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Feasibility of Paper–based Activated Carbon Fibers as Fried Oil Adsorbing Material

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This study used corrugated cardboard (CC) as a precursor, and prepared the paper–based activated carbon fibers (PACFs) using steam activation, and evaluated the feasibility of using PACFs as fried oil adsorbing material. The yield of PACFs prepared under different activation conditions was 16.50–27.16%, the iodine value was 205.77–586.05 mg/g, and the methylene blue adsorption was 226.98-311.61 mg/g. Under the BDDT classification, the PACFs belonged to Type Ⅱ, characterized by a large amount of mesopores and hysteresis loop phenomenon. Based on the IUPAC classification, PACFs were the slit pores of Type H3. According to the physicochemical properties of frying oil after heating, the unsaturated fatty acid generated free radicals or performed polymerization to form polymers as the heating time increased. The color change and viscosity were increased. Fried oil generated free fatty acid due to heating and hydrolysis and then the acid value increased, while the smoke point decreased. Fried oil generated hydroperoxide and nonvolatile substance due to oxidation reaction. The peroxide value and thiobarbituric acid value were increased. In addition, the PACFs were ground into GPACFs (Granular PACFs) and DPACFs (Powdered PACFs), and different percent weight of them was used as the adsorbing material. The viscosity, acid value, peroxide value, and thiobarbituric acid value of the fried oil were influenced. The 5% impregnating was able to decrease the aforesaid items by 30.6, 17.1, 40.9, and 71.3%, respectively.

Key words: Corrugated Cardboard (CC), Paper–based Activated Carbon Fibers (PACFs), Fried Oil, Acid Value, Peroxide Value, Thiobarbituric Acid Value

INTRODUCTION

The Environmental Protection Administration of Taiwan (2018) indicates that Taiwan's average annual waste yield is about 3 714 541.67 t/year, and waste paper is 1 968 408.33 t/year, which is about 53% of the total waste yield. Waste paper is treated to make recycled paper, and the usability is degraded as the fiber length is shortened or/and the strength is decreased. To enhance the reproducibility of waste paper, such as the carbon material with absorbability, the waste paper recycling is one of the reused methods (Ellis and Sedlachek, 1993). As activated carbon is a carboniferous material with high specific surface area and porosity, it contains lots of pores with good absorbability and is extensively used for gas/liquid phase adsorption. The fibrous structures of wood pulp and spent kraft pulp are composed of plant cellulose. While the wall pores of the wood fiber are carbonized and activated to prepare wood–based activated carbon fibers (WACFs) (Huanh *et al*., 2010; Lin *et al*., 2015a; Lin *et al*., 2015b), the pore structure and absorption remain (Asakura *et al*., 2004). WACFs is free of mutagenicity (Lin *et al*., 2014a; Lin *et al*., 2015a) according to the Ames test (Ames, 1975), and there is no adverse effect according to the results of 28–day feeding toxicity test on animals (Lin *et al*., 2014b; Lin *et al*., 2015b).

Foods fried at high temperatures have special aroma and crispness, and because the cooking time is short, it is well accepted by common people. However, food fried at high temperatures performs hydrolysis, oxidation, polymerization, pyrolysis, and thermal polymerization to generate free fatty acid, alcohols, cyclic peroxide, dimer, and polymer (Choe and Min, 2006), which are toxic, and there are some of adverse effects on human health (Ziaiifar *et al*., 2008). Various countries have specified the required quality of fried oil. In terms of fried oil quality regulations in Taiwan, according to Letter Wei–shu–Shi–Tze–Di#098461015 (2010), the F&B services fried oil check management principle–The Food nutrient database from the Ministry of Health and Welfare, the smoke point lower than 170℃, the fried oil has a deep color and rancid odor, the foam area exceeds one and half of the fryer, and the acid value exceeds 2.0 mgKOH/g oil. Moreover, the present adsorbents are mostly activated carbon, zeolite, silicate, bentonite, diatomite, and perlite powder (Paul *et al*., 1997). Lin *et al*. (1998) indicate that activated carbon, calcium silicate, and magnesium silicate can reduce free fatty acid, lighten the color of fried oil, and extend oil and fat oxidation stability for effectively slowing the deterioration of fried oil. Yates and Caldwell (1993) use activated carbon or magnesium silicate to remove oil soluble substance, which postpones the rancidity of oil and fat more effectively than diatomite. The Ministry of Health and Welfare (2010) indicates the range of use and limit of food additives and specification standards in Letter

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Shou–Shi–Tze–Di#1001301966, meaning that, while synthetic magnesium silicate is approved as a food processing filter aid (below 2%), the effects of synthetic magnesium silicate, diatomite, and perlite powder remain uncertain for human health.

Therefore, this study used corrugated cardboard (CC) to prepare paper–based activated carbon fibers (PACFs) to investigate the effect of fried oil adsorbing material. The CC was used as the precursor to prepare different PACFs under different steam activations of physical activation. The basic properties of PACF were investigated. Afterwards, soybean oil was heated for the fried oil test. PACFs of different percent weight and sizes were impregnated into the fried oil, and the physical analysis method was used to determine color change, smoke point, and viscosity. The chemical analysis method was used for the acid value, peroxide value, and thiobarbituric acid value. Rancimat oxidation induction time was used to determine oil and fat oxidation stability as well. The fried oil quality before and after impregnating was determined to evaluate the effect of the PACFs made of CC on adsorbing the fried oil.

MATERIALS AND METHODS

Specimen and its basic properties

Precursor

Corrugated cardboard (CC), the precursor, was obtained from C company after being cut with the dimension of 1 × 1 cm (without printing), and then oven–dried at 105°C.

Oil specimen

Soybean oil was purchased from Haibao powder store in Chiayi, Taiwan.

Basic properties of CC

1. Moisture content (MC): the MC of CC was measured by the Chinese National Standards (CNS) 1356 (2010) Method of Test for Determination of Moisture Content in Pulp, Paper and Board (Oven–drying Method). The air–dried MC of CC was measured.
2. Ash: According to the CNS 1356 (2008) Methed of Test for Residue Ash Content of Pulps, Paper and Board on Ignition at 525°C. The ash content of CC was measured.

Chemical composition analysis of CC

The chemical composition analysis of CC were included the contents of holocellulose and lignin and ethanol–toluene extractives.

1. The holocellulose of the CC was measured according to the CNS 4713 (2005) Method of Test for Holocellulose Content of Pulpwood and Other Fibrous Materials. The formula for holocellulose (%) = (the absolute dried weight of holocellulose / the absolute dried weight of precursor) × 100.
2. The lignin content of CC was tested by the CNS 2721 (2010) Method of Testing for Determination of Acid–Insoluble Lignin in Pulp and the CNS 12108 (1987) Method of Testing for Acid–Soluble Lignin (Klason lignin) in Wood and Pulp. The formula for acid–insoluble lignin (%) = (the absolute dried weight of lignin / the absolute dried weight of precursor) × 100. The formula for acid–insoluble lignin (%) is (B × V × 100) / (1000 × W), where B is the concentration of acid–soluble lignin (g/L); V is the total volume of solution (mL), and W is the absolute dried weight of the specimen. Furthermore, B = (A × D) / 110 where A is the absorbance and D is the diluted fold. Lignin content (%) = acid–insoluble lignin + acid–soluble lignin.
3. The ethanol–toluene extractives of CC were measured according to the CNS 4713 (2005) Method of Testing for Ethanol–Toluene Extractives in Wood. The formula for ethanol–toluene extractives (%) = (the absolute dried weight of extractives / the absolute dried weight of precursor) × 100.

Evaluation of PACFs as Fried Oil Adsorbing Material

Preparation of PACFs

The precursor, CC, was dried in an oven at 105°C for 24 h. For the first step–carbonization, the resulting specimen was loaded in a crucible, which was placed inside an upright high–temperature activation furnace (inner diameter, 26 cm; inner height, 40 cm; Ch–1116, Taiwan) and was heated under a nitrogen (N_2 gas) flow rate of 200 mL/min for carbonization at a rate of 10°C/min. N_2 gas was added to make the container oxygen free. The carbonization temperature was set at 700, 750, 800, and 850°C. The second step–activation inserted the activation gas–steam, which was heated from deionized water with the flow rate set at 90 and 120 mL/h. The activation temperature was set at 700, 750, 800, and 850°C with activation duration of 60 min. In the third step, the paper–based activated carbon fibers (PACFs) was cooled by N_2 gas to a normal temperature for 4 h and taken out. The aforesaid preparation conditions refer to (Kim *et al*., 2001; Zhang *et al*., 2004; Amuda *et al*., 2007; Tseng *et al*., 2007; Aworn *et al*., 2008; Huang *et al*., 2010; Wu *et al*., 2010; Peng *et al*., 2012; Lin *et al*., 2014a and Lin *et al*., 2015a). The resulting PACFs code was an activation temperature–flow rate, such as: T700–90 for PACFs that was prepared with the carbonization temperature of 700°C with a flow rate of 90 L/h.

Characterization of PACFs

1. The yield of PACFs (based on a dry basis) was calculated using the following equation (Eq.): Yield (%) = (the absolute dried weight of PACFs / the absolute dried weight of CC) × 100.
2. The iodine value of PACFs was determined according to the Japanese Industrial Standard (JIS) K 1474 (1991) Test Methods for Activated Carbon. The formula for iodine adsorption capacity is: I = [(10 – K × f) × 12.69 × 5] / M. The abbreviations for the formula are I: iodine adsorption capacity (mg/g); K: the volume of titrated sodium thiosulfate (mL); f: the ratio of 0.1 N sodium thiosulfate to 0.1 N iodine solu-

tion, and M: the weight of absolute dried PACFs (0.5 g).

3. The methylene blue adsorption value of PACFs (1 mg) was added to a 25 mL aqueous solution containing 1 g/L of methylene blue solution and shaken at room temperature (30˚C). When the aqueous solution became colorless, methylene blue solution was repeatedly added to the flask to assure equilibrium adsorption of the MB. After filtration, the concentration of residual methylene blue solution was determined using a UV–vis spectrophotometer (CECIL, CE3041) at a wavelength of 664 nm. (Wu *et al.*, 2010)
4. The pore structure characteristics of PACFs were measured by nitrogen adsorption–desorption isotherms at 77 K using a Micromeretics ASAP2000, Accelerated Surface Area and Porosimetry System at a relative pressure (P/Po) ranging from 10–2 to 1. The BET specific surface area (SBET) was determined using the standard BET equation. (Hu and Srinivasan, 1999)

Preparation of different particle–like PACFs

The PACFs with better yield, iodine value, and methylene blue solution were selected. The GPACFs (Granular PACFs, 8 mesh) and DPACFs (Powdered PACFs, 60 mesh) were prepared and absolutely dried for future use.

Preparation of fried oil specimen

3 L soybean oil (as Blank) was heated at 180 ± 10˚C for 4 h per day for 5 consecutive days, 150 g of the fried oil specimen (as Control) was put in a conical flask after cooling every day, and sealed at 4˚C for determining the physicochemical properties.

Fried oil impregnating test

The PACFs of different percent weight (1, 3, and 5%) and different particle sizes were impregnated into above fried oil specimens. The test procedures were–described, as follows.

1. 150 g specimen was put in a 250 mL beaker.
2. 1.5, 4.5, and 7.5 g PACFs were put in the beaker, and placed in shade for 24 h.
3. The PACFs impregnated in the fried oil specimen were filtered by filter paper, and the filtered fried oil specimen was prepared for determining the physicochemical properties.

Fried oil properties determination

The fried oil heated at 180 ± 10˚C for 4, 8, 12, 16, and 20 h was the control group (Control), and the PACFs impregnated in the fried oil after different heating time were the test group. The analytical items included:

1. Physical analysis method

(1) Color change refers to the measuring method of AOCS Official Method Cc 13b–45 (2017).

(2) Smoke point refers to the measuring method of AOCS Official Method Cc 9a–48 (2017).

(3) Viscosity: 200 mL fried oil specimen was put in a 250 mL beaker and measured by viscosimeter (Brookfield DV–I + Viscometer).

2. Chemical analysis method

(1) Acid value was tested according to AOCS Official Method Cd 3d–63 – Acid Value (2017). Eq.: acid value (mg KOH/g) = [(V1– V2) × N × 56.11]/ W

Where V1: KOH ethanol solution (mL) consumed by titration sample; V2: KOH ethanol solution (mL) consumed by titration blank test; N: KOH ethanol solution strength (N); W: fried oil specimen weight (g)

(2) Peroxide value was tested according to AOCS Official Method Cd 8b–90 – Peroxide Value Acetic Acid–Isooctane Method (2017). Eq.: peroxide value =[(V1–V2) × N × 1000]/ W

Where V1: $Na_2S_2O_3$ solution (mL) consumed by titration sample; V2: $Na_2S_2O_3$ solution (mL) consumed by titration blank test; N: $Na_2S_2O_3$ solution strength (N); W: fried oil specimen weight (g)

(3) Thiobarbituric acid value was tested according to AOCS Official Method Cd 19–90 – 2–Thiobarbituric Acid Value (2017), Direct Method. Eq.: thiobarbituric acid value = [50 × (A–B)]/ m

Where 50: dilution factor; A: absorbance value of test; B: absorbance value of blank group; m: fried oil sample weight (mg)

(4) Oil and fat oxidation stability was determined by Rancimat methods referring to the AOCS Official Method 12b–92 – Oil Stability Index method (2017).

Statistical analysis

The test results are represented by a mean (standard deviation), and the test groups are compared by Duncan's Analysis. If the ρ value is smaller than 0.05, meaning a significant difference among the test groups, it is represented by different superscript upper case letters.

RESULTS AND DISCUSSION

Basic properties of CC

The air–dried moisture content of CC was 6.70%, the ethanol–toluene extractives was 2.37%, the holocellulose was 75.51%, the lignin was 11.29%, and the ash was 10.76%. The additional adhesive for making CC is possibility with a higher inorganic content (Bivainis and Jankauskas, 2015).

Characterization of PACFs

Yield, iodine value and methylene blue adsorption value

As the organic substance was volatilized after the precursor was carbonized at high temperature, the yield of PACFs decreased as the activation temperature increased (Table 1). This is because the gasified carbon content increases, the tar is dissipated and then effectively removed at activation temperatures of 800 and 850˚C (Teng and Hus, 1999). In addition, the yield decreases as the steam activation flow increases because

Table 1. Yield, iodine value and methylene blue adsorption of paper–based activated carbon fibers with different activation conditions

Specimen	Yield (%)	Iodine value (mg/g)	Methylene blue adsorption value (mg/g)
T700–90[1)]	27.16 (1.63)[a 2)]	205.77 (11.11)[d]	–[3)]
T750–90	23.16 (0.54)[b]	329.86 (10.75)[c]	240.28 (3.89)[b]
T800–90	19.50 (0.68)[c]	586.05 (16.93)[b]	247.37 (1.80)[a]
T850–90	18.38 (1.16)[c]	526.41 (16.03)[a]	245.85 (1.13)[ab]
T700–120	26.99 (0.44)[a]	288.63 (30.27)[b]	–
T750–120	22.23 (0.42)[b]	319.85 (7.26)[b]	230.69 (2.56)[b]
T800–120	17.52 (0.44)[c]	545.89 (61.20)[a]	226.98 (0.73)[b]
T850–120	16.50 (0.60)[d]	556.56 (08.98)[a]	311.61 (15.06)[a]

[1)] T700–90: T (Activation temperature) –flow rat
[2)] Mean (standard deviation) with the different superscripts are significantly different ($\rho < 0.05$) by Duncan's multiple range tests
[3)] –: non detected

steam activation strongly erodes the precursor, and the surface oxidation (Okada *et al*., 2003; Asakura *et al*., 2004). The iodine value increased with activation temperature, and in the same activation duration the iodine adsorbance of PACFs increased with activation temperature. Aworn *et al*. (2008) reports that the steam increases with activation temperature, the activated carbon micropores are widened continuously in Stage 2 of physical activation, and adjacent micropores are lost by activation and disintegrate to form wider micropores and mesopores. Therefore, the results were showed that the pore volume was increased, leading to a higher methylene blue adsorption and lower yield.

Porosity of PACFs

The BET specific surface area of the PACFs prepared from CC at the activation temperature of 800°C and steam flow of 90 mL/h was 191.45 m^2/g, the total pore volume was 0.15 cm^3/g, the microporosity was 25.7%, the mean pore size was 3.16 nm, the isotherm was classified according to BDDT classification (Gregg and Sing, 1982), and T800–90 was a Type Ⅱ, meaning the pores were mostly mesopores (results not shown in Table). In the adsorption process, the monolayer adsorption is formed first, and then multilayer adsorption is performed. Afterwards, there is a capillary condensation phenomenon in the pores, where the adsorption/desorption isotherm generates a hysteresis loop, and it is Type H3 according to International Union of Pure and Applied Chemistry classification, meaning the pores are slit type (Brunaver, 1943).

Physicochemical properties of fried oil

The peroxide and polymer resulted from the oxidation, hydrolysis, and isomerization of fried oil influences the color change, and increases the turbidity of the fried oil (Lin *et al*., 1998). After the soybean oil was heated for 4, 8, 12, 16, and 20 h, its color gradually changed from golden yellow into reddish brown (Table 2). The red value (R value) increased slightly, the yellow value (Y value) increased from 0.17 to 2.67, and the blue value (B value) unchanged.

The smoke point of frying oil is influenced by the oxidative pyrolysis product content of micromolecules, such as free fatty acid, where the free fatty acid is derived from the hydrolytic reaction, which increases

Table 2. Physicochemical properties of frying oil before/after heating

Specimen	Heating time (h)	Color change		Smoke point (°C)	Viscosity (cP)	Acid value (mg KOH/g oil)	Peroxide value (meq/kg oil)	Thiobarbituric acid value (mg/kg oil)	OSI[4)] (h)
		R value[3)]	Y value						
Blank[1)]	0	0.00 (0.00)[5)]	0.17 (0.05)[c]	237.0 (4.57)[a]	51.40 (1.81)[c]	0.11 (0.00)[d]	0.00 (0.00)[c]	0.154 (0.02)[b]	7.33 (0.24)[a]
Control[2)]	4	0.00 (0.00)	0.50 (0.05)[c]	229.3 (0.00)[ab]	53.73 (3.21)[c]	0.15 (0.03)[d]	1.33 (0.11)[c]	0.156 (0.05)[b]	5.31 (0.37)[b]
	8	0.00 (0.00)	0.63 (0.05)[c]	227.3 (2.46)[b]	58.33 (5.22)[c]	0.24 (0.03)[c]	1.79 (0.17)[c]	0.191 (0.06)[b]	4.23 (0.09)[c]
	12	0.00 (0.00)	0.80 (0.00)[bc]	221.5 (5.92)[b]	66.57 (5.21)[bc]	0.26 (0.03)[c]	4.56 (1.46)[b]	0.196 (0.05)[b]	3.53 (0.41)[d]
	16	0.00 (0.00)	1.30 (0.50)[b]	209.4 (4.41)[c]	80.90 (3.91)[b]	0.33 (0.00)[b]	5.31 (1.03)[b]	0.234 (0.05)[b]	3.46 (0.25)[de]
	20	0.20 (0.00)	2.67 (0.47)[a]	204.7 (2.82)[c]	121.67 (14.09)[a]	0.41 (0.03)[a]	13.43 (0.97)[a]	0.682 (0.07)[a]	2.91 (0.00)[e]

[1)] Soybean oil without heating
[2)] Control (C): The fried oil heated at 180±10°C for 4, 8, 12, 16, and 20 h without impregnating paper–based activated carbon fibers (PACFs)
[3)] R value: Red value; Y value: Yellow value; B value: Blue value (non detected)
[4)] OSI: Oil Stability Index
[5)] See Table 1[2)]

with frying time, thus, the oil smoke yield increases as the smoke point decreases. The fried oil is pyrolyzed into a micromolecular substance by oxidation, which reduces the smoke point (Bracco *et al*., 1981). Sun *et al*. (2007) indicate that oil smoke contains complex chemical substances, including various compounds, such as volatile substance and Polycyclic Aromatic Hydrocarbons (PAHs), thus, long–term exposure increases the risk of lung cancer. The results from Table 2 were also indicated that the smoke point of heated frying oil decreased slowly, and the temperature decreased rapidly after 16 and 20 h. The smoke point was 237.0°C before the frying oil was heated, which was decreased by 13% to 204.7°C after 20 h. When frying oil is heated, the high temperature pyrolysis and oxidation generate many free radicals, the polymerization is likely to form high polymer, and the viscosity is increased with heating temperature (Al–Harbi and Al–Kabtani, 1993). The viscosity of frying oil increased gradually with heating time, and after 20 h heating, the viscosity of frying oil was increased from 53.73 to 121.67 cP (Table 2).

After the fatty acid of frying oil is oxidized and the triglyceride oil is hydrolyzed, the free fatty acid is formed, which is an important index for determining frying oil. The longer the frying time, the more free fatty acids are generated, and the higher the acid value (Frega *et al*., 1999). After 20 h heating of frying oil at 180 ± 10°C, the acid value increased from 0.11 mg KOH/g oil to 0.41 mg KOH/g oil. After 8, 12, 16, and 20 h of heating, the acid value exceeded the CNS 749 acid value standard (2015) (0.15 mg KOH/g oil) for edible soybean oil, but the results were less than 2.0 mg KOH/g oil, according to Letter Wei–shu–Shi–Tze–Di#098461015 (2009), the Frying oil replacement standard from the Ministry of Health and Welfare.

The peroxide value is used to determine the peroxide content in frying oil, as the unsaturated fatty acid of frying oil is combined with oxygen, which forms hydroperoxide due to the autoxidation reaction in the frying process. The hydroperoxide is an unstable material, which is rapidly generated in the initial stage of frying. The peroxide value; therefore, is an index only applicable to determining deterioration in the initial stage of the oxidation of frying oil. The split product derived from heated frying oil can induce intestinal mucosa dysfunction, large intestine cell proliferation, and hepatomegaly, which are harmful to the human body (Fritsch, 1981; Stevenson *et al*., 1984). It was also known that the peroxide value of frying oil increased with heating time (Table 2), and exceeded the CNS 749 peroxide value standard (2015) (10 meq/g oil) of edible soybean oil after 20 h.

The thiobarbituric acid value is used to determine the resultant malondialdehyde of frying oil after oxidation, meaning it is an index for evaluating oil rancidity. The thiobarbituric acid value increases when the soybean oil is heated, which is due to the malondialdehyde generated by C18:3 oxidation (Buck, 1981; Huang *et al*., 1981; Keijbets *et al*., 1985). According to Table 2, the thiobarbituric acid value was 0.154 mg/kg oil before the frying oil was heated, and this value gradually increased when the oil was heated at 180 ± 10°C for different periods of time, and was 0.682 mg/kg of oil after 20 h of heating.

The electrical conductivity of a frying oil split product is determined by using the Rancimat method to evaluate the oil and fat oxidation stability (Jain *et al*., 2005). Läubliand Bruttel (1986) indicates that the induction time of the Rancimat method is mainly influenced by the content of volatile oxide; the longer the induction time, the higher the oxidation stability (Oil Stability Index, OSI). The SOI of induction time of unheated soybean oil was 7.33 h, it was 2.91 h after 20 h of heating, and the induction time was shortened, and the volatile oxide content in the frying oil increased gradually during different heating time, and the induction time of frying oil gradually shortened, meaning the SOI decreased (Table 2).

After 20 h of heating, the color change, viscosity, acid value, and peroxide value of frying oil was increased with heating time; whereas, the smoke point and oil and fat oxidation stability decreased as the heating time extends. The follow experimental was the PACFs of different sizes (8 and 60 mesh) and different percent weight (1, 3, and 5%), which were impregnated in frying oil heated for different periods of time, in order to evaluate the quality change before and after heating.

Evaluation of fried oil by PACFs impregnating

Color change

When the frying oil is heated, the unsaturated fatty acid in the oil and fat structure generates free radicals, or polymerization occurs, the frying oil is darkened, the molecular weight is increased, and the viscosity is increased. Moreover, there are even carcinogenic cyclics if the oil is overheated (Stevenson *et al*., 1984; Gutierrez *et al*., 1988; Lumley, 1988). The Y value and R value of different frying oils were increased after different heating time. The PACFs impregnating treatment was able to decrease the Y value of frying oils heated for different time (results not shown in Table). It may be because the PACFs have mesopores, which can adsorb the peroxides and polymers resulted from oxidation, hydrolysis, and isomerization. The 5% DPACFs and GPACFs impregnated in the frying oil heated at 180 ± 10°C for 8 and 12 h had better results.

Smoke point

The smoke point is one of the indexes for testing the freshness of frying oil, meaning the temperature when thin smoke begins to occur when frying oil is heated. Frying oil generates volatile or nonvolatile compounds due to hydrolysis, oxidation, and polymerization, and most of these volatile compounds drift into the atmosphere, such as aldehydes, ketones, and short chain alkane and olefin, and the free fatty acids are increased. When the oil is heated and oxidized to generate cyclic fatty acid or combined with oxidation products into dimer and polymer, the frying oil viscosity is increased, the colloid is generated, and the heat transfer is

reduced, thus, the smoke point drops (Choe and Min, 2007). Sun *et al*. (2007) indicate that frying oil heated at high temperatures generates lots of oil smoke, and long–term exposure can induce inflammatory reaction, higher oxidative stress, and cell proliferation; moreover, the oil smoke contains complex compounds, most of which are toxic, and correlated with pulmonary carcinogenesis.

The DPACFs and GPACFs of different percent weight impregnated in frying oils heated at 180 ± 10°C for 4, 8, 12, 16 and 20 h, and D5, G3 and G5 were able to increase the smoke point (results not shown in Table). As the activated carbon surface contains a basic functional group that performs neutralization between acid and a base with free fatty acids, the smoke point can be increased by reducing the free fatty acids (Proctor and Gnanasambandam, 1997). This may be because the pores after the PACFs are activated can adsorb the dimer and polymer in the frying oil, meaning the smoke point increases and the probability of lung cancer is reduced.

Viscosity

The viscosity of frying oil is influenced by the molecular length of fatty acid, meaning viscosity increases with the average length of fatty acid. In long–term heating of frying oil, the unsaturated fatty acid in the lipid structure performs polymerization, which generates high polymers, and viscosity is increased. As heat transfer is reduced as viscosity increases, the smoke point drops, which generates gases harmful to the human body (Sun *et al*., 2007).

The viscosity changes of PACFs of different sizes and percent weight impregnated in frying oils heated for different times are shown in Table 3. The viscosity of frying oil increased with heating time. This is because the soybean oil contains a lot of polyunsaturated fatty acids, and there is polymerization when it is heated at high temperature. The viscosity decreases after impregnating treatment with different PACFs, which may be because the mesopores in the PACFs after steam activation can adsorb the polymers in the frying oil, such as the results of methylene blue adsorption obtained (Table 1). The oil heated for 12 h and treated with 3 and 5% GPACFs, as well as that heated for 20 h with all PACFs, were better.

Acid value

Frying oil is hydrolyzed due to water content and heating action. The fatty acids in triglyceride oil are hydrolyzed to form free fatty acids. The longer the heating time, the more free fatty acids are formed, and the higher the acid value (Stevenson *et al*., 1984). Bennion and Hanning (1956) indicate that, the higher the free fatty acid content in oil, the lower the smoke point, the more toxic oil smoke is generated, and the greater the probability of lung cancer. According to Table 4, the acid value of frying oil increased significantly with heating time. It has reached the CNS 749 acid value standard of edible soybean oil (2015) (0.15 mgKOH/g) after 4 h heating. However, It is lower than the 2.0 mg KOH/g of frying oil (Ministry of Health and Welfare, 2009), meaning that because it may no adequate water for hydrolytic reaction, the free fatty acids are formed slowly.

The acid value of frying oil heated for 8 h was 0.24 mg KOH/g oil. The acid values of that treated with 1, 3, and 5% DPACFs impregnating were 0.18, 0.15, and 0.19 mg KOH/g oil, respectively; the acid values treated with 1, 3, and 5% GPACFs impregnating decreased to 0.18, 0.15, and 0.20 mg KOH/g oil, respectively, while the acid value decreased to 37.5% after D3 and G3 impregnate treating. The PACFs were impregnated in frying oil heated for 12 h, the acid value of oil treated with D1, D3, and different percent weight of GPACFs decreased to 0.16–0.19 mg KOH/g oil, while that of D1, D3, G3, and G5

Table 3. Viscosity of different frying oil before/after impregnated with different particle size and percent weight of paper–based activated carbon fibers

Specimen	Code \ Heating time (h)	Viscosity (cP)				
		4	8	12	16	20
Control[1)]	C	53.73 (3.21)[ax 3)]	58.33 (5.22)[ax]	66.57 (3.21)[ax]	80.90 (3.91)[ax]	121.67 (14.09)[ax]
DPACFs	D1[2)]	51.13 (1.48)[a]	51.60 (2.14)[a]	55.40 (3.71)[a]	76.40 (3.54)[a]	184.60 (8.06)[b]
	D3	49.20 (0.49)[a]	51.80 (2.51)[a]	54.80 (2.42)[a]	65.00 (3.71)[a]	186.00 (7.09)[b]
	D5	47.40 (6.00)[a]	48.83 (5.07)[a]	54.80 (8.33)[a]	70.77 (13.62)[a]	184.40 (12.64)[b]
GPACFs	G1	50.80 (0.28)[x]	54.00 (0.49)[x]	58.40 (0.75)[xy]	63.60 (5.52)[x]	185.20 (8.91)[y]
	G3	49.60 (0.75)[x]	54.00 (4.67)[x]	53.00 (2.87)[y]	64.60 (6.93)[x]	194.00 (6.65)[y]
	G5	50.67 (2.54)[x]	51.20 (5.42)[x]	55.37 (7.20)[xy]	71.87 (10.85)[x]	180.43 (11.07)[y]

[1)] See Table 2[2)]

[2)] Specimen code: D1: 1% percent weight of Granular PACFs; DACFs; G1: 1% percent weight of Powdered PACFs

[3)] Mean (standard deviation) with the different superscripts are significantly different (ρ <0.05) by Duncan's multiple range tests between different particle size of DPACFs and GPACFs

Table 4. Acid value of different frying oil before/after impregnated with different particle size and percent weight of paper–based activated carbon fibers

Specimen	Code \ Heating time (h)	Acid value (mg KOH/g oil)				
		4	8	12	16	20
Control[1)]	C	0.15 (0.03)$^{ax\,3)}$	0.24 (0.03)^{ax}	0.26 (0.03)^{ax}	0.33 (0.00)^{ax}	0.41 (0.03)^{ax}
DPACFs	D1[2)]	0.11 (0.00)^{a}	0.18 (0.00)^{b}	0.18 (0.02)^{ab}	0.31 (0.04)^{a}	0.37 (0.03)^{a}
	D3	0.11 (0.03)^{a}	0.15 (0.01)^{bc}	0.16 (0.05)^{b}	0.26 (0.03)^{ab}	0.34 (0.00)^{a}
	D5	0.13 (0.03)^{a}	0.19 (0.03)^{b}	0.22 (0.04)^{ab}	0.22 (0.05)^{b}	0.37 (0.07)^{a}
GPACFs	G1	0.11 (0.04)^{x}	0.18 (0.01)^{yz}	0.19 (0.02)^{y}	0.30 (0.02)^{x}	0.39 (0.00)^{x}
	G3	0.13 (0.03)^{x}	0.15 (0.01)^{z}	0.17 (0.00)^{y}	0.26 (0.04)^{xy}	0.37 (0.02)^{x}
	G5	0.13 (0.05)^{x}	0.20 (0.03)^{yz}	0.17 (0.05)^{y}	0.22 (0.04)^{y}	0.37 (0.07)^{x}

[1), 2), 3)] See Table 3

is 30.8–38.5%.

The PACFs were impregnated in frying oil heated for 16 h, the acid values of the oils treated with 5% DPACFs were reduced by 33.3%, to 0.22 mg KOH/g oil, respectively. The acid value of frying oil heated for 20 h was 0.41 mg KOH/g oil. After DPACFs impregnating of different percent weight, the acid values were 0.37, 0.34, and 0.37 mg KOH/g oil, respectively, meaning all of them were decreased, while that of 3%DPACFs was 17.1%, and the acid value of frying oil treated with GPACFs impregnating was 0.37–0.39 mg KOH/g oil, which was better than that without impregnating. Proctor and Gnanasambandam (1997) indicate that the basic functional group on the activated carbon surface performs acid–base action with free fatty acid, meaning the acid value of frying oil can be reduced.

Peroxide value

The peroxide value is one of the common indices for judging the degree of oxidation of oil and fat, and for determining the content of hydroperoxide generated in the initial stage of the oxidation of oil and fat. Hydroperoxide is colorless and tasteless, and it is likely to be oxidized to reoxide (Iqbal and Bhanger, 2007). According to Table 5, the peroxide value of frying oil (Control) increased from 0.00 meg/kg oil to 13.43 meg/kg oil with heating time, and that of frying oil heated for 20 h exceeded the CNS 749 peroxide value standard of 10 meg/kg of edible soybean oil. The peroxide value of frying oil is increased when the high temperature and oxygen in the air induce autoxidation, thermal polymerization, and hydrolysis of fatty acids (Chugh and Dhawan, 2014). The hydroperoxide is likely to induce intestinal mucosa dysfunction and abnormal proliferation of intestine cells, which is likely to be split into peroxide, thus, damaging different cells and contributing to cell aging or death, as well as other gastrointestinal dysfunctions, such as hepatomegaly, angiopathy, diarrhea, and maldigestion (Boatella–Riera *et al*., 2000).

When PACFs were impregnated in frying oil heated for 4 h, the peroxide value decreased from 1.33 meg/kg oil to 0.92–1.32 meg/kg oil, and the D5, G3, and G5 had the better sink rate of 25.6–30.8% (Table 5). When DPACFs and DPACFs were impregnated in frying oil

Table 5. Peroxide value of different frying oil before/after impregnated with different particle size and percent weight of paper–based activated carbon fibers

Specimen	Code \ Heating time (h)	Peroxide value (meq/kg oil)				
		4	8	12	16	20
Control[1)]	C	1.33 (0.11)$^{ax3)}$	1.79 (0.11)^{ax}	4.56 (1.46)^{ax}	5.31 (1.03)^{ax}	13.43 (0.03)^{ax}
DPACFs	D1[2)]	1.32 (0.08)^{a}	1.62 (0.04)^{ab}	2.16 (0.18)^{b}	4.46 (0.07)^{ab}	11.14 (1.10)^{ab}
	D3	1.26 (0.10)^{a}	1.44 (0.09)^{b}	2.05 (0.25)^{b}	4.31 (0.20)^{ab}	10.97 (1.08)^{ab}
	D5	0.92 (0.25)^{b}	1.39 (0.01)^{b}	1.91 (0.27)^{b}	3.82 (0.22)^{b}	7.94 (2.05)^{b}
GPACFs	G1	1.31 (0.10)^{x}	1.65 (0.04)^{x}	4.40 (0.07)^{x}	4.97 (0.12)^{x}	11.36 (1.26)^{xy}
	G3	0.99 (0.16)^{xy}	1.53 (0.11)^{x}	3.47 (0.64)^{x}	4.47 (0.20)^{x}	11.17 (1.10)^{xy}
	G5	0.93 (0.19)^{y}	1.66 (0.09)^{x}	3.44 (1.30)^{x}	4.05 (0.27)^{x}	9.56 (1.04)^{y}

[1), 2), 3)] See Table 3

heated for 8 h, the D3, D5, and G3 had better effect, and the peroxide value decreased from 1.79 meg/kg oil to 1.44, 1.39, and 1.53 meg/kg oil, respectively. The peroxide value of frying oil heated for 12 h was 4.56 meg/kg oil, while that of 1, 3, and 5% DPACFs was 52.0, 55.0, and 58.1 meg/kg oil, respectively, which was reduced by 52.0–58.1%. The peroxide value of 5% of DPACFs and GPACFs after 16 h heating decreased to 3.82 and 4.05 meg/kg oil, respectively. The peroxide value after 20 h heating increased to 13.43 meg/kg oil, but decreased after impregnating different percent weight of PACFs.

The peroxide values were able to be lower than the CNS 749 peroxide value standard (10 meg/kg oil) (2015) of edible soybean oil after D5 and G5 impregnating treatment, which were reduced by 40.9 and 28.8%, respectively (Figure 1). Boki *et al*. (1991) indicate that with a large pore volume the BET specific surface area and functional group on the surface of the activated carbon can effectively adsorb hydroperoxide, and then reduce the peroxide value of fried oil.

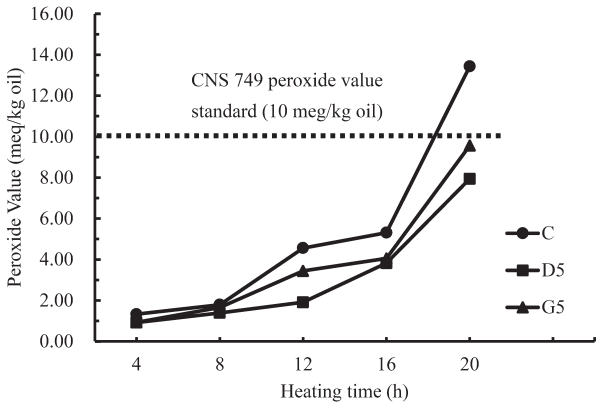

Fig. 1. Peroxide value of different frying oil before/after impregnated with different particle size and percent weight of paper–based activated carbon fibers.
Legends: C: The fried oil heated at 180±10°C for 20 h without impregnating PACFs
D5: 5% percent weight of Powdered PACFs impregnating with fried oil for 20 h
G5: 5% percent weight of Granular PACFs impregnating with fried oil for 20 h

Thiobarbituric acid value

The thiobarbituric acid value is a common index for determining the nonvolatile product malondialdehyde in the late oxidation stage of oil and fat, which is the oxidation product derived from the oxidation reaction of three unsaturated fatty acids above the double bond (Kanner *et al*., 2012). Del Rio *et al*. (2005) indicate that malondialdehyde has mutagenicity and carcinogenicity, and can cause cardiovascular diseases, such as cardiovascular tissue hardening. Malondialdehyde is likely to react with the basic group of nucleic acid, and this basic group combination can cause oxidative damage to cells, influence the physiological function of nucleic acid, and even disturb DNA to initiate hereditary disease (Marnett, 1999). The thiobarbituric acid value of unheated frying oil was 0.154 mg/kg oil, which increased to 0.682 mg/kg oil after 20 h heating at 180 ± 10°C. The thiobarbituric acid value of frying oil heated for different periods of time increased with heating time (Table 6). As activated carbon has better porosity, pore volume, BET specific surface area, and functional groups on its surface, it can effectively adsorb the organic compounds decomposed by hydroperoxide, such as aldehyde, ketone, alcohol, and acid products, in order to reduce the thiobarbituric acid value (Boki *et al*., 1991). The thiobarbituric acid values of frying oils treated with different percent weight of PACFs were reduced; frying oil heated for 16 h and treated with 5% DPACFs and GPACFs, as well as that heated for 20 h and treated with PACFs, had better thiobarbituric acid values.

CONCLUSIONS

The study evaluated the feasibility of using PACFs made of CC as the adsorbing material for heated soybean oil. The PACFs yield was 16.50–27.16%, the iodine value was 205.77–586.05 mg/g, and the methylene blue adsorp-

Table 6. Thiobarbituric acid value of different frying oil before/after impregnated with different particle size and percent weight of paper–based activated carbon fibers

Specimen	Code \ Heating time (h)	Thiobarbituric acid value (mg/kg oil) 4	8	12	16	20
Control[1)]	C	0.156 (0.05)[a3)]	0.191 (0.06)[a]	0.196 (0.05)[a]	0.234 (0.05)[a]	0.682 (0.07)[aA]
DPACFs	D1[2)]	0.146 (0.01)[a]	0.175 (0.01)[a]	0.177 (0.01)[a]	0.221 (0.01)[a]	0.228 (0.01)[a]
	D3	0.136 (0.04)[a]	0.172 (0.03)[a]	0.174 (0.01)[a]	0.208 (0.02)[ab]	0.219 (0.02)[ab]
	D5	0.133 (0.04)[a]	0.164 (0.04)[a]	0.171 (0.02)[a]	0.182 (0.01)[b]	0.196 (0.01)[b]
GPACFs	G1	0.146 (0.03)[x]	0.176 (0.01)[x]	0.181 (0.01)[x]	0.223 (0.02)[x]	0.237 (0.01)[x]
	G3	0.144 (0.00)[x]	0.175 (0.01)[x]	0.179 (0.00)[x]	0.212 (0.01)[xy]	0.227 (0.00)[x]
	G5	0.134 (0.02)[x]	0.166 (0.03)[x]	0.176 (0.01)[x]	0.188 (0.01)[y]	0.204 (0.01)[y]

[1), 2), 3)] See Table 3

tion was 230.69–311.61 mg/g. The PACFs belonged to Type Ⅱ, as characterized by a large amount of mesopores and hysteresis loop phenomenon. It was also slit type pore of Type H3. The color change, viscosity, acid value, peroxide value, and thiobarbituric acid value of soybean oil were increased with heating time, while the smoke point and oil and fat oxidation stability were decreased as the heating time extended. The 1, 3, and 5% DPACFs and GPACFs were impregnated in oil heated for different time, and the smoke point and viscosity were decreased. The oils heated for 12 h and treated with 3 and 5% GPACFs, and oils heated for 20 h and treated with all PACFs, had better results. The acid values of oil heateded for different time decreased after impregnating treatment with different percent weight of DPACFs and GPACFs. Different PACFs decreased the peroxide value of oil heated for different time. The oil heated for 12 h and treated with different DPACFs had better effect. The peroxide values of oils heated for 20 h and treated with 5% DPACFs and GPACFs were greatly decreased by 40 and 28%, respectively. The thiobarbituric acid value of oils heated for different times was decreased by different PACFs. The oils heated for 16 and 20 h and treated with 5% DPACFs and GPACFs were the best results in this study.

AUTHOR CONTRIBUTIONS

Han Chien LIN designed this study and wrote the paper. Miao–Han Yang performed the experiments, analyzed the data and the statistical analysis. Noboru FUJIMOTO participated in the design of the study and supervised the works. All authors assisted in editing of the manuscript and approved the final version.

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