



Faculty of Resource Science and Technology

**PREPARATION AND CHARACTERIZATION OF ACTIVATED
CARBON FROM USED POLYETHYLENE TEREPHTHALATE
(PET) BOTTLES FOR POTENTIAL WASTEWATER
TREATMENT APPLICATION**

Aileen Sim Kai Fang

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PREPARATION AND CHARACTERIZATION OF ACTIVATED CARBON FROM USED
POLYETHYLENE TEREPHTHALATE (PET) BOTTLES FOR POTENTIAL
WASTEWATER TREATMENT APPLICATION

AILEEN SIM KAI FANG

This project is submitted in partial fulfillment of
the requirements for the Degree of Bachelor of Science with Honours
(Resource Chemistry)

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DECLARATION

No portion of the work referred to in this report has been submitted in support of an application for another degree of qualification of this or any other university or institution of higher learning.



Aileen Sim Kai Fang (9748)

Programme of Resource Chemistry

Faculty of Resource Science and Technology

Universiti Malaysia Sarawak

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Preparation and Characterization of Activated Carbon from Used Polyethylene Terephthalate (PET) Bottles for Potential Wastewater Treatment Application

Aileen Sim Kai Fang

Resource Chemistry
Faculty of Resource Science and Technology
Universiti Malaysia Sarawak

ABSTRACT

Activated carbons were prepared from used PET bottles by physical carbonization and activation in nitrogen gas. The weight percentage of carbon yield was about 18%. The physical and chemical properties of activated carbon prepared from PET were compared with commercial activated carbon samples. The pH of the PET derived activated carbon was above pH 7 with both the acidic and basic functional groups coexisting on the surface of PET derived activated carbon. No distinctive pores were being observed from the SEM micrograph of PET activated carbon. The adsorption of Pb(II) ions by PET derived activated carbon had been studied at different initial pH and contact time. PET derived activated carbon followed the Pseudo-second order kinetic model on the adsorption of Pb(II) ions. The removal efficiency of Pb(II) ions by PET derived activated carbon was observed to be less than 20%.

Keywords: Activated carbon, SEM, Adsorption

ABSTRAK

Karbon teraktif disediakan daripada botol PET terpakai melalui proses pengeluaran karbon dan pengaktifan dengan menggunakan gas nitrogen. Peratusan penghasilan karbon adalah kira-kira 18%. Perbandingan ciri-ciri fizikal dan kimia di antara karbon teraktif PET dan karbon komersial telah dijalankan. pH karbon teraktif PET adalah melebihi pH 7 dan kedua-dua kumpulan berfungsi asid dan alkali wujud serentak pada permukaan karbon teraktif PET. Tidak ada liang ketara pada karbon teraktif PET apabila diperhatikan melalui mikroskop SEM. Penjerapan Pb(II) telah dikaji pada pH permulaan dan masa tindakan yang berlainan. Karbon teraktif PET mematuhi model Pseudo-second order dalam penjerapan Pb(II). Kecekapan penyingkiran Pb(II) oleh karbon teraktif PET adalah kurang daripada 20%.

Kata kunci: Karbon Teraktif, SEM, Penjerapan

CHAPTER ONE

INTRODUCTION

Interests in activated carbon received a big boost during and after the First World War. The pollution of the environment due to rapid industrialization and ever increasing use of the amount and the variety of chemicals in almost every facet of life had initiated the attention to the production of activated carbon. Although activated carbon was the first recognized adsorbent, it is still widely being used in industries. The developments of appropriate method of preparation and in-depth understanding of their porous structure of activated carbon are still continuing.

Activated carbon is a crude form of graphite. It is not truly an amorphous material but has a microcrystalline structure which differs from that of graphite with respect to interlayer spacing which is 0.34-0.35 nm in case of activated carbon. Generally, the starting materials for the production of activated carbons are those with high carbon but low inorganic contents such as wood, fruit shells, brown coals, lignites and highly volatile bituminous (Tansel and Nagarajan, 2004; Rodriguez-Reinoso and Molina-Sabio, 1998). Granular activated carbon is most commonly produced by grinding the raw material, adding a suitable binder to give it hardness, recompact and crushing it to desire size. It is then undergone thermal decomposition under anaerobic condition and controlled heat. As reported in the literature, pore development by activation reactions is governed by the diffusivity and reactions of the activation agents. Mesopores develop under the conditions where micropores development is restricted and vice versa (Nakagawa *et al.*, 2003).

Generally, adsorption is the process where molecules are concentrated on the surface of the activated carbon. This adsorption arises as a result of the unsaturated and unbalanced

molecular forces which are present on every solid surface. The adsorption can be divided into two types: physical adsorption and chemisorption. Physical adsorption refers to relatively weak Van der Waals forces exist between the adsorbate and the carbon surface. Physical adsorption occurs because all the molecules exert attractive forces, especially molecules at the surface of activated carbon (pore walls of carbon), and these surface molecules seek other molecules to adhere to. Adsorption from solution occurs as a result of difference in adsorbate concentration in the solution and in the carbon pores. The molecules will move from high concentration area to low concentration area through the pore channels to reach the area where the strongest attractive forces are. Water contaminants adsorb because the attraction of the carbon surface for them is stronger than the attractive forces that keep them dissolved in solution. Thus, it can be concluded that for the activated carbon to be effective, the adsorbates must have lower solubility, are organic, high molecular weight and a neutral or non-polar chemical nature (Nicholas and Paul, 1993).

On the other hand, chemisorption involves exchange or sharing of electrons between the adsorbate molecules and the surface of the adsorbent resulting in chemical reaction. For example is the formation of disinfection-by-product, DBP's (Clark, 1989). This is the reaction where the chlorine reacts with carbon to form chloride ions. These DBP's are often carcinogenic and therefore highly undesirable. Physical adsorption is non specific while chemisorption only takes place at certain adsorbate-adsorbent system.

Due to its high adsorption capacity, activated carbon is regarded as one of the most widely used adsorbents for water and gaseous emissions purifications. According to Nicholas and Paul (1993), the great advantage that activated carbon has over other recovery system is the outstanding ability to recover organics from low concentrations easily and inexpensively. Gas purifications applications involving the removal of low concentrations of impurities

include the deodorization of air, the removal of traces of organic impurities from gas streams to prevent catalyst poisoning, the removal of traces of oil vapors from compressed gases, and the removal of similar substances from fluid streams.

Activated carbons are useful for the removal of color, odor and taste, organic and inorganic pollutants from drinking water, in the treatment of industrial waste water and in a variety of gas phase applications. The most important application of activated carbon adsorption is the purification of air and water. For example, the catalytic converters are being used in the automobiles to convert volatile organic compound into carbon dioxide.

The effectiveness of activated carbon for the removal of organic compounds from fluids by adsorption is enhanced by its large surface area. The surface area of activated carbon typically can range from 450-1,800 m²/g. Besides surface area, the effectiveness of the activated carbon to remove specific contaminants depends on the presence of the functional groups on the carbon surface, which strongly influence the adsorption (Tansel and Nagarajan, 2004). In other words, the precursor used to prepare activated carbon will affect its adsorption capacity. Hence, in this study the used or post consumer polyethylene terephthalate (PET) bottles have been chosen as the precursor to prepare activated carbon and to determine the adsorption capacity of PET derived activated carbon for heavy metal ions removal. There are only few studies on preparing activated carbon from PET bottles.

Activated carbon can be characterized by physical properties and activity properties. Physical properties such as surface area, density, mesh size, abrasion resistance and ash content are important. In water treatment applications, carbon density is expressed as backwashed and drained (BWD) or bulk density. Activity characterizations are key indicators of a carbon's potential performance for removing contaminants from water. This is attributed to the pore size distribution of the activated carbon. Particle size is an important parameter in

specifying carbon for specific applications, affecting such operating conditions as pressure drop, filtration capabilities, backwash rate requirements and the rate of adsorption of contaminants (Nicholas and Paul, 1993).

Current attention has been focused on the municipal solid wastes which create a lot of pollution problems because most plastic wastes are non-biodegradable. Among the plastic wastes, polyethylene forms the largest fraction, which is followed by polyethylene terephthalate (Subramanian, 2000). According to Zhong and Itoh (2003), the recycle rate for polyethylene terephthalate (PET) in Japan was only 30% compared with 50% for paper in year 2000. The process of recycling is not generally profitable, thus it would be beneficial to convert the used PET bottles into value-added adsorbents in the form of activated carbons. This would not only help to reduce plastic waste pollution and activated carbons derived from these used polyethylene terephthalate bottles can be utilized to remove heavy metals especially in waste water and for potential applications in electrochemical devices.

In carbon manufacturing polymer precursors are being used when carbon of low inorganic impurity content is needed. Polyethylene terephthalate (PET) might therefore be a promising precursor as it contains a high percent of carbon (about 62.5% wt.). Nowadays, it is also being used commercially replacing glass in soft drink bottles and containers. Hence, its conversion to activated carbon offers a way to reduce solid polymer waste as well (Laszlo and Szucs, 2001). Besides, the preparation of activated carbon from used polyethylene terephthalate bottles can be profitable since the raw materials can be obtained free of charge from the recycling and collection centre.

1.1 Objectives of study

The objectives of this study include:

1. To prepare activated carbon from carbonaceous precursor materials derived from post consumer or used polyethylene terephthalate (PET) bottles such as coca-cola and mineral water bottles.
2. To characterize the physical and chemical properties of the activated carbon prepared.
3. To evaluate the potential applications of such activated carbon in wastewater treatment.

CHAPTER TWO

LITERATURE REVIEW

The general process to produce activated carbon is based on carbonizing and activating the carbonaceous material. The carbonization process involves heating carbonaceous material to 600-800 °C in the absence of air to eliminate the volatile substances. The activating process can be carried out in two ways, either physically or chemically. For physical activation, the precursor will be carbonized and followed by gasification of the crude char (Martinez *et al.*, 2005; Ganzalez *et al.*, 1994). Whereby for chemical activation, the starting material is impregnated with a chemical agent either acid or base (usually are zinc chloride and phosphoric acid), and the resulting blend is heated to a temperature of 450-700 °C (Martinez *et al.*, 2005). The fine capillary structure is formed during the activation process. The amorphous carbon and hydrocarbons of raw material become active when the hydrocarbons held in the carbon are removed by oxidation.

Many researchers have reported the production of activated carbon from various types of precursor materials, but there are only few publications related to the preparation of activated carbon from used polyethylene terephthalate bottles. Laszlo and Szucs (2001) had carried out a study on the surface characterization of polyethylene terephthalate (PET) based activated carbon and the effect of pH on its adsorption capacity from aqueous phenol and 2,3,4-trichlorophenol solutions. The activated carbon was prepared from granulated polyethyleneterephthalate (PET) obtained from Mitsubishi (Singapore). The granulated PET was carbonized at 750 °C in nitrogen atmosphere for 30 minutes before they were grounded and sieved. The activation was carried out at 900 °C with burn off 50% in a steam/nitrogen flow (molar ratio 1:1) for 90 minutes. The activated carbon was evaluated for the surface

chemical composition by X-ray photoelectron spectroscopy (XPS). It was found out that the surface of the PET carbon contained 95.7 atomic % of carbon and 4.3 atomic % oxygen, giving an O/C ratio of 0.045. The atomic O/C ratio calculated from the elemental analysis was varied between 0.047 and 0.052. This means, there was no difference between the O/C ratio in the bulk and on the surface. The adsorption for phenol and 2, 3, 4-trichlorophenol on this carbon had been studied from the aqueous solution of different pH (pH=3, 11 and unbuffered). The adsorption mechanism depended both on the pH of the solutions and the pK_a of the phenol. A possible explanation of the elevation of pH following the aqueous extraction could be the retention of phthalic acid monomer units from the degradation of the polymer, even after the activation process. The phenol adsorption isotherms belong to type L according to Giles' Classification (Laszlo and Szucs, 2001; Giles *et al.*, 1960), except for the unbuffered 2,3,4-trichlorophenol curve. The L or Langmuir type means that the aromatic ring absorbs parallel to the graphitic surface, and there is no strong competition between the adsorbent and the solvent to occupy the surface sites.

Nakagawa *et al.* (2003) have tested the gas adsorption on activated carbons from PET mixtures with a metal salt. Metal salt such as $Ca(NO_3)_2 \cdot 4H_2O$, $Ca(OH)_2$, $CaCO_3$, ZnO and $AlNH_4(SO_4)_2 \cdot 12H_2O$ were mixed with PET before carbonization and with acid treatment after carbonization. From this paper, it was concluded that the % of burn off increases with increasing activation. The porous properties of the activated carbon were ranging from 370 to 2200 m^2/g for the BET surface area. It was confirmed that the metal salts used in the pretreatment for activation did affect the microporous structures but did not influence the mesoporous structures.

Another group of scientists, Zhang and Itoh (2003) have reported the production of adsorbents from waste ashes and post consumer PET which were subsequently utilized in

wastewater treatment. According to them, the PET was first melted and blended with ashes and the mixture was being carbonized to form different types of adsorbents. The BET surface area of the adsorbents ranged from 115 to 485 m²/g. It was believed that the adsorbents produced in this manner can be used in wastewater treatment for discoloration and heavy metal removal. Some carbons have been known to have a surface area up to 2,500 m²/g and examples are shown in Table 2.1

Table 2.1 Surface areas of typically available activated carbons

Origin	Surface area (m ² /g)
Bituminous coal	1,200-1,400
Bituminous coal	800-1,000
Coconut shell	1,100-1,150
Pulp mill residue	550-650
Pulp mill residue	1,050-1,100
Wood	700-1,400 ,

Adapted from Nicholas and Paul (1993)

According to Boonamnuayvitaya *et al.* (2005), six types of activated carbons could be prepared from coffee residues by varying activating agents such as zinc chloride, nitrogen, carbon dioxide and steam (Table 2.2). The activated carbons were used in formaldehyde adsorption. The coffee residues in this experiment were provided by Nestle Products (Thailand), Inc. The dried coffee residues were divided into two portions in which powdered ZnCl₂ was added into it one portion and another portion was without ZnCl₂. Both portions of coffee residues were treated with three procedures: (a) pyrolysis in a stainless steel tube held in a horizontal tube furnace under nitrogen flow; (b) pyrolysis under nitrogen flow followed by activation with carbon dioxide and (c) pyrolysis under nitrogen/steam flow. For example, sample prepared with ZnCl₂ under nitrogen flow was named as CZn-N₂. In this analysis, it was found that CZn-N₂-CO₂, CZn-N₂, CN₂-ST, CZn-N₂-ST and CN₂ showed the N₂

adsorption-desorption by mesoporous solids. Meanwhile, the isotherm of $\text{CN}_2\text{-CO}_2$ showed the adsorption of gases by non-porous solid. Lastly, the N_2 isotherms of the commercial activated carbon (CH-I1000) are typical of microporous solid.

Table 2.2 Sample identification for activation conditions and production yield

Sample identification	ZnCl_2 impregnation	Pyrolysis in N_2	CO_2 activation	Steam activation	Yield (%)
CN_2	No	Yes	No	No	22.20
$\text{CN}_2\text{-CO}_2$	No	Yes	Yes	No	21.67
$\text{CN}_2\text{-ST}$	No	Yes	No	Yes	13.33
CZn-N_2	Yes	Yes	No	No	44.33
$\text{CZn-N}_2\text{-CO}_2$	Yes	Yes	Yes	No	32.60
$\text{CZn-N}_2\text{-ST}$	Yes	Yes	No	Yes	80.30

Although the carbon yield from nitrogen/steam flow was the highest but it was noticed that the specific surface areas and pore volumes of carbons activated by ZnCl_2 were higher than that of none ZnCl_2 . This might due to the widen of atomic layers of carbon after the ZnCl_2 intercalated in the carbon matrix. During pyrolysis, ZnCl_2 caused hydrogen and oxygen atoms to be removed from the source materials as water rather than as hydrocarbons, thus increasing the amount of pores. As a conclusion from this study, the carbon with ZnCl_2 and pyrolysis under nitrogen flow followed by activation with carbon dioxide exhibit the highest specific surface areas and pores volumes.

Toles *et al.* (1999) studied the different activation conditions with phosphoric acid as the impregnating agents on various shell such as almond, black walnut, English walnut, macadamia nut and pecan. There were five activation methods mentioned in this paper. The condition of activations was listed in Table 2.3.

The activated carbons were characterized and examined its heavy metal removal capability. The result showed that the carbon under 'activation' only had the lowest amount of titrable surface functional groups and also the lowest copper uptake.

Table 2.3 Description of activation methods used to produce granulated activated carbons from nutshells

Method	Activation condition(gas) (temperature/time)	Initial wash step	Oxidation conditions	Cooling in furnace	Final wash step
Activation only	N ₂ 170 °C/0.5-1 h then 450 °C/1 h	100 °C/24 h	None	Yes	None
Standard	N ₂ 170 °C/0.5-1 h then 450 °C/1 h	100 °C/24 h	300 °C/4 h	Yes	None
Continuous	N ₂ 170 °C/0.5-1 h then 450 °C/1 h	None	300 °C/4 h	Yes	100 °C/24 h
Air activation	N ₂ 170 °C/0.5-1 h then 450 °C/1 h	None	None	Yes	100 °C/24 h
Quench	N ₂ 170 °C/0.5-1 h then 450 °C/1 h	None	None	No	100 °C/24 h

Rio *et al.* (2005) also carried out the study of activated carbon by chemical activation. Activated carbon preparation from sewage sludge can be considered as an attractive method in reducing sludge volume. In this case, liquid sludge was impregnated with sulfuric acid of impregnation ratio (0.5-1.5 g⁻¹), followed by varying activation temperature (600-800 °C) and activation time (60-180 minutes). As indicated in the literature, surface groups' decomposition increased with increasing activation time. The increase of impregnation ratio lead to increase in acidic character of the adsorbents produced because this factor has a strong positive impact on amount of acidic surface groups and a negative effect on amount of basic surface groups.

A similar research was done by Lua and Yang (2005) who using pistachio-nut shells to as precursors. They using different impregnation ratio ranging from 1:4 to 3:2. In the study, the pistachio-nut shells were activated in two kind of atmosphere, one was under nitrogen and the other was under vacuum conditions. As a result, the lowest impregnation ratio of ZnCl₂ to shell masses gave a better yield of activated carbon and the activated carbon prepared under vacuum conditions was always higher than that for nitrogen gas atmosphere. In vacuum case,

reduced pressure results in a decrease in temperature. Hence, the sample temperature would be lower than the temperature set for the furnace. A lower temperature would mean a reduced release of volatiles, giving rise to a higher yield as compared to that for the N₂ atmosphere.

Besides using ZnCl₂, nitrogen, carbon dioxide and steam as activation agents, another activation method by using sulphuric acid has been reported. (Legrouri *et al.*, 2005). The preparation of the activated carbon from molasses had been carried out by impregnation with sulphuric acid, followed by carbonization. The adsorption tests and the results of characterization showed that it was possible to produce high quality activated carbon from molasses.

In addition, activated carbon would also be prepared from olive stones and walnut shells (Martinez *et al.*, 2005). This could increase economic return and reduce pollution since olive stones and walnut shells are agricultural waste. They were treated with 50% and 75% (w/w) of KOH and evaluated by iodine absorption. A 25% increase in KOH concentration resulted in increased carbon yields of 102% for walnut shells and 55.2% for olive pits. In general, the olive stone activated carbon was more effective on their iodine adsorption capacity than walnut shell. This was proved when powdered carbon from olive pit obtained at a 75% KOH concentration had an adsorption isotherm similar to commercial carbon. Carbons obtained from walnut shells had a homogeneous structure with a predominance of macropores with an average 12 µm diameter. A comparison between olive pits carbon and walnut shell carbon did not show major differences in their external surface characteristics. But from the observation, it was clear that concentration of the chemical agent used during activation would contribute to the development of internal microporous cavities resulting in an increased surface area.

The activated carbons are usually characterized by a polydisperse porous structure consisting of different pore sizes and shapes. The classification of pores suggested by Dubinin and accepted by International Union of Pure and Applied chemistry (IUPAC) is based on their width which represents the distance between the walls of a slit shaped pore or the radius of a cylindrical pore (Nicholas and Paul, 1993). According to Galiatsatou *et al.* (2001), there are three classifications of the activated carbons depending on pore width (pw) similar to Dubinin which are micropores ($pw < 20 \text{ \AA}$), transitional mesopores ($20 \text{ \AA} < pw < 500 \text{ \AA}$) and lastly macropores ($pw > 500 \text{ \AA}$). Among the three types of pores, micropores constitute the largest surface area. Thus, the micropores determine the adsorption capacity of the activated carbon. The macropores are not very important in adsorption process because their contribution to surface area of the activated carbon is only about $0.5 \text{ m}^2/\text{g}$. Macropores allow rapid passage of the adsorbate into micro and mesopores. Consequently, build up a well develop network. Micropores are the most important for gas adsorption, mesopores for liquid adsorption and macropores for adsorption of large molecular species like colour molecules.

The characteristic of the starting material, the activating agent concentration, and the carbon particle size will influence the adsorption capacity. Hence, in this study powdered PET has been chosen as the starting raw material and examines its application in wastewater treatment.

CHAPTER THREE

METHODOLOGY

3.1 Sample preparation

In this study, polyethylene terephthalate samples were obtained from post consumer soft drink bottles such as Coca-Cola, Pepsi Cola or mineral water bottles.

3.1.1 Contaminants removal from used PET bottles

Post-consumer PET samples were being grounded into flakes and thoroughly washed with distilled water and detergent in the sequence: distilled water, detergent, distilled water to ensure that most contaminants had been removed.

3.1.2 Drying

After washing, the PET flakes were dried at 105 °C until constant weight to ensure total removal of the water present in the samples.

3.2 Preparation of activated carbon

The carbonization of PET samples and activation was based on the method reported by Laszlo and Szucs (2001) as shown in Figure 3.1. PET samples were being heated in a tube furnace at 750 °C under a constant flow of nitrogen for 30 minutes. The carbonized samples were then cooled to room temperature. For activation, the carbonized samples were subsequently being heated to 600 °C under constant nitrogen flow for 4 hours.

Figure 3.1 Flow chart on the preparation and characterization of activated carbon from used PET bottles

