this material is not a good adsorbent, it adsorptive ability may be improved by altering its structure chemically providing a waste to wealth opportunity.

Key words: 2, 5-dichlorophenol, soot, adsorption, isotherm, kinetics

INTRODUCTION

Industrial wastewater remediation is currently a major concern for the scientific community as society looks up to them to discover new means to clean and make usable contaminated wastewater. Wastewater which contain contaminate compounds must undergo water treatment as these contaminants can affect consumer health. The water treatment methods are dependent on the contaminants that need to be removed. Commercial wastewater treatment methods consist of chemical, physical and biological treatment methods (Droste, 1997). For chemical treatment, the examples are chemical oxidation, high pressure impulsive discharge and wet oxidation. While for physical treatment methods, adsorption and reverse osmosis are highly favoured. Whereas for biological treatment methods, microbes are commonly utilized. In chemical treatments, the cost of operation is high and the by-product generated from secondary reactions creates other problems which are released into environment at end of the process. Biological treatment has very slow

biology reaction and some contaminants may be too toxic for the biological treatment methods. While in physical treatment, the contaminants in the liquid will be transferred to the solid surface (adsorbent) due to Van der Waals' forces (Wang *et al.*, 2005). Physical treatment methods are usually the fastest and with the least cost but requires further treatment.

The 2, 5-DCP is one of the 18 family members of the chlorophenol group. This compound can form as a result of water and wastewater treatment processes, incineration processes, wood pulp processing and the degradation or metabolism of 1,4-dichlorobenzene and 2,4,5-trichlorophenol based pesticide. The 2, 5-DCP has been detected in groundwater, sewage effluents, surface waters as well in industrial effluents as a pollutant (Krishnaiah *et al.*, 2013; Anisuzzaman *et al.*, 2014). It also has been detected in human urine and amniotic fluid. Therefore, in this study, 2, 5-DCP is chosen as the target pollutant to be removed by using soot.

Soot is form when the fuels (like gasoline, diesel fuel, coal) undergo incomplete combustion when the

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temperature is low or oxygen is limited in supply (Jensen and Malter, 1995). It is also frequently referred as carbon black. Soot is an air-pollutant which is commonly released into the atmosphere from vehicles especially poorly serviced and maintained vehicles. This particles is commonly collected at the exhaust pipes of vehicles forming black plumes when the accelerator is depressed during driving. It is one of the most stable anthropogenic pollutant particles in the environment. The soot migrate into the lungs, resulting in lung cancer due to the Polycyclic Aromatic Hydrocarbon (PAH) (Kaden *et al.*, 1979). Soot has an almost similar structure and component content with carbon black but the method of producing soot and carbon black is quite different. Carbon black is widely used in industrial especially in production of tires in rubber industry (Donnet *et al.*, 1993). The tires tensile strength and wear resistance will be improved with addition carbon black. Carbon black is also used as black pigment in printing ink and in the plastic industry (Donnet *et al.*, 1993). Generally, soot is classified as a nanoparticle material which varies in sizes within the nanometre (nm) range. Thus, it will have a large surface area per unit mass from 1-500 $m^2 g^{-1}$ (Prokhorov, 1979). Due to the expected high surface area, it can be a good nonporous adsorbent, similar to a low price adsorbent like carbon black. However, the possibilities of using soot in these industries have not been explored to date.

In this study, soot was selected as an adsorbent to remove 2, 5-DCP from wastewater. The characterization of soot which was collected from the exhaust pipe of vehicles was conducted by using several instruments including Fourier Transform Infrared (FTIR) spectroscopy and Scanning Electron Microscope (SEM) while the reaction kinetic and isotherm of adsorption of soot in the removal of 2, 5-DCP in aqueous medium will be studied using a few conventional theories like the pseudo-first-order, pseudo-second-order and intra-particle diffusion. UV-Vis Spectrophotometer was used to detect the residual concentration of 2, 5-DCP from the extracted samples which were carried out in triplicates to ensure reproducibility of the experiments.

MATERIALS AND METHODS

Preparation of soot: Soot was collected from the vehicle exhaust pipe. Approximate 100 g of soot was obtained. The sample was placed in a 1000 mL beaker and filled with distilled water until the distilled water level covered the soot. The compound was stirred and heated until a gentle boil was established, creating a homogeneous mixture.

The mixture was continuously stirred for 24 h. After 24 h, the mixture was left idle to enable the soot to form a sediment. Most of the contaminated compounds which are heavier, formed a sediment layer on the bottom of the beaker. The soot which is light, was remained on top part of the distilled water and was filtered out. The sample was then dried in an oven at 110°C for 24 h. This procedure was followed to ensure that all the water molecules were removed from the soot particle prior to the start of any experiment.

Preparation stock solutions of 1000 ppm of 2, 5-DCP and standard solutions: About 1000 ppm of 2, 5-DCP stock solution were prepared by dissolving 1.00 g of 2, 5-DCP in 1000 mL of distilled water. The appropriate concentration was prepared and kept in a dark environment to avoid exposure to ambient light which will influence the stock solution concentration of 2, 5-DCP. 10, 20, 30, 40 and 50 ppm of 2, 5-DCP standard solutions were prepared from the stock solution and used directly in the following adsorption experiments.

Effect of initial concentration: 2, 5-DCP solution with the initial concentrations of 10, 20, 30, 40 and 50 ppm was agitated with 500 mg of soot to study the effect of initial concentration and contact time on adsorption uptake. During the experiment, the solution pH was kept natural without adjustment. The reactions were run for 45 min at room temperature. For every 5 min, a small amount of solution was extracted and analysed.

Effect of dose: Effect of adsorbent dose on adsorption was investigated by agitating 50 ppm of solution with different adsorbent dosage (0.5, 1.0, 1.5, 2.0 and 2.5 g). The reactions took place at room temperature and the pH of the solution was kept natural. The solution was removed after 45 min to test the amount of 2, 5-DCP was removed.

Calculation of the percentage of 2, 5-DCP: The percentage of 2, 5-DCP that had been adsorbed by soot can be calculated by using Eq. 1:

$$\text{Percentage of adsorption} = \frac{C_i - C_t}{C_i} \times 100 \qquad (1)$$

where, C_i is the initial and C_t is the final concentration of the 2, 5-DCP. The removed amount was calculated by using Eq. 2:

$$\text{Removal amount} = \frac{C_i - C_t}{x}(y) \qquad (2)$$

where, x is the amount of soot used, y is the volume of 50 ppm of 2, 5-DCP.

Determination of moisture content: About 1 g of soot was weighted in a 50 mL Erlenmeyer flask. Then, the flask was placed in a drying oven for 3 h at 110°C. After that, the sample was cooled in desiccators and was weight repeatedly until the weight was constant. Equation 3 was used to carry out the calculation:

$$\text{Moisture content (\%)} = \frac{\text{Loss of weight (g)}}{\text{Weight of sample (g)}} \times 100 \qquad (3)$$

Determination of ash content: After the moisture content was determined, the dried sample was transferred to a crucible and placed in a muffle furnace to completely burn off the carbon material in the sample at 500°C for 4 h. The material was cooled in a desiccator and weighted. The ash content was calculated according to Eq. 4:

$$\text{Ash content (\%)} = \frac{\text{Loss of weight (g)}}{\text{weight of dried sample (g)}} \times 100 \qquad (4)$$

Determination of pH: About 1.00 g of soot was weighted and placed into a 200 mL Erlenmeyer flask. Hundred milliliter of distilled water was added and heated for 5 min under gentle boiling. The sample was then allowed cool to room temperature before the pH was a nalysed and recorded.

Morphological structure of soot: A small amount of soot was put on top of a stub and coated with platinum by using JFC-1600 Auto Fine Coater. Then, the morphology and the pore size of the surface of the sample were determined by using Scanning Electron Microscopy (SEM).

Functional group of soot: The surface functional group of soot was determined by using Fourier Transform Infrared Spectroscopy. By using this technique, the surface chemistry of the soot can be explained.

Brunauer, Emmett and Teller (BET) surface area: The BET surface area analysis was used to estimate the surface area and pore size, volume and distribution of the selected soot sample. The BET surface area was calculated from the isotherms using the Brunauer-Emmett-Teller (BET) equation.

RESULTS AND DISCUSSION

Effect of concentration of solution: Figure 1 shows the result of adsorption by soot at different concentrations. Generally, the adsorptions proceeded at a fast pace in the beginning for all the various concentrations due to the availability of large surface area on the soot particles at the beginning and plateau between the 240th to 360th min when equilibrium was achieved. However, the best removal of 2, 5-DCP is at 20 ppm as shown in Fig. 2. As the initial concentration increased after 20 ppm, the percentage of removal for 2, 5-DCP decreased. As the amount of adsorbent was fixed, the availability of active sites are also fixed. Therefore increasing the initial concentration will result in a reduced adsorption rate.
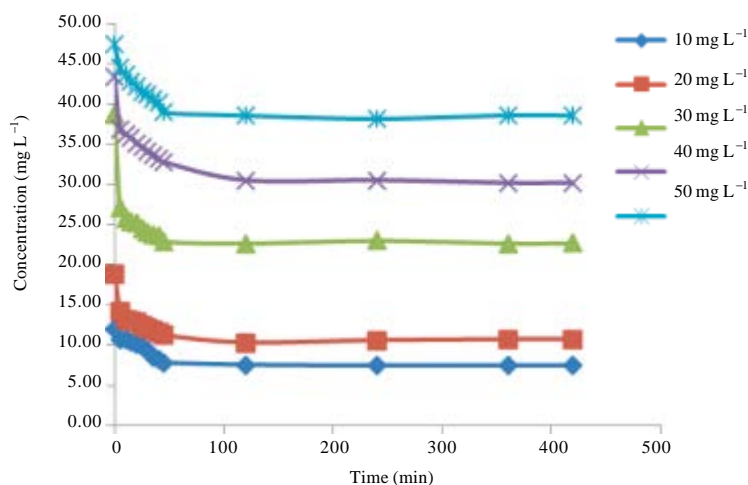

Fig. 1: Different initial concentration 2, 5-DCP that was adsorbed against time
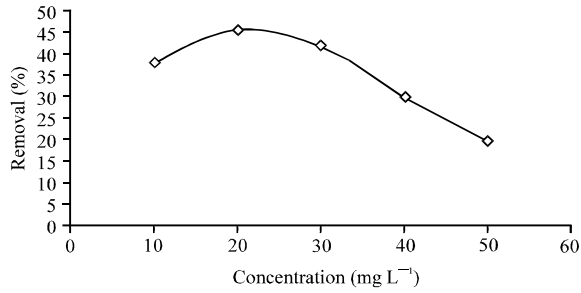
Fig. 2: Percentage removal of 2, 5-DCP against different initial concentrations
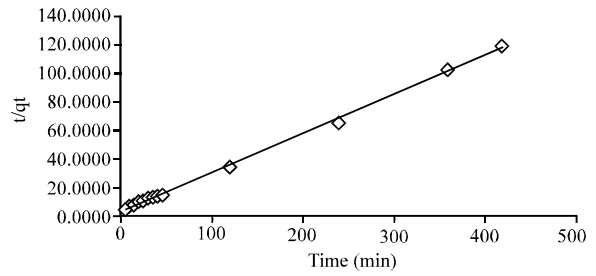

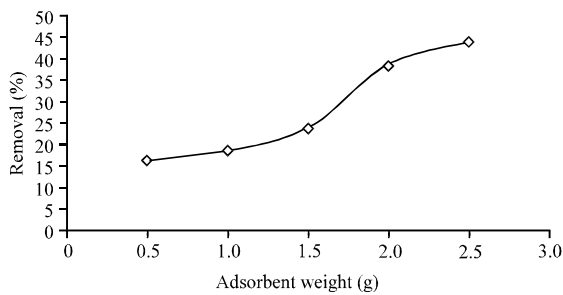Fig. 3: Percentage removal of 50 ppm of 2, 5-DCP against different amount of dose

Fig. 4: Pseudo-first order kinetics
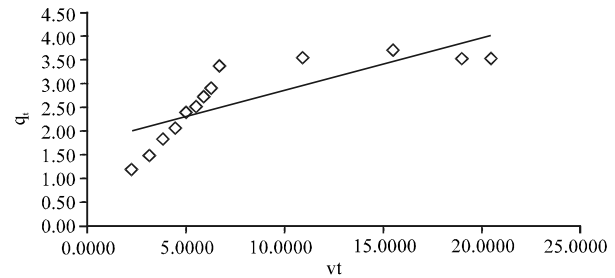
Effect of adsorbent dosage: From Fig. 3, the percentage of removal for 2, 5-DCP is directly proportional to the weight of the adsorbent. According to Gehlot *et al.* (2011), the increase of the removal percentage in tandem with the adsorbent dosage is due to the enhanced surface area of the adsorbent. Normally, after the optimum adsorbent dosage, the adsorption will achieve a maximum adsorption capacity due to the saturation capacity of the adsorbent. However, in this study, the saturation capacity of the adsorbent was not observed due to the low adsorption ability of the adsorbent.

Adsorption kinetics: Several kinetic models were used to describe 2, 5-DCP adsorption by soot which included the pseudo-first order, pseudo-second order and intra-particle diffusion. To determine which model gave the best fit, the

Fig. 5: Pseudo-second order kinetics

Fig. 6: Intra-particle diffusion kinetics
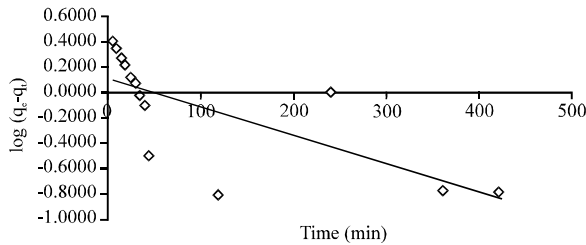
Table 1: Correlation coefficient (R^2) for pseudo-first order, pseudo-second order and intra-particle diffusion for different concentration

Parameters	Concentration (ppm)			
	10	20	30	40
Pseudo-first order kinetics	0.6681	0.7114	0.7710	0.6189
Pseudo-second order kinetics	0.9960	0.9995	0.9999	0.9996
Intra-particle diffusion	0.6430	0.6368	0.5659	0.8285

value, R^2 had to be > 0.95. Figure 4, 5 and 6 presents the graph for pseudo-first order, pseudo-second order and intra-particle diffusion, respectively, tabulated from the experimental data. It can be seen that pseudo-second order was obeyed for all the adsorption experiments indicating that adsorption of 2, 5-DCP by soot was via chemisorption process. Table 1, showed the R^2 value for three kinetic models in different concentrations where pseudo-second order was fitted by all concentration.

Adsorption isotherm: The quantity of adsorbate that can be accumulated by an adsorbent is one of the most important characteristics of the adsorbent. So, adsorption isotherm is use to determine the relationship between the quantity of adsorbate per unit of adsorbent, q_e and it equilibrium solution concentration, C_e at constant temperature. There have several models are available to test for adsorption isotherm but in this study, only two equations were widely which are, Langmuir and Freundlich equation. The summary of the result of Langmuir (Fig. 7) and Freundlich (Fig. 8) can be seen in
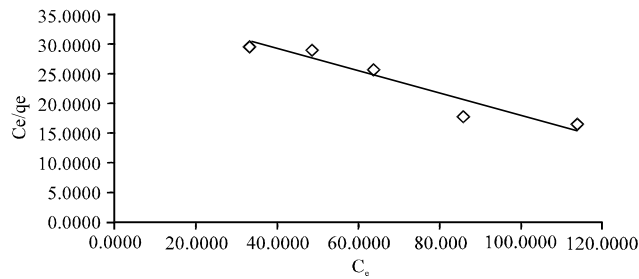
Fig. 7: Langmuir isotherm of the adsorption of 2, 5-DCP by soot
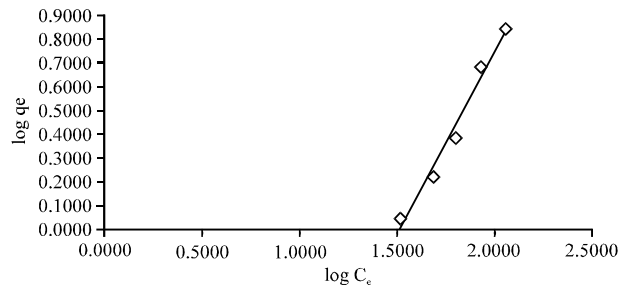
Fig. 8: Freundlich isotherm of the adsorption 2, 5-DCP by soot

Table 2: Correlation coefficient (R^2) for Langmuir and Freundlich isotherm

Isotherm model	Correlation coefficient (R^2)
Langmuir	0.9202
Freundlich	0.9825

Table 2. The data was found to fit the Freundlich isotherm model where R^2 is more than 0.95 indicating multilayer adsorption between the 2, 5-DCP and soot.

Determination of moisture content: The moisture content in the soot was approximately 2.50%. This is relatively low. If the concentration of the moisture is high, this can indicate that the adsorbent have high potential to adsorb chemical compound. But if moisture content was too high, it will have the probability to compete with other adsorbate, thereby reducing the active site of the soot.

Determination of ash content: Ash is a measure of the amount of inorganic materials still contained within the soot. This includes metals such as iron, aluminium, calcium, sodium, potassium, magnesium, etc that does not chemically combined with the carbon surface. Ash contains mainly oxides and small amount of sulphates, carbonates and metals. The percentage of ash that was recorded in the soot was 4.62%. According to Watson and Valberg (2001), actually soot is an unwanted by-product from the combustion of carbon-based materials without controlled for generation of energy or heat like production from diesel engine which is quite different from commercial carbon black which is product

from manufactured under controlled conditions for commercial use as a good adsorbent. The sulfur content in the soot varies with the type of fuel used in the vehicle engine. While the volatile organic compound in the soot was found to vary from a little to more than 20%.

From the experiment, the ash content percentage is considered high and this could possibly be due to contamination from the ceramic filter and its casing (Kandas *et al.*, 2005). The ability to adsorb is inversely proportional to the ash content. So, soot contains relatively high ash content may affect the adsorption potential (Syna and Valix, 2003; Leboda *et al.*, 1997). The ash in soot can be removed by treating it with HCl-HF solution. This was evident with ash content of soot sample that reduced from 22.37-2.62% after washing with HCl-HF solution (Chen and Huang, 2011).

pH value of soot samples: The pH is one of the most important parameter that will influence in adsorption because adsorbent and adsorbate characteristics will be vary with pH. The pH of soot most probably is depend on the extent of oxidant on the surface of the particle especially the present of inorganic compound. Adsorption for neutral compound like phenol and 2, 4-dichlorophenol will be more favourable at pH below 8. When the pH is increase to 10 or above, desorption of molecule will occur. This is because the compound becomes anionic (Fox *et al.*, 1973). At low pH, H^+ may compete with adsorbate to the adsorption sites of
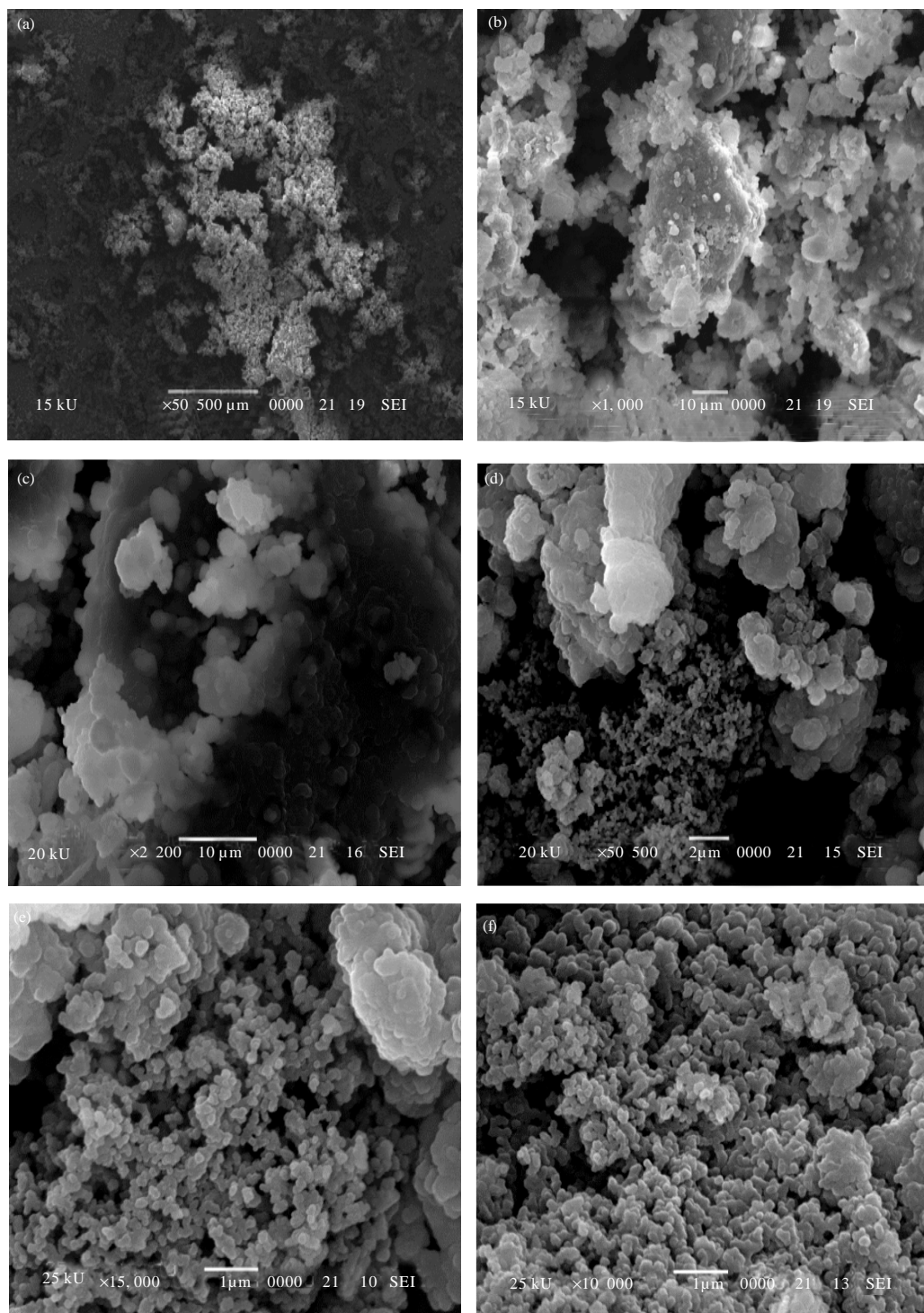
Fig. 9(a-f): SEM image for soot in varieties magnifications. (a) X 50 magnification; (b) X 1,000 magnification; (c) X 2,200 magnification; (d) X 5,500 magnification; (e) and (f) X 15,000 magnification

the adsorbent, so will affect maximum adsorption. Same will be happen to high pH value where OH^- will also compete with adsorbate. So, adsorption process will prefer neutral pH value to achieve maximum adsorption. The 2,5-DCP solution that was prepared had a pH in the range of 5-6. So, soot was washed by distilled water to become pH in the range of 5-6 for optimum adsorption condition. In this study, the pH of soot obtained, was 5.12.

Morphological structure of soot: The morphology and pore size of the surface of the soot was studied and presented. In Fig. 9, the particles of soot can be observed to have spherical size and cluster together to form aggregates (Luo *et al.*, 2009). The average size of the particle is very small, at around about 100 nm. From the SEM images, no pores were visible due to the clustering of the particles and the formation of aggregates. However, even in this condition, 2, 5-DCP can still be adsorbed and trapped by the soot which is evident by the adsorption analysis.

According to Chen and Huang (2011), the soot particles were composed of clustered nanometer sized globules has micropores where the radius is less than 2 nm. It also consist micrometer-sized small boulders with a more solid construction (Endo *et al.*, 2009). According to Chiang *et al.* (1998), micropores is mainly used for

the adsorption in adsorbent like activated carbon. While according to Ghzaoui *et al.* (2004), soot particles are not porous. So, although soot have high surface area and more active sites for adsorption due to its nanoparticle size, the adsorption was low due to the great quantity of organic molecules adsorbed (Okada *et al.*, 1992). However, the lack of pores or pores of suitable sizes could be the reason for the low percentage removal for 2, 5-DCP was which was less than 50%.

Functional group of soot: Figure 10 shows the FTIR spectrum for soot. The FTIR analysis was done to determine the type of functional g roup present on the surface of soot in the spectral region from 4000-600 cm^{-1}. The FTIR was mainly used as qualitative technique for the determination of the chemical structure of carbon material but it is not easy to get good spectra. According to Zawadzki (1988), this is because carbons are black material and almost all of the radiation in the visible spectrum will be adsorbed and the peaks obtained normally were combination of different types of groups. In Table 3, it showed some chemical functional groups that represent the IR frequency (Lopez *et al.*, 2000; El-Hendawy, 2003; Fanning and Vannice, 1993; Przepiorski *et al.*, 2004; Quan *et al.*, 2004; Tomaszewski *et al.*, 2003).
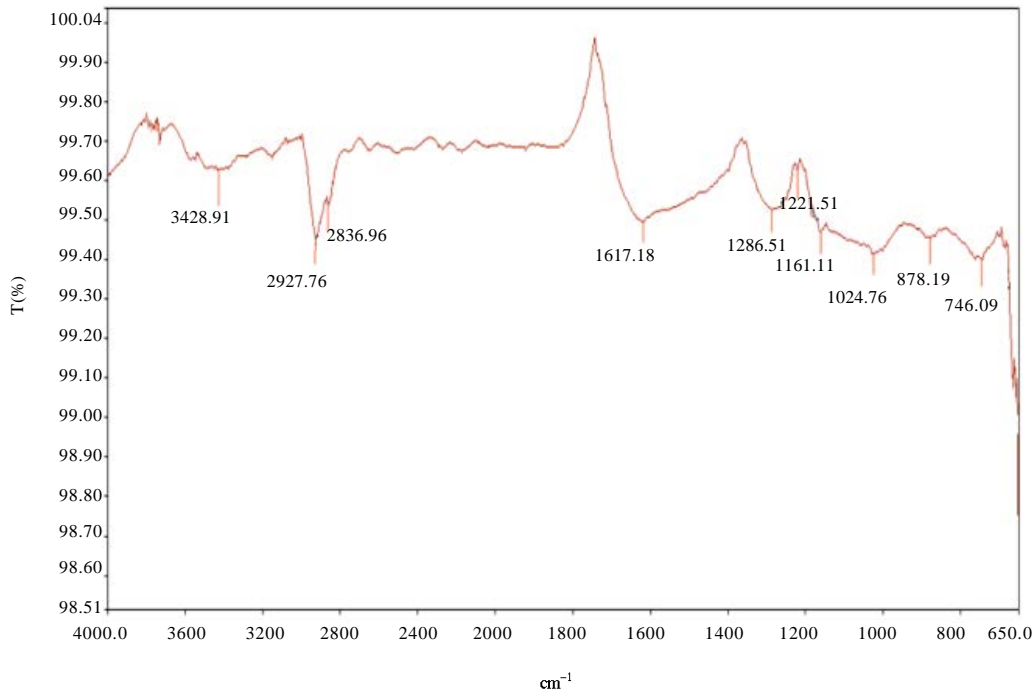

Fig. 10: FTIR spectrum for soot

The absorbance bands from the FTIR spectrum of soot have been found to have peaks at 3428.91, 2927.76, 2863.96, 1617.18, 1286.51, 1221.51, 1161.11, 1024.76, 878.19 and 746.08 cm^{-1}. From the Fig. 10, it can be seen that existence of OH vibration at 3428.91 cm^{-1} which is reflected from water (Meldrum and Rochester, 1990). Band characteristic on adsorbent result from soot can be seen at around 2927.76 and 2863.96 cm^{-1} showing vibration of C-H group. The higher intensity at 2927.76 cm^{-1} peak is due to the simultaneously presence of CH_2 and CH_3. The band at 1617.18 cm^{-1} peak was assigned to be aromatic $C = O$ stretching that may be either quinines or ketone conjugated. The peak in region 1300-1000 cm^{-1} are generally represented phenolic structures from different structural environments (Meldrum and Rochester, 1990). While for the band at 1024.76 and 746.08 cm^{-1} represented contribution from aromatic compound substituted with large alkyl groups and aliphatic deforming, respectively. Important region appeared between 950 and 650 cm^{-1} which is due to C-H out of plane aromatic deformations.

Theoretically, all the soot supposedly submerged into the water due to density where diesel soot have density between 1.17 to 1.34 g cm^{-3} which is denser than water, 1.00 g cm^{-3} (Kirkland *et al.*, 1989). But in this experiment, the particle of soot was not submerged in the solution. It was accumulated together on the surface of the solution and only few amount of the soot submerged into the solution although the solution was stirred for 7 h. Thus, the contact between soot and 2, 5-DCP was less and possibly resulting in a weak adsorption. This may due to the surface of soot that still consist of fuel and separated the soot and solution to contact (Esangbedo *et al.*, 2012). So, although the amount of the soot increase, the percentage does not increase much due to most soot does not contact with the solution resulting in poor adsorption of 2, 5-DCP. So, fuel need to be removed from soot by using acetone or washing with HCl-HF for several times before commencement of adsorption experiments (Chen and Huang, 2011).

Brunauer, Emmett and Teller (B.E.T) surface area: Figure 11 shows the isotherms from N_2-sorption measurements of the soot which contains specific information on the porosity of the particles at the temperature of liquid nitrogen (T = 77 K). A type III isotherm without hysteresis loop was observed indicating that the isotherm was achieved at higher relative pressures. This was caused by the weak adsorbate-adsorbent interactions which is collaborated with the adsorption kinetics and liquid adsorption isotherm (Freundlich isotherm). Substances without relative large pores (mesopores) will also show a total reversibility at desorption which confirms the week interaction between the adsorbate-adsorbent. Table 4 confirmed that mesopores were present and very little micropore, if any were on the surface of the soot. However, this was not corroborated by SEM due to the clustering of the soot particles.

Table 3: IR frequency for functional groups on soot surfaces

| Group or functionality	IR frequency (cm^{-1})
C-O in ethers (stretching)	1300-1000
Alcohols	1276-1049, 3640-3200
Phenolic groups	1300-1000
-C-OH (stretching)	1220-1000
O-H	1200-1160, 3620-2500
Carbonates; carboxyl-carbonates	1500-1100, 1600-1590
-C-C aromatic (stretching)	1600-1550
Quinones or ketone conjugated	1680-1550
Lactones	1370-1160, 1790-1675
Carboxylic anhyrides	1300-980, 1880-1740
C-H (stretching)	3000-2600
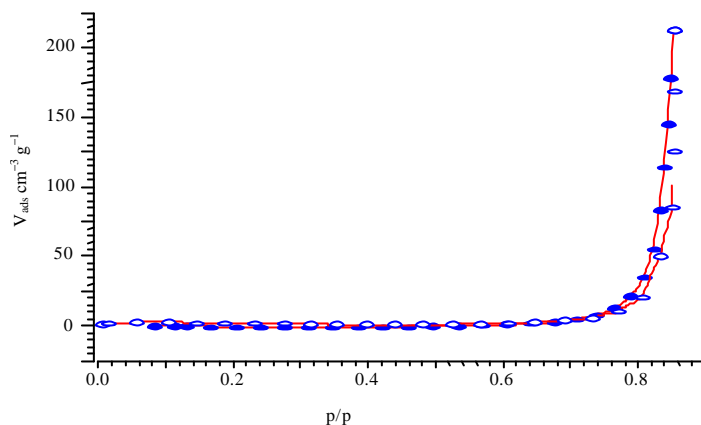
Fig. 11: BET isotherm for soot showing type III isotherm without hysteresis loop

Table 4: Porous and surface properties of AC

Description	Langmuir	Brunauer-Emmett-Teller (BET)	Mesopores Barrett-Joyner-Halenda (BJH)	Mesopores (Cranston and Inkley)	Mesopores (Dollimore and Heal)	Micropores (Horvath and Kawazoe)	Micropores (Saito and Foley)
Surface Area (m^2 g^{-1})	4.1013	5.396	-	-	-	-	-
Cumulative pore area (m^2 g^{-1})	-	-	117.28	117.29	116.98	6.1945	0.002
Pore volume (cm^3 g^{-1})	0.9423	1.2397	-	-	-	-	-
Cumulative pore volume (cm^3 g^{-1})	-	-	0.3635	0.3635	0.3628	0.002	0.002

CONCLUSION

This study shows that the removal of 2,5-DCP was not very good where less than 50% of the target pollutant were remove. Adsorption of 2,5-DCP by using carbon black was conducted for 7 h. However, equilibrium was achieved between the 240th and the 360th min. The experimental data has showed that all the adsorption kinetics of 2,5-DCP by carbon black is the pseudo-second order kinetic model which assumed that the adsorption is via chemisorption process. It was proved that the adsorption of carbon black in removing 2,5-DCP was fitted to Freundlich isotherm. The carbon black that prepared contains of low moisture (2.50%) and high ash (4.62%). The SEM showed that the carbon black particle was spherical shaped and clustered together to form aggregates. The minimum size of the particle that can be measured was roughly 100 nm. By using FTIR spectrometer, proved that few functional groups were present for adsorption which include O-H group, C-H group and C=O groups. BET isotherm indicated that soot was a type III isotherm without the presence of hysteresis loop. In conclusion, soot which washed by distilled water was not a good adsorbent in removing 2,5-DCP but this study serves as a document for future adsorption investigators employing soot from vehicle engine as an adsorbent.

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