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# Adsorption Efficiency of the Activated Charcoal Produced from Spent Coffee Ground for Removal of the BTEX Released from Indoor Paint

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**Abstract**

This study aimed to find the optimum condition to prepare activated charcoal from spent coffee ground and to examine the adsorption efficiency of the BTEX released from water and oil base paints. The optimum condition was found to be carbonization at 400°C for 1 h, followed by activation with $H_3PO_4$ at a 1:1 (w/w) impregnation ratio and then carbonized at 700°C for 1 h. This condition gave the highest iodine number (300.6 ± 5.1 mg/g), Brunaeur-Emmett-Teller (BET) surface area (1,769 $m^2$/g), pore width (0.7947 nm) and pore volume (0.7517 $cm^3$/g). The efficiency of the activated charcoal to adsorb the benzene and toluene emitted from normal and low volatile organic compounds water base paints was 99.8% and 99.7%, respectively. Furthermore, the adsorption efficiency of the activated charcoal from spent coffee ground (96.99 ± 0.54%) to remove the total BTEX released from oil base paints was higher than that achieved with the charcoal without chemical activation (66.97 ± 2.11%) or without carbonization (51.83 ± 10.32%). The produced activated charcoal from spent coffee ground by chemical activation was suitable for use as an adsorbent of the BTEX released from water and oil base paints.

***Keywords:*** activated charcoal; adsorption efficiency; BTEX; chemical activation; spent coffee ground

## 1. Introduction

Activated carbon or activated charcoal is a high porosity material that is useful as an adsorbent and has been widely used in a large range of applications, such as recovery of solvents, separation and purification media and as a catalyst, in addition to the removal of pollutants. Currently, various lignocellulosic materials are commonly used as the starting materials to produce activated carbon, including the shells of coconut, palm, durian and almond (Daud and Ali, 2004; Martínez de Yuso *et al*., 2013; Nabais *et al*., 2011; Tham *et al*., 2011). The textural and chemical characteristics of the adsorbent depend on the nature of the precursor used for its production as well as the methods and condition of its preparation.

One of the most beverages in the world is coffee and it is also ranked as the second largest traded product after petroleum, and produces a large amount of residues (Mussatto *et al*., 2011; Nabais *et al*., 2011). In Thailand, coffee has become very popular and coffee consumption is increasing every year, which creates the problem of an increasing level of waste (spent coffee ground) for disposal. Normally, the spent coffee ground has been sent to landfill or burned for fuel, resulting in air pollution, or it occasionally has been used as a garden fertilizer. However, spent coffee ground has some interesting features, such as a high carbon and low ash content, such that when it is used to produce activated carbon the resultant charcoal has a high surface area and pore volume (Boudrahem *et al*., 2009; Nabais *et al*., 2008). Therefore, to reduce the amount of spent coffee ground for waste disposal and to increase its value, it is of interest to evaluate the potential of spent coffee ground to produce an effective activated carbon.

Basically, there are two processes for preparing activated carbon: carbonization following the activation process, or to perform the activation stage in conjunction with the carbonization. The activation process can be divided into physical and chemical activation. Steam activation is the most common method of producing activated carbons, but acid and carbon dioxide activation has also been used (Kalsson *et al*., 2009).

Currently, volatile organic compounds (VOCs) have received the most attention in the assessment of the indoor air quality and its effects on living

organisms, including human beings (Zabiegała, 2006). The most common types of VOCs that are important pollutants in indoor air are benzene, toluene, ethylbenzene and xylene (or BTEX), which are principally derived from some decoration materials, clothing, cleaners, detergents and paint. Relatively high or short term exposure concentrations of these substances can cause adverse symptoms, such as skin and sensory irritation and central nervous system depression (headache, eye, nose or throat irritation, dizziness, nausea, dry skin and itching, and dry cough), whilst higher and long term exposure levels still can cause serious long term health risks such as kidney, liver and blood disorders and cancer. Owing to the fact that indoor air pollution is becoming a more serious problem, the removal of VOCs from the air using activated carbon is one alternative method to achieve a reduction in the exposure level to people from indoor air.

This work aimed to find the optimum condition for producing activated charcoal from spent coffee ground by chemical activation that was suitable for use as an adsorbent of the BTEX released from water and oil base paints.

## 2. Materials and Methods

### *2.1 Starting material used and its characterization*

Spent Arabica coffee ground was used as the starting material for producing activated charcoal. The elemental analysis of the residue was performed using an Elemental Analyzer Model FlashEA 1112 to determine the carbon, hydrogen and nitrogen content, whilst the cellulose, hemicellulose and lignin content were analyzed by hydrolysis with $H_2SO_4$ and drying technique.

### *2.2 Preparation of activated charcoal*

Activated charcoal was prepared from the spent coffee ground in a two-stage process. The first stage was carbonization and the second stage was chemical activation and combustion. To optimize the first stage carbonization, the spent coffee ground was first dried at 80°C to remove residual moisture and then carbonized at one of three different temperatures (300, 400 and 500°C) in a Nabertherm model Controller P 320 laboratory furnace for 1 h to remove the non-carbon content and other volatile compounds. After carbonization, the charcoal was allowed to cool down to < 130°C before removing it from the furnace and storing in a desiccator. The carbonization temperature that resulted in the charcoal with the highest iodine number was then selected as the optimal first stage carbonization temperature for all subsequent work. For the second stage chemical activation, three chemicals ($ZnCl_2$, NaOH and $H_3PO_4$; all analytical grade) were evaluated as the activating reagent, each as a 1:1 activant: charcoal ratio by weight. Impregnation was performed at room temperature for 24 h to ensure access of the respective activating reagent into the interior of the char. Then, the impregnated sample was dried before combustion in the furnace at the desired temperature (500, 600, 700 and 800°C) for 1 h. The samples were washed with hot distilled water until the filtrate reached pH 6-7 in order to eliminate the residual activating reagent, and the activated charcoal then dried at 120°C.

### *2.3 Physical and chemical analysis of the charcoal and activated charcoal*

The surface structure and porosity of the charcoal prepared at different temperatures (first stage carbonization) were studied by scanning electron microscopy (SEM) using a JEOL model JSM-5410 LV microscope, whilst the adsorption property of the charcoal was evaluated in terms of the iodine adsorption capacity (Iodine number) to reflect the surface area using the ASTM D 4607-94 standard method. The iodine number refers to the amount of iodine absorbed (mg $I_2$/g carbon) and indicates the porosity of the activated charcoal. The higher the iodine number the better the ability of the activated charcoal as an absorbent.

For the activated charcoals produced from the spent coffee ground at various conditions, the surface area was determined by nitrogen ($N_2$) adsorption-desorption isotherm at 77 K by use of a constant volume adsorption apparatus (Surface Area Analyzer). The Brunaeur-Emmett-Teller surface area ($BET_{SA}$) of the activated charcoal was calculated by the BET method from the $N_2$ adsorption isotherm. In addition, the t-plot method was applied to calculate the micropore width and the pore volume. Fourier transform infrared spectrometry (FTIR) was used to qualitatively identify the chemical functionality of the obtained charcoals to select the more optimal condition of activated charcoal preparation. The spectra were recorded from 4000 to 40 $cm^{-1}$.

### *2.4 BTEX adsorption*

The activated charcoal prepared from the spent coffee ground was used to adsorb the BTEX released from paints, and the efficiency of adsorption was compared with that obtained from a commercial

activated charcoal (Anasorb CSC Model 226-01). The apparatus used to study the BTEX adsorption, illustrated schematically in Fig. 1, was comprised of (1) glass bubbler containing 1 ml of indoor paint (the BTEX emission source), (2) charcoal gas tube for determination of the BTEX amount from the source before passing through the adsorbent, (3) glass bubbler containing 5 g of the test activated charcoal used for the adsorption testing and (4) charcoal gas tube for determination of the residual BTEX level after adsorption by the activated charcoal in part (3). Two types of indoor paint, normal and low VOCs water base, were used and each type consisted of three different products. The removal efficiency of the BTEX that was released from the respective paints was then investigated. Moreover, comparison of the adsorption efficiency of the spent coffee ground, the charcoal and the activated charcoal produced from the spent coffee ground was performed using an oil base paint as the BTEX source since it releases a higher amount of all BTEX components than the water base paints.

Before collecting the BTEX sample in each batch, the paint inside the glass bubbler was left for 24 h at constant temperature (25 ± 2°C) in order to get complete volatilization. After 24 h, the BTEX sampling at part (2) and (4) was performed by opening a valve and operating a personal air pump simultaneously at a flow rate of 100 ml/min for 30 min. To prevent absorption of (interference from) background levels of BTEX in the ambient air in the test room, a charcoal glass tube containing activated charcoal was attached to the inlet of the glass bubbler (part 1; Fig. 1).

The quantity of BTEX adsorbed by charcoal in a charcoal gas tube either before or after the adsorption was calculated in a unit mass of BTEX per a mass of adsorbent (i.e. spent coffee ground, charcoal and activated charcoal), as $\mu$g/g adsorbent, and the adsorption efficiency of the activated charcoal is expressed in the term of removal percentage (%) which can be calculated by the following equation (1),

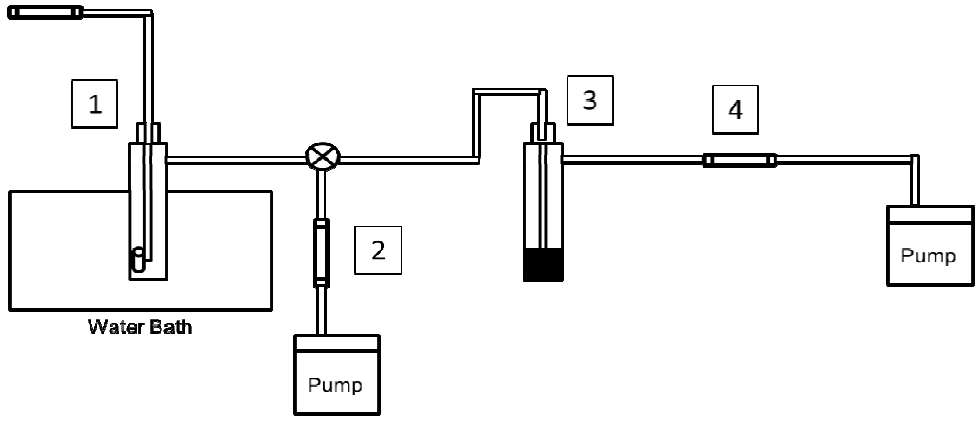
$$\text{Removal efficiency (\%)} = \frac{(M_b - M_a)}{M_b} x\,100 \qquad (1)$$

where $M_b$ = A mass of BTEX adsorbed by adsorbent before the adsorption ($\mu$g/g adsorbent)

$M_a$ = A mass of BTEX adsorbed by adsorbent after the adsorption ($\mu$g/g adsorbent)

### 2.5 BTEX analysis

Since the pollutants in this study, including BTEX, are VOCs, each charcoal glass tube was closed immediately with a cap after sampling was finished. The upper and the lower activated charcoal in each gas tube were analyzed separately. The BTEX analyzed from the upper part represented the actual amount absorbed, while that from the lower part was used to check for breakthrough (saturation of the sorbent). Samples were spiked with 100 $\mu$l of toluened 8 (6,350 ng/ml) as an internal standard and the upper and lower activated charcoal was extracted by 2 ml and 1 ml of carbon disulfide for 1 h, respectively.




Part 1 is a glass bubbler containing indoor paint.
Part 2 is a charcoal gas tube before the absorption.
Part 3 is a glass bubbler containing the test activated charcoal.
Part 4 is a charcoal gas tube for determination of the residual BTEX after absorption.

Figure 1. Schematic diagram of the experimental apparatus for the BTEX adsorption

Gas chromatography, using a model HP 6890N instrument and Agilent HP5 capillary column (30 m × 0.32 mm × 0.25 $\mu$m; 19091J-413) connected with a flame ionization detector, was used to analysis the extracted samples. Carrier gases were $N_2$, helium, hydrogen and air zero. The initial oven temperature was set at 35°C and then increased to 120°C at 5°C/min and then to 230°C at 20°C/min and held for 5 min at 230°C.

### *2.6 Statistical analysis*

The significance of difference between average concentrations of BTEX evaporated from water base paints as well as average adsorption capacity of the activated charcoal for BTEX emitted from water base paints were analyzed by *T*-Test. The difference of efficiency of the different spent coffee ground adsorbents for the removal of the BTEX emitted from oil base paints were tested by one-way ANOVA.

## 3. Results and Discussion

### *3.1 Characterization of spent coffee ground*

The elemental analysis (*n=3*) of the Arabica spent coffee ground revealed that the major element by weight was carbon (65.5 ± 3.02%) followed by hydrogen (7.42 ± 0.14%) and nitrogen (3.85 ± 0.05%). The results are consistent with the previously reported elemental composition of coffee endocarp, where the carbon was the major element (Nabais *et al*., 2008), as well as in almond, walnut and hazelnut shells, apricot stones (Aygün *et al*., 2003), cassava stem biochar (Prapagdee *et al*., 2014), coconut shell powder (Seoka *et al*., 2016) and bamboo biochar (Hassan and Kaewsichan, 2016).

The composition by weight (*n=3*) of cellulose, hemicelluloses and lignin in the Arabica coffee residue was 8.68 ± 1.23%, 90.8 ± 1.29% and 0.28 ± 0.25%, respectively, which concurs with the report that hemicellulose was the main component of coconut shells (Daud and Ali, 2004). However, the main component in almond and palm shells was cellulose and lignin, respectively (Daud and Ali, 2004; Nabais *et al*., 2011).

### *3.2 Effect of the first stage carbonization temperature on the charcoal*

The surface structure and porosity of the charcoal prepared from the spent coffee ground at different carbonization temperatures (300, 400 and 500°C for 1 h) was examined by SEM (Fig. 2). The charcoals obtained from carbonization at 500°C had explicit and homogeneous pores rather than those carbonized at 300 and 400°C. However, only the external surface structure of charcoal could be derived from the SEM analysis and so the iodine adsorption capacity (Iodine number) of each charcoal was also evaluated.

Increasing the carbonization temperature from 300°C to 400°C increased the average iodine number from 228.1 ± 2.95 to 300.6 ± 5.11 mg/g (*n=3*). However, increasing the carbonization temperature further to 500°C decreased the iodine number to 252.9 ± 2.66 mg/g. Therefore, a first stage carbonization at 400°C was selected for the preparation of charcoal before chemical activation.

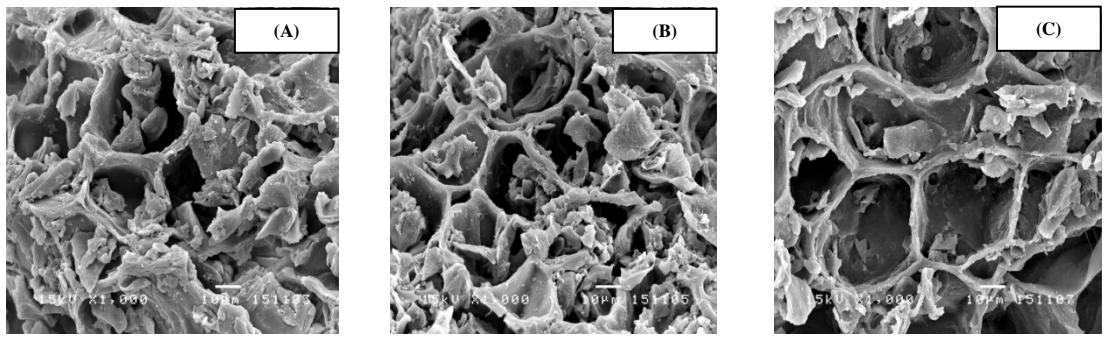



Figure 2. SEM images (1,000 x magnification) images showing the surface structure of the charcoal carbonized at (A) 300, (B) 400 and (C) 500°C for 1 h. Scale bars represent 10 $\mu$m.

*3.3 Optimum conditions for the second stage chemical activation and combustion of the charcoal*

Comparison of the different chemicals ($ZnCl_2$, NaOH and $H_3PO_4$) and combustion temperatures (500, 600, 700 and 800°C for 1 h) on the BETSA and micropore characteristics of the different activated charcoals are shown in Fig. 3. Activation with $H_3PO_4$ at a 1:1 weight impregnation ratio and subsequent carbonization at 700°C for 1 h was the most optimal condition in terms of giving an activated charcoal with the highest average $BET_{SA}$ (1,769 $m^2/g$) pore width (0.7947 nm) and pore volume (0.7517 $cm^3/g$), and these was markedly higher (1.62-, 1.25-and 1.73-fold, respectively) than those for the commercial activated charcoal. Moreover, the $BET_{SA}$ of the activated charcoal from spent coffee ground was higher than those reported for the carbon produced from durian shell and oil-palm stone that have been used to adsorb pollutants in the air, such as toluene, $NH_3$ and $NO_2$ with values of 1,404, 1,563 and 1,408 $m^2/g$, respectively (Guo and Lua, 1999; Tham *et al*., 2011). The pore width of this activated charcoal from spent coffee ground was broadly similar to that previously reported (0.772 nm) for the activated charcoal from coffee residue activated with $ZnCl_2$ (Boudrahem *et al*., 2009). Activated charcoals with a pore size of < 2 nm are classified as micropore materials according to IUPAC (Biniak *et al*., 2001).

The results of this study suggest that $H_3PO_4$ was an appropriate activating reagent for the preparation of the activated charcoal from coffee residue, supporting that $H_3PO_4$ worked as dehydration reagent and played an important role in retarding tar escape during the carbonization process (Boudrahem *et al*., 2009). In addition, the use of a suitable combustion temperature (700°C) could enhance the activated charcoal via generating a higher porosity level in the activation step.

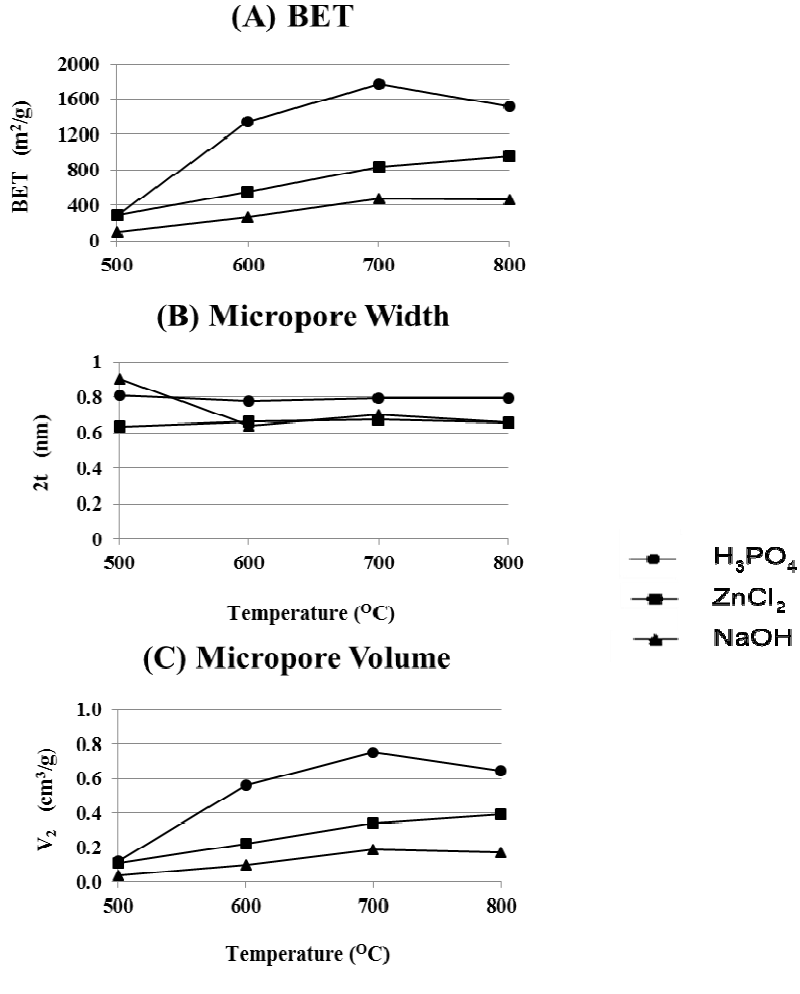



Figure 3. Effect of different chemical activants ($H_3PO_4$, $ZnCl_2$ or NaOH) and combustion temperatures of (500, 600, 700 or 800°C for 1 h) on the (A) $BET_{SA}$, (B) micropore width and (C) micropore volume of the obtained activated charcoal

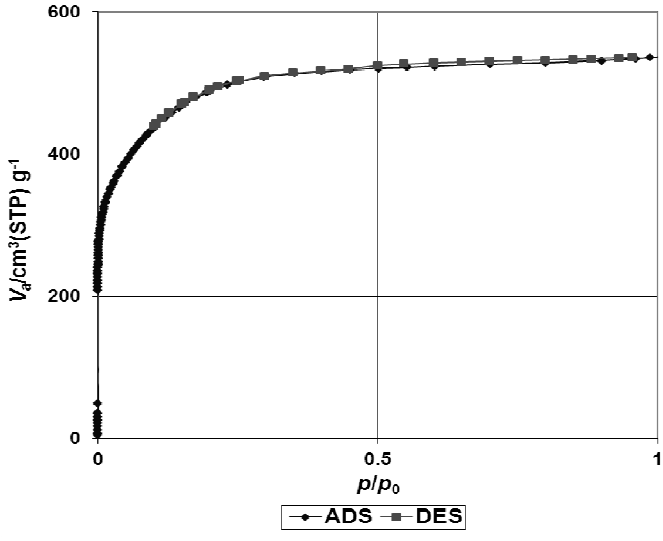



Figure 4. $N_2$ adsorption-desorption isotherms at 77 K of the activated charcoal prepared by activation with $H_3PO_4$ and subsequent heating at 700°C for 1 h. In the legend, ADS = adsorption and DES = desorption.

The $N_2$ adsorption-desorption isotherms at 77 K on the activated charcoal, prepared by an initial 400°C carbonization for 1 h and a second stage chemical activation with $H_3PO_4$ and subsequent heating at 700°C for 1 h, are shown in Fig. 4. The adsorption-desorption isotherms of the activated charcoal prepared from spent coffee ground, according to the Brunauer, Deming, Deming and Teller (BDDT) classification, was classified as Type I indicating that they are microporous in nature.

The FTIR analysis of the functional groups presention the activated charcoal revealed two significant peaks, one at wave number 3422 cm$^{-1}$ that is related to the hydroxyl group (O-H) and the other at 1566 cm$^{-1}$ that represents the carbonyl group (C=O). These spectra agreed with previous results where the bands at 3422 cm$^{-1}$ and 3200-3740 cm$^{-1}$ corresponded to the hydroxyl (O-H) group (Nabais *et al*., 2008). The carbonyl group was visible in all spectra with the presence of ketones (22-2500 cm$^{-1}$), carboxylic acids (1-1790 cm$^{-1}$) and quinones (1550-1680 cm$^{-1}$) (Guo and Lua, 2003). Moreover, these results are consistent with the surface functional groups found on activated charcoal derived from other biomasses, such as coffee endocarp (Nabais *et al*., 2008), almond shell (Nabais *et al*., 2011), durian shell (Tham *et al*., 2011) and bamboo biochar (Hassan and Kaewsichan, 2016).

### *3.4 Absorption of the BTEX released from indoor water base paints*

Benzene and toluene, but neither ethylbenzene nor xylene, were detected in the volatiles from the different water base paints. The level of benzene that evaporated from the normal (1,101.2-1,824.5 $\mu$g/m$^3$) and low VOCs (582.34-1,911.01 $\mu$g/m$^3$) water base paints (Fig. 5(A)) were not significantly different. However, the level of toluene that emitted from the normal (598.0-11,052.9 $\mu$g/m$^3$) and low VOCs (BLQ (below limit of quantification)-248.4 $\mu$g/m$^3$) water base paints were significantly different (Fig. 5(B)). The average concentration of benzene and toluene in the normal and low VOCs water base paints are summarized in Table 1.

The concentrations of BTEX found in this study were in the high range when compared with another study in Mumbai (India) at a range of 48.6-397.7 $\mu$g/m$^3$ for benzene and 136.8-265.7 $\mu$g/m$^3$ for toluene, although that study measured the concentration of VOCs in the indoor environment while painting (Srivastava *et al*., 2000) as opposed to after 24 h in this study.

Table 1. Average concentration of benzene and toluene evaporated from water base paints

| Pollutant | Average concentration ± SD ($\mu$g/m$^3$) | |
|---|---|---|
| | Normal type | Low VOCs type |
| Benzene | 1,567.7 ± 379.7 [a] | 1,300.3 ± 610.0 [a] |
| Toluene | 7,104.3 ± 4,941.2 [a] | 205.2 ± 91.7 [b] |

Data are shown as the mean ± 1 SD, derived from three independent analyses. Means within a row followed by a different lowercase superscript letter are significantly different ($p < 0.05$).

Benzene and toluene from the different water base paints, at the concentrations indicated in Table 1, were passed through the optimal activated charcoal prepared from coffee residue to study its adsorption efficiency. After adsorption the residual benzene and toluene level in the air stream was clearly decreased (Fig. 6). The concentration of benzene was reduced by ~2.0- to ~ 30-fold to 2.66 (BLQ)-924.9 $\mu g/m^3$ for the normal water base paints and ~1.9- to ~15.0-fold to 122.8-269.5 $\mu g/m^3$ for the low VOCs type paints. The level of toluene emitted from the normal water base paints was reduced to 30.2 (BLQ)-8,395.2 $\mu g/m^3$, whereas the toluene level remained below the LOQ (30.2 $\mu g/m^3$) for the low VOCs water base paints.

### *3.5 Efficiency of BTEX removal by the optimal activated charcoal*

The amount of BTEX adsorbed onto 1 g of activated charcoal produced from the spent coffee ground was investigated using the same six indoor water base paints, and the average absorption capacity ($\mu g$/g activated charcoal) is summarized in Table 2. The maximum average benzene and toluene adsorption capacity from the normal (1.10 ± 0.04 and 6.15 ± 0.41 $\mu g$/g, respectively) and low VOCs (1.09 ± 0.15 and 0.14 ± 0.04 $\mu g$/g, respectively) water based paints were not statistically different. Thus, the variation in the amount of benzene and toluene present in the different types

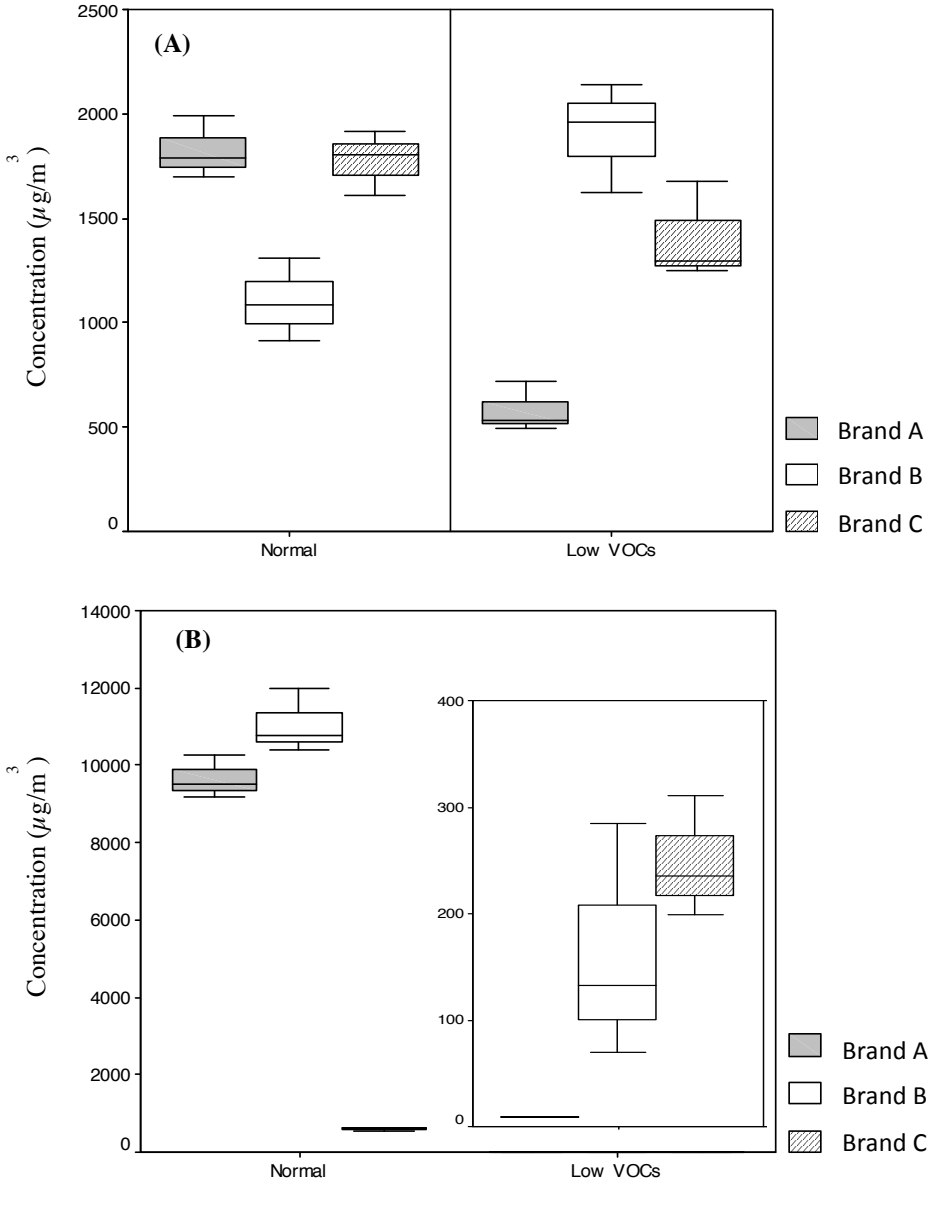
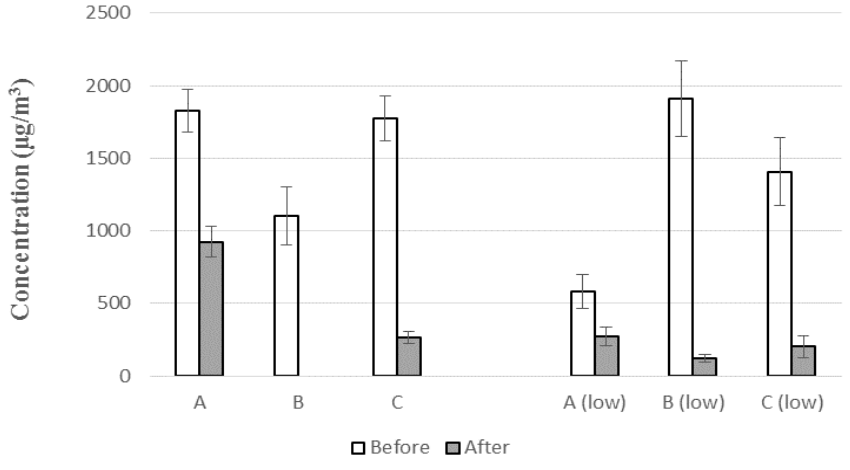
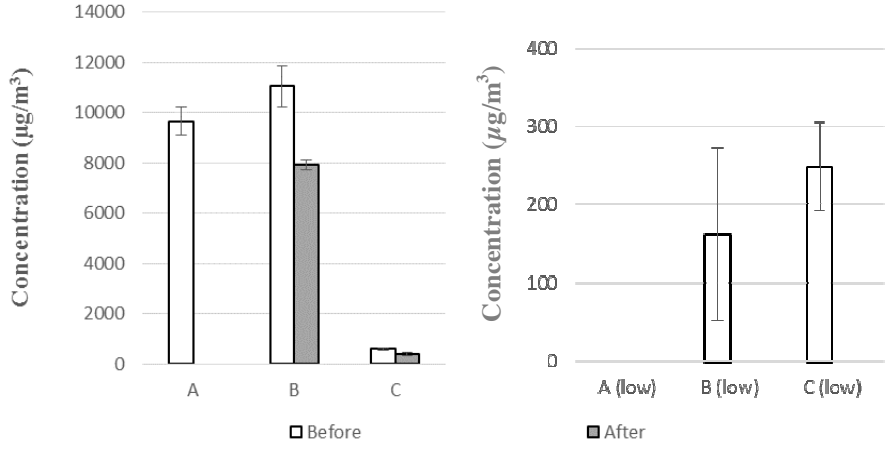



Figure 5. Concentration of (A) benzene and (B) toluene released from the normal and low VOCs water base paints. Data are shown as the mean ± 1SD, derived from three independent analyses.




(A)




(B)

Figure 6. Concentration of (A) benzene and (B) toluene in the air stream emitted from three brands (A to C) of each type (normal and low VOC) of water base paints before and after adsorption by the activated charcoal. Data are shown as the mean ± 1 SD, derived from three independent analyses. Means with a different lowercase letter are significantly different ($p < 0.05$).

of water base paint seemingly did not affect the adsorption capacity of the activated charcoal produced from the spent coffee ground. The %removal of benzene and toluene from the volatiles emitted from the water base paints was 99.8% and 99.7%, respectively.

The adsorption efficiency of the activated charcoal produced from the spent coffee ground was compared to those for the spent coffee ground and first stage charcoal (400°C for 1 h) prior to activation with $H_3PO_4$ and heating at 700°C for 1 h. Oil base paints were used as the BTEX emission source because all of the BTEX components could be detected in the volatiles from this paint type. The adsorption efficiency of the total BTEX released from the oil base paints was significantly different ($p < 0.05$) between three types of materials (Table 3), with the activated charcoal produced from the spent coffee ground giving the highest adsorption efficiency, followed by the coffee residue char carbonized at 400°C and the spent coffee ground before carbonization.

Table 2. Average adsorption capacity of the activated charcoal for benzene and toluene emitted from water base paints of each type

| Pollutant | Average adsorption ± SD (µg/g) | |
|---|---|---|
| | Normal type | Low VOCs type |
| Benzene | 0.76 ± 0.20 [a] | 2.74 ± 2.67 [a] |
| Toluene | 2.75 ± 2.68 [a] | 0.11 ± 0.06 [a] |

Data are shown as the mean ± 1 SD, derived from three independent analyses. Means within a row followed by a different lowercase superscript letter are significantly different ($p < 0.05$).

Table 3. The efficiency of the different spent coffee ground adsorbents for the removal of the BTEX emitted from oil base paints

| Pollutant | Removal efficiency (%) | | |
|---|---|---|---|
| | Spent coffee ground | Charcoal | Activated charcoal |
| Benzene | 12.2 ± 1.04 [a] | 30.2 ± 6.02 [b] | 95.2 ± 0.94 [c] |
| Toluene | 59.2 ± 2.61 [a] | 98.0 ± 0.46 [b] | 98.4 ± 0.33 [b] |
| Ethylbenzene | 74.3 ± 16.53 [a] | 91.7 ± 1.84 [ab] | 99 ± 0.06 [b] |
| m,p-Xylene | 78.2 ± 14.71 [a] | 91.6 ± 1.47 [ab] | 100 ± 0.01 [b] |
| o-Xylene | 82.5 ± 7.77 [a] | 88.8 ± 3.77 [ab] | 95 ± 0.15 [b] |
| Total BTEX | 51.83 ± 10.32 [a] | 66.97 ± 2.11 [b] | 96.99 ± 0.54 [c] |

Data are shown as the mean ± 1SD, derived from three independent analyses.
Means within a row followed by a different lowercase superscript letter are significantly different ($p < 0.05$).

## 4. Conclusions

A microporous activated charcoal could be prepared by carbonization of the spent coffee ground at 400°C followed by chemical activation with $H_3PO_4$ and heating at 700°C for 1 h. This more optimally activated charcoal could adsorb the benzene and toluene emitted from water base paints with an efficiency of 99.8% and 99.7%, respectively, and could remove total BTEX released from oil base paints at approximately 97.0% efficiency. Consequently, spent coffee ground has the potential to produce a low-cost activated charcoal that could be utilized for the removal of BTEX released from water and oil base paints.

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