

INVESTIGATION ON SPENT TEA LEAVES DERIVED ACTIVATED CARBON FOR CO₂ ADSORPTION

R. MENON, J. SINGH, V. DOSHI, XIAO Y. LIM*

Energy Research Group, School of Engineering,
Taylor's University, Taylor's Lakeside Campus,
No. 1 Jalan Taylor's, 47500, Subang Jaya, Selangor DE, Malaysia
*Corresponding Author: xiaoyien.lim@taylors.edu.my

Abstract

In this study, spent tea leaves are used as raw material to produce activated carbon (AC). Characterization of used tea leaves-derived AC is studied as low-cost adsorbents to capture carbon dioxide (CO₂) which has been a major contributing factor in relation to global warming. Effects of parameters such as activation sequence, activating chemical agents, ratio of activating agent to tea leaves, activation temperature and heating rate during AC production and its potential to adsorb CO₂ are investigated. The characterization of the spent tea leaves derived AC was analysed using Thermogravimetric analysis (TGA) and Brunauer-Emmett-Teller (BET). For an effective CO₂ adsorption to occur, properties such as large surface area, considerable amount of micropores and suitable pore width are desired. From this study, the total surface area of the ACs produced varied from 311 to 1044 m²/g and total pore volume is ranged from 0.18 to 0.59 cm³/g respectively. Chemically activated sample using H₃PO₄ at a ratio of 2:1 (CAP2) generated the highest micropore volume of 0.59 cm³/g and surface area of 1044 m²/g, making it the most optimum parameters in this study. Changes in activation temperature and activation sequence have the most extensive effect on the properties of the AC.

Keywords: Activated carbon, Tea leaves, Micropores, Adsorption, Carbon dioxide

1. Introduction

In recent decades, air pollution has become a major concern amongst the community. The increase in volume of human activities such as industrialization and urbanization has been the major contributing factor to air pollution. One of the primary air pollutants is carbon dioxide (CO₂). CO₂ is primarily produced

from the combustion of fossil fuels such as coal and petroleum distillates which are widely used as a source of energy in transportation and electricity generation. In accordance to the International Agency report (2007) on world energy statistics, coal contributed about 29 billion metric tons of CO₂ which made up to 42% of the world CO₂ emissions in 2007 alone [1]. Unfortunately, the utilization of fossil fuels in these areas is unlikely to reduce in the near future as there is no practical alternative yet. Thus, preventing CO₂ emissions from these activities is very unlikely at this stage. Therefore, there is an urgent need to capture and control the emission of CO₂.

There are many available technologies for CO₂ removal. The technology that is recently receiving much attention is adsorption due to its simple process and low cost of operation [1]. Adsorption uses adsorbents as a capturing material, and one of the most widely used adsorbent is activated carbon (AC). AC's large active surface area, favourable pore size distribution, surface chemistry and the degree of polarity makes it very suitable for many environmental applications [2-5]. Charcoal is the most suitable raw material for AC production due to its rich carbon content. Unfortunately, the source of charcoal is limited and expensive. Therefore, alternative carbonaceous sources which are sustainable, easily available and cheap are being investigated. Numerous studies have been done to investigate the potential of biomass materials as an alternative raw material for AC production and many have been proven effective. Coconut husk, palm oil materials, grass cuttings, horse manure, wood, orange peel, treated sewage, wall nut shell are some of the few examples of raw biomass materials that has been used to produce effective activated carbon. [5-9].

In this study, the potential of used tea leaves as AC precursor for CO₂ adsorption will be investigated. Tea leaves are widely used all over the world to produce the world's second most consumed beverage, tea. Tea leaves are chosen because it is largely produced in Malaysia and the used tea leaves are mostly discarded as waste. This makes tea leaves a fairly cheap and environmental friendly carbonaceous source for AC production. Studies have been done previously to investigate the potential of tea leaves as AC. Akar et al. reported that the AC produced from tea leaves can be used as an efficient adsorbent for the removal of malachite green from aqueous solution [3]. Besides, another study conducted by Gundogdu et al. has shown that the activated carbons produced from tea industry waste have different physicochemical characteristics by undergoing different pre-treatment process by utilizing the same precursor [4]. Therefore, it is evident that tea leaves exhibit as a good raw material for AC precursor. From the best of author knowledge, no investigation have been made on used tea leaves derived AC for CO₂ removal applications. Thus, this study will focus on investigating the potential of spent tea leaves derived CO₂ adsorbent by manipulating the carbonization temperature, heating rate, activating agent, ratio of activating agent to biomass, and activation sequence.

2. Experimental Procedure

2.1. Materials

The raw material used for the production of activated carbon is spent tea leaves. An average of 4 kg spent tea leaves were obtained from each restaurant around Subang

Jaya area, Selangor, Malaysia daily. The spent tea leaves obtained from the restaurant has a mixture of all types of tea leaves however majority of the tea leaves are fermented black tea. This study will be focusing on five different parameters which are activating sequence, activating agents, activating agent to biomass impregnation ratio, carbonization temperature and carbonization heating rate. The collected sample were washed several times with distilled water for the removal of impurities and dried in a forced air convection oven (Model FAC -50, Tech-Lab manufacturing Sdn Bhd., Malaysia) at 110°C for 24 hours. The two chemical activating agents used were anhydrous Potassium Carbonate (Lab grade, R&M Chemicals Ltd., United Kingdom) and Potassium Hydroxide (Lab grade, R&M Chemicals Ltd., United Kingdom). For the neutralization process 1% Hydrochloric Acid (Lab grade, R&M Chemicals Ltd., United Kingdom) was used [10-12].

2.2. Preparation of activated carbon

2.2.1. Chemical activation

For the chemical activation, Phosphoric acid (H_3PO_4), Potassium Hydroxide (KOH) and Potassium Carbonate (K_2CO_3) were added into the spent tea leaves. The dried tea leaves were first impregnated in 20 wt% H_3PO_4 with impregnation chemical to tea leaves ratios of 1:1 and 2:1 (g/g). The tea leaves were then allowed to stand for 24 hours at room temperature. After that, the mixture was filtered and the impregnated product is dried for about 15 hours in an oven at 110°C. The product was then carbonized at temperature of 700°C for 2 hours in a furnace (Model KSL-1100X, MTI Corporation, USA). After being cooled down to room temperature, the product was neutralized with an alkaline solution until neutral pH of 7 was achieved. Finally, the product was washed with distilled water to remove any impurities. The washed sample was then placed in the forced air convection oven (Tech- Lab manufacturing Sdn Bhd., Model FAC -50, and Malaysia) and dried at 110°C for 24 hours [12-13].

The same steps were repeated by replacing H_3PO_4 with 50wt% KOH as the impregnation chemical with 1:1 and K_2CO_3 of 1.5:1 ratio [10, 12, 14]. The selected temperatures were 600°C and 800°C, at a heating rate of 5°C/min and 10°C/min. These samples were neutralized using 1% of hydrochloric acid and washed repeatedly with distilled water until it reached the required pH of 7.

2.2.2. Pre-treated chemical activation

For pre-treated chemical activation, the dried tea leaves were first pyrolysed (carbonized) in a furnace under nitrogen gas flow. The furnace was set at a rate of 10°C/min to the optimal temperature of 600°C. Next, the carbonized substance was impregnated with 20 wt% phosphoric acid with activating agent to tea leaves ratios of 1:1 and 2:1 (g/g), respectively. The mixture was allowed to stand for 24 hours at room temperature. After 24 hours, excess solution was filtered out and the impregnated tea leaves were dried at 110°C for 15 hours in oven. The obtained sample was heated at 800°C for 2 hours in a furnace. After being cooled down to room temperature, the product was neutralized by an alkaline solution

until pH value of 7 is obtained. Finally, the product was washed with distilled water and dried at 110°C in an oven for about 15 hours. Each sample was labelled with an ID name for future reference as listed in Table 1 below:

Table 1. Sample identification of spent tea leaves derived AC.

Sample ID	Method	Activating Agent	Activation ratio	Activation Temperature (°C)
CAK8-10	Chemical Activation	KOH	2:1	800
CAK6-10	Chemical Activation	KOH	2:1	600
CAK6-5	Chemical Activation	KOH	2:1	600
CAKC6	Chemical Activation	K ₂ CO ₃	1.5:1	600
CAP1	Chemical Activation	H ₃ PO ₄	1:1	700
CAP2	Chemical Activation	H ₃ PO ₄	2:1	700
PCAP1	Pre-treated chemical activation	H ₃ PO ₄	1:1	700
PCAP2	Pre-treated chemical activation	H ₃ PO ₄	2:1	700

2.3. Proximate and ultimate analysis

TGA analyzer was used to perform proximate analysis to obtain information on moisture, ash, fixed carbon and volatile matter content in used tea leaves. Firstly, 15 mg of tea leaves sample was heated to 110°C under nitrogen gas for 20 minutes to eliminate the moisture content in the sample. Then, the temperature was increased to 950°C and held for 30 minutes under nitrogen flow to remove volatile matters from the sample. After 30 minutes of heating at 950°C, the nitrogen gas supply was cut off and switched to air for 20 minutes to allow combustion of carbon to take place, leaving only ash behind. The heating was done at a rate of 10°C/min.

Elemental analyzer (Euro Vector SPA., Model:EA3000 series, Milan) was used to perform ultimate analysis in which carbon, hydrogen, nitrogen and sulphur contents of used tea leaves and pre-treated used tea leaves can be determined. Firstly, 0.5 g of sample was combusted in sample of tin and vanadium pentoxide catalyst, purified by a reactor packed with electrolytic copper and copper oxide. Gasses such as N₂, CO₂, H₂O and SO₂ enter the chromatographic column for separation to take place. Eluted gasses are sent to the thermal conductivity (TCD) where electrical signals processes by the Eager 300 software provide values of nitrogen, carbon, hydrogen and sulphur content in percentage that is in the sample.

2.4. Surface area, pore volume and pore size distribution analysis

Nitrogen adsorption isotherms at 77 K were determined using a Quantachrome Instrument system after an outgas pretreatment at 300 °C for 5 hours. The surface area was obtained from the application of multipoint N₂ adsorption isotherm with

the Branauer-Emmett-Teller (BET) equation at a pressure of $P/P_0 = 0.3$. As for the pore size distribution, a slit pore model using Non Local Density functional

Theory (NLDFT) method was used. The Dubinin-Radushkevich (DR) model was used to calculate the total and micropore volume with a relative pressure of 0.95 bar. A relative fitting error between the measured isotherm and the fitted isotherms at a range of 0.03 to 0.35% was varied throughout the whole experiment.

3. Results and Discussion

3.1. Proximate analysis and ultimate analysis

Table 2 shows the moisture, volatile matter, fixed carbon and ash content respectively on the raw and carbonized spent tea leaves. Carbonization reduces the volatile matter of 70 % in raw spent tea leaves sample to 17 %. At high temperatures, the organic substances in the material would degrade causing instability in the carbon matrix which will result in formation of gas and liquid tar as well as high carbon content [15, 16]. From Table 2, before carbonization process, the raw spent tea leaves had a fixed carbon content of 22% and after carbonization it increased to 68%. For the ash content, the raw spent tea leaf had extremely little ash content of 0.5%, which is an ideal material to be used as AC carbon precursor as the ash content in biomass generally range between 2% to 10%. High ash content can cause catalytic effect and increase hydrophilic structure during regeneration process [15, 17]. The increase in ash content in carbonized spent tea leaves is due to the residual remains of the inorganic substances in the material.

Table 2. Proximate analysis of raw sample and carbonized sample.

	Moisture level (%)	Volatile matter (%)	Fixed Carbon (%)	Ash Content (%)
Raw spent tea leaves	7.5	70.0	22.0	0.5
Carbonized spent tea leaves	5.0	17.0	68.0	10.0

Table 3 shows the ultimate analysis of the raw and carbonized spent tea leaves. Ultimate analysis of the raw tea leaves had a reasonable amount of carbon content of 46.40% and after carbonization the amount of carbon increased to 71.91%. As for the amount of hydrogen, the percentage of hydrogen decreases when exposed to high temperature because hydrogen element has a very low molecular weight thus, the hydrogen content is easily vaporized from the hydrocarbon formation when carbonized [18,19]. The sulphur content in the raw spent tea leaf was around 1 % and after carbonization it increases to 2.3%. Low sulphur content is preferred as high sulphur content in the activated carbon can cause corrosion, clinking and slugging in the equipment [20-22].

Table 3. Ultimate analysis of raw and carbonized spent tea leaves.

	Carbon (%)	Hydrogen (%)	Nitrogen (%)	Sulphur (%)
Raw spent tea leaves	46.40	6.81	3.83	1.02
Carbonized spent tea leaves	71.91	2.42	4.07	2.30

3.2. Surface area and pore analysis on spent tea leaves derived activated carbon

Table 4 shows the surface area and micropore volume of the activated carbons derived from spent tea leaves. It is found that pre-treated chemical activation samples, PCAP1 and PCAP2 yield the lowest surface area of 336 m²/g and 311 m²/g respectively, compared with the rest which used the chemical activation route. It is noted that the pre-treated chemical activation also gives the lowest micropore volume of 0.14 - 0.15 cm³/g among all the samples. This result is aligned with the findings in Lee study, where the acid treatment sequence is a significant indicator during AC preparation process [23]. Similar findings were seen, when comparing pre-treated chemical activation samples (PCAP1 and PCAP2) with the chemically activated samples (CAP1 and CAP2). Chemical activation samples yielded much higher surface area of 761 and 1044 m²/g for CAP1 and CAP2, respectively when compared with pre-treated chemical activation samples. The large difference between the surface area of these two sets of sample is due to the activating sequence. In the chemical activation method, the acid was added before carbonization process hence, the presence of acids in sample may have contributed to the devolatilization process and generating pores, which subsequently resulted in much better porosity. On the other hand, for pre-treated chemical activation sample, the acid treatment was done after the carbonization process and a reduction of more than 50% in BET surface area and total pore volume is observed. As the elemental form of sulphur is generated after carbonization, it is deduced to remain on the surface of char that blocked the acid passage to the pore entrance [23]. Therefore, this inhibited the acid treatment which promote particle shrinkage and volume contraction that yielded substantial pore volumes. These results indicated that the sequence of acid treatment in producing AC does play an important role in development of internal surface area and pore volume.

With the chemical activation method, all the activated carbons show a higher BET surface area of 478 m²/g and above with micropore volume at least 0.19 cm³/g. The KOH activating agent shows a lower micropore volume compare with K₂CO₃ and H₃PO₄. Between both alkaline activating agents, K₂CO₃ performs better than KOH as chemical activating agent. This could be that carbonates has better catalytic reactivity towards the dehydration of spent tea leaves compared to hydroxides [10, 24-25]. In short, K₂CO₃ has better ability to decompose the lignocellulosic biomass of the tea leaves compared to KOH. Thus, for a more efficient and environmental friendly AC, the use of K₂CO₃ as an activated carbon is much encouraged [10]. Besides, the CAKC6 has the highest micropore volume (86%) out of the total pore volumes among all the AC samples. On the other hand, H₃PO₄ shows the highest total surface area of 1044 m²/g and largest micropore volume of 0.39 cm³/g. Few studies have concluded that with the impregnation of phosphoric acid into the biomass, the phosphoric acid transforms into different phosphorous compounds at temperature higher than 450°C [26, 27]. The volatilisation of these compounds includes H₂PO₄⁻¹ and H₂P₂O₇⁻² can produce new channels in the carbon structure which leads to an increase in the porosity at high temperature. For H₃PO₄ activation, the H₃PO₄ to spent tea leaves ratio plays an important role. An increase in H₃PO₄ impregnation ratio from 1 in CAP1 to 2 in CAP2 increases the amount of phosphoric acid incorporated into the spent tea leave structure, and therefore, increases the surface area, total pore volume as well

as the mesopore volume. This is in good agreement with other findings that linked to the polymeric species length of dehydrated acid, polyphosphoric acid involved in the activation process. At higher impregnation ratio, the polymers length increases and therefore promotes wider pores [26].

For chemical activation method, CAK8-10 and CAK6-10 samples are used to compare the effect of carbonization temperature. CAK8-10 which carbonized at higher temperature of 800 °C produces a lower surface area of 654 m²/g compared with 711 m²/g of CAK6-10 which carbonized at 600 °C. Similar results were obtained in other studies which indicated that when carbonization temperature increases, the total pore volume and micropore volume increase too. However, at higher temperature of 800 °C, the surface area decreases due to the breakdown of micropore wall at high temperature [10, 28-30]. Although CAK8-10 has reduced surface area, its micropore volume of 0.24 cm³/g (60% of total volume) is still higher than CAK6-10 of 0.19 cm³/g (42% of total volume). This can be shown that pores developed at high carbonization temperature of 800 °C where volatilization of organic matters is at its peak are mainly micropores, less than 2nm.

From samples CAK6-5 and CAK6-10, it is observed that heating rate does not have any impact on micropore volume since the micropore volume remains stable, 0.19 cm³/g. However, higher heating rate shows a noticeable effect in formation of mesopores which contribute to the increase in total pore volume of 0.45 cm³/g in CAK6-10. Slow heating rate increases the time for the volatiles to reside within the carbonizing particles, and facilitates the polymerization of some volatile components [27]. The difference in pore volume is because at lower heating rate the volatile product removed per unit time is lower compared to a higher heating rate. At lower heating rate, a longer time is taken to complete carbonization process, allowing complete removal of the volatile matter in sample thus, allowing complete breakdown of the micropore wall. This reaction would also contribute in the reduction of micropore volume, and increase the formation of mesopores in the sample [31, 32].

Table 4. Surface area and pore analysis of spent tea leaves derived activated carbons.

Sample ID	Surface area (m ² /g)	Micropore volume (cm ³ /g)	Total volume (cm ³ /g)
CAK8-10	654	0.24	0.40
CAK6-10	711	0.19	0.45
CAK6-5	478	0.19	0.39
CAKC6	733	0.31	0.36
CAP1	761	0.31	0.42
CAP2	1044	0.39	0.59
PCAP1	336	0.15	0.19
PCAP2	311	0.14	0.18

3.3. Pore size distribution on spent tea leaves derived activated carbon

Among the factors of types of activating agent, activating agent to carbon precursor ratio, activation sequence carbonization temperature and heating rate,

heating rate shows the least effect on the pore size distribution in the spent tea leaves derived ACs. As shown in Fig. 1, the micropores formed on both CAK6-5 and CAK6-10 has same pore width which peaked at 0.6nm and 1nm. For an effective gas adsorption processes the pore size of an adsorbent to capture the gas has to be maximum 5 times the adsorbents molecular size. Thus, with CO₂ having a molecular size of 0.209 nm the pore size of an activated carbon has to be lesser than 1nm for effective adsorption [6]. However, too small pore sizes (<0.26nm) also prevent carbon dioxide adsorption.

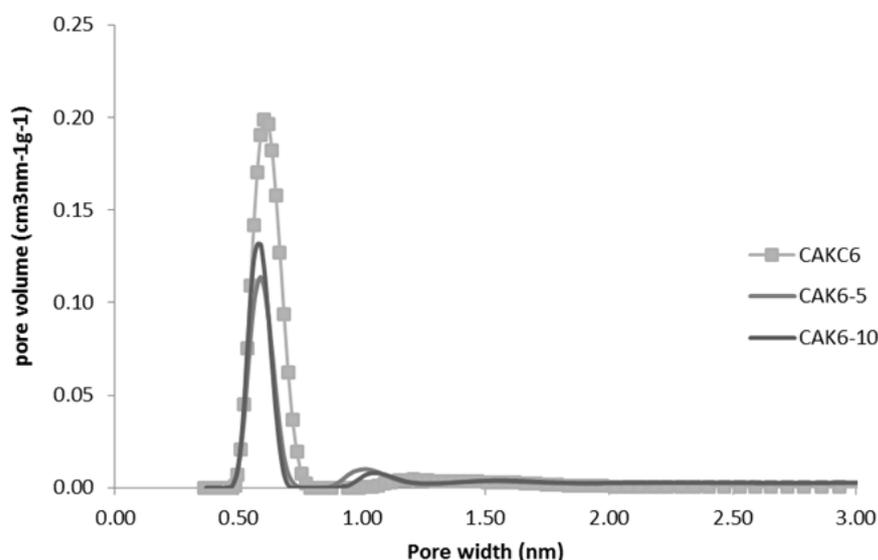


Fig. 1. The pore size distribution of CAK6-5 and CAK6-10.

Figure 2 shows the pore size distribution of CAK6-10, CAK8-10 and CAP2. With same activation agent of KOH, CAK6-10 and CAK8-10 showed a distinct difference in the pore size distribution. At higher carbonization temperature of 800 °C, the CAK8-10 sample had a broader range of micropore width compared to the CAK6-10 sample which produced a more uniform pore width of 0.6nm. This supports the surface area and micropore volume findings in Table 4 that indicated the high temperature of 800°C generates more micropore due to volatilization effect but with the friable carbon framework, contraction or collapse of pores would lead to the expansion of pore size. Another interesting point found in this study when comparing two sets of sample of different activating agent was that, CAKC6 sample in Fig. 1 and CAK samples (include CAK6-5 in Fig. 1, CAK8-10 and CAK6-10 in Fig. 2) which used K₂CO₃ and KOH respectively, had produced a corresponding pore size distribution for both the samples where the maximum pore width was around 0.65 nm. On the other hand, more peaks and higher intensity are observed in the CAP samples (CAP1 and CAP2) at pore width of 0.6, 0.9 and 1.7 nm compared with the alkaline activating agent. This indicates that the chemical activation of spent tea leaves using phosphoric acid resulted in development of micropores of a few sizes.

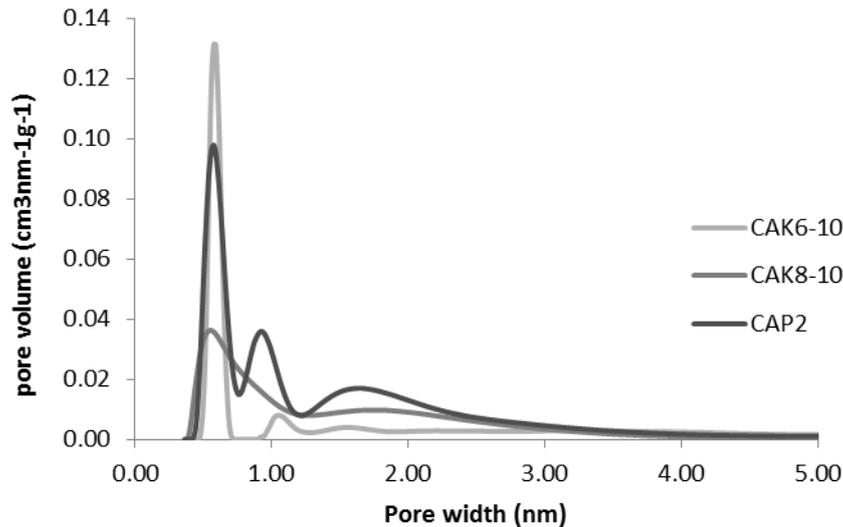


Fig. 2. The pore size distribution of CAK6-10, CAK8-10 and CAP2.

4. Conclusions

In this study, a low cost and environmental friendly activated carbon adsorbent is developed from spent tea leaves. Based on the proximate and ultimate analysis, it is found that the spent tea leaves have good carbon content, low ash and moisture content making it a suitable raw material for activated carbon. Activation method is compulsory to improve the characteristics of spent tea leaves for activated carbon purposes to adsorb CO₂. The effect of activation sequence strongly affects the total BET surface area which chemical activation is highly recommended. In terms of activating agent, H₃PO₄ produced pores with highest micropore structure (86 % of the total pore volume) and surface area of 1044 m²/g. In addition, for the effect of temperature towards the surface area and micropore size, it was found that the optimum temperature is 600°C as higher surface area was obtained compared to 800°C and contains more pores with pore width of 1nm which is suitable for CO₂ gas adsorption. Another parameter, heating rate is found to have the least effect on the formation of micropore structure and pore size distribution. This implies that it would not have significant impact during the production of spent tea leaves derived activated carbon for CO₂ adsorption process.

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