RESEARCH NOTE

Synthesis of adsorbent from food industry waste for purification of used cooking oil [version 1; peer review: 2 approved with reservations]

Sulistyo Prabowo¹, Muflihah Muflihah²

¹Agricultural Products Technology, Mulawarman University, Samarinda, Kalimantan Timur, 75119, Indonesia
²Chemistry Education, Mulawarman University, Samarinda, Kalimantan Timur, 75119, Indonesia

Abstract
This study aimed to utilize food industry waste (i.e. banana (Musa paradisiaca L.) peel), as a raw material for making banana peel activated carbon (BPAC). The activated carbon-making process was conducted at varying temperatures (200, 400 and 600°C) and furnacing times (1, 2 and 3 hours). The purification function of the BPAC obtained from the optimization process was assessed with used cooking oil (UCO) from the food industry. The purified oil was tested for three quality parameters, the peroxide value, free fatty acid value and iodine value. The results of this testing showed that BPAC could slightly improve the quality of used cooking oil.

Keywords
Activated carbon, banana peel, used cooking oil, purification, quality

Open Peer Review

This article is included in the ICTROPS 2018 collection.
Corresponding author: Sulisty Prabowo (sulprab@gmail.com)

Author roles: Prabowo S: Conceptualization, Data Curation, Formal Analysis, Funding Acquisition, Investigation, Methodology, Supervision, Validation, Visualization, Writing – Original Draft Preparation, Writing – Review & Editing; Muflihah M: Conceptualization, Data Curation, Formal Analysis, Funding Acquisition, Investigation, Methodology, Project Administration, Validation, Writing – Original Draft Preparation

Competing interests: No competing interests were disclosed.

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Introduction
Frying food with cooking oils, such as palm oil, is a common cooking method. Cooking oil functions as a heat transfer medium, improves the physical appearance and texture of the food, provides savory flavors, and adds nutritional and calorific value to foodstuffs.

The need for cooking oil in Indonesia was estimated at 1.1 million tons and 3.5 million tons on particular days of high use, such as religious holidays. This situation led to the rising price of palm oil, forcing some producers of small-medium sized enterprises (SMEs) to use cooking oil repeatedly to save money.

The repeated use of cooking oil at high temperatures will change the physicochemical properties, damaging the oil, due to oxidation. A concise review of number of studies has been carried out, which has demonstrated the adverse effects of using UCO. Thus, UCO tends to be discarded since there is no further use for it. As a result, increased cooking oil consumption can become a major environmental issue. Based on this concern, this present research was conducted to assess the potential for UCO purification utilizing other unused materials, such as banana peels.

Several publications have described the re-use of cooking oil so it is not wasted. One method is adsorption using adsorbents. Common adsorbents include carbon compounds obtained from sources such as coconut shells, wood and coal, which have been pretreated, turning them into activated charcoal.

Taking into account the abundance of banana peel waste, this study attempted to produce activated charcoal derived from this resource. The aim was to determine the optimum temperature and time for the banana peel carbonation process to produce adsorbents, which could later be used to improve the quality of UCO.

Methods
Research sample
UCO samples were randomly taken from three SMEs foodstalls in Samarinda, Indonesia. All samples were then mixed together and tested in duplicate. As the control, we used the most widely oil brand used by them. Banana peels were collected from fried banana traders in the same location. Samples were collected on August 8, 2018, before the SME closed their stalls in the night.

Materials and equipment
The tools used in this study were an oven, furnace, sieve, burette, Erlenmeyer flask, stirring rod, pipette, measuring glass and hotplate. The materials used were distilled water, acetic acid, chloroform, KI, 0.01 M Na₂SO₃, starch, phenolphthalein, ethanol, 0.5 M KOH, 0.5 M HCl, iodine-bromide solution, and NaOH.

Optimization of temperature and time of carbonation
The dried banana peel was furnace for 3 hours at 200°C, 400°C or 600°C until the materials turned into charcoal. Subsequently, the temperature of the charcoal was allowed to cool, and the charcoal was immediately sieved with a 200-mesh-size sieve and then activated using 1 M NaOH solution at ambient temperature. The charcoal was stirred for 2 hours and then neutralized by washing with distilled water until the pH is neutral. The obtained charcoal was then dried in the oven at 80°C.

Sample preparation
Some impurities and foreign materials were initially removed in the precipitation and separation process. Samples were put in a glass tube (30 cm height, 5 cm diameter) and the impurities were left to settle at room temperature by gravitation. This process was performed without reducing the amount of free fatty acids in the UCO.

Refining process
In the UCO refining process, 10 g of BPAC was added to 200 mL UCO and stirred with magnetic stirrer at the room temperature. After 24 hours, the UCO was filtered out using a paper filter.

Oil quality testing
The color of the oil was analyzed using a scoring sensory test by 16 untrained panelists, using a scale of 1 (very dark brown), 2 (dark brown), 3 (rather dark brown), 4 (clear yellow) and 5 (very clear yellow, equivalent to fresh oil). Judgement of oil color was performed using UCO filtered with charcoal heated to 600°C for 3 hours. Peroxide value (PV), free fatty acid value (AV) and iodine value (IV) were analyzed using the official methods of analysis of the Association of Official Analytical Chemists (AOAC). PV, AV and IV were evaluated using charcoal heated at each temperature for each length of time.

PV was determined as follow. Approximately 5.0 g of UCO sample was placed into iodine flask and was dissolved in the solution mixture of 50 mL glacial acetic-acid isooctane (3: 2, v/v). The solution was added with 0.5 mL saturated solution of KI. The mixture was then shaken vigorously for 0.5 min and allowed in the dark condition for another 3 min. The solution was added with 30 mL distilled water and was titrated using sodium thiosulphate 0.01 N using 1 mL of starch indicator 0.05%. Titration was stopped if blue colour of solution just disappeared. The blank titration was also carried out under similar condition without addition of samples. PV was calculated as:

\[ PV = \frac{(Vs - Vb)}{g_{sample}} \times N_{thio} \times 1000 \]

where PV is peroxide value (in meq/kg), Vs is volume (in mL) of thiosulphate used for sample titration, Vb is volume (in mL) of thiosulphate used for blank titration, Nthio is normality of thiosulphate.

Acid value (AV) was determined using a titration method. A 10.0 g of samples were accurately weighed and dissolved in 100 mL ethanol-ethyl ether mixture (1:1 v/v). This solution was then titrated using standardized KOH-ethanolic solution using phenolphthalein as indicator until pink-violet color was observed. AV was expressed as the number of mg KOH needed to neutralize free fatty acids in 1 g of sample. AV was calculated as the volume of KOH needed per gram sample multiplied by N KOH and 56.1.
**Determination of iodine value.** A-1.0 g of samples was added with 20 mL cyclohexane-acetic acid mixture (1:1 v/v). The solution was added with 25 mL of Wijs solution (iodine monochloride, ICl) and was kept in the dark condition for 1 h. The mixture was added with 20 mL saturated KI solution and 150 mL distilled water, shaken homogeneously, and titrated with 0.1 N sodium thiosulphate 0.1 N using 1 mL of starch indicator 0.05% until the color became clear. The blank titration was also carried under similar condition without addition of FO samples. IV was calculated as:

$$ IV = \frac{(V_b - V_s) \times N_{thio} \times 12.69}{g \text{ sample}} $$

Vb is volume (in mL) of thiosulphate used for blank titration and Vs is volume (in mL) of thiosulphate used for sample titration.

**Results and discussion**

**Color of filtered vs. unfiltered cooking UCO**

Table 1 presents the results of the score observations of color. Initially, the color of fresh cooking oil is very clear yellow due to the presence of \( \alpha \) and \( \beta \)-carotene or xanthophyll. This color and clarity becomes darker and brown, respectively, after several uses for frying as a result of the degradation of natural dyes and oxidation. Some foodstuffs with high protein and sugar contents will initiate browning reactions as a result of Maillard and caramelization reactions. During the refining process, significant color changes occurred. Banana peel is able to absorb dark colors in UCO because of its lutein content.

**Chemical analysis**

**Figure analysis**

Figure 1 presents the results analysis on the quality of samples before and after the treatments using BPAC which were made from different temperatures and time. Increasing the temperature and time in the charcoal production process has a positive effect on the quality of cooking oil refinery. Higher temperatures and longer casting processes make the activated carbon more refined to make the material surface wider. Surface area is one factor that affects the rate of adsorption of dirt in used cooking oil.

**Free fatty acid content**

The level of free fatty acids is an important parameter when determining the quality of cooking oil since it could determine the age, purity and the level of oil hydrolysis. Free fatty acid content exceeding 0.30% of the oil mass result in an undesirable flavor. Based on the results of UCO sample analysis, the level of free fatty acids was quite high (1.23%). However, after soaking with BPAC the AV levels decreased to 0.71%, but this number still does not conform to Indonesia’s standard (0.3%) of oil quality. The high levels of free fatty acids in UCO indicate the degree of oil damage. In this research, soaking with BPAC gives a quite efficient effect in reducing about 40–50% damage.

**Saponification number**

In this study, the saponification number of UCO cannot be improved to the level of recommended quality standard. Instead, the value tends to decrease. It was also found that UCO has a higher molecular weight than fresh oil, meaning that it has bound many other foreign materials other than oil; so that it is no longer suitable for use.

**Peroxide values**

The peroxide compound concentration in oil indicates the level of cooking oil degradation due to oxidation in heating process.
The content of peroxide compounds in cooking oil can accelerate rancidity and drive the presence of undesired flavor. Interestingly, peroxide values significantly dropped during soaking with BPAC. Yet, the peroxide value still does not conform to the quality standard which is at maximum of 2 mEq/ kg. Immersion with BPAC reduced these values by about 50–60%.

Conclusion
Producing activated carbon from banana peels was found to have an impact in improving the quality of used cooking oil.

Data availability
Raw data for oil color assessments and effect of charcoal filtering on free fatty acids, saponification and peroxide values of used cooking oil are available on Zenodo. DOI: https://doi.org/10.5281/zenodo.1673940.

Data are available under the terms of the Creative Commons Attribution 4.0 International license (CC-BY 4.0).

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The funders had no role in study design, data collection and analysis, decision to publish, or preparation of the manuscript.

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Current Peer Review Status:  ?  ?

**Version 1**

Reviewer Report 06 June 2019

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**Abdul Rohman**

Faculty of Pharmacy, Gadjah Mada University, Yogyakarta, Indonesia

- The statistical analysis should be improved. How many replicates? Please add error bars for the results in Figure 1.
- Please, compare the results with those published regarding the quality of cooking oils from other frying oils.
- The Conclusion must be more elaborated.
- Please add newer references from primary sources. There are some papers published on this issue.

**Is the work clearly and accurately presented and does it cite the current literature?**

Yes

**Is the study design appropriate and is the work technically sound?**

Partly

**Are sufficient details of methods and analysis provided to allow replication by others?**

Yes

**If applicable, is the statistical analysis and its interpretation appropriate?**

Partly

**Are all the source data underlying the results available to ensure full reproducibility?**

Yes

**Are the conclusions drawn adequately supported by the results?**

Yes
**Competing Interests:** No competing interests were disclosed.

**Reviewer Expertise:** Fats and Oils analysis

I confirm that I have read this submission and believe that I have an appropriate level of expertise to confirm that it is of an acceptable scientific standard, however I have significant reservations, as outlined above.

Reviewer Report 04 March 2019

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**Nattapol Tangsuphoom**
Food Science Unit, Institute of Nutrition, Mahidol University, Salaya, Thailand

The manuscript entitled “Synthesis of adsorbent from food industry waste for purification of used cooking oil” demonstrates the application of activated carbon prepared from banana peels in improving the quality of used cooking oil. Although the experiment was reasonably designed and executed, there are some significant points that need to be improved. I thus recommend major revision on these following points before accepting for indexing:

**Abstract:**
- Line 8: Quantitative description of “slightly improve” should be given.

**Methods:**
- In general this part has a lack of statistical analysis. Means and standard deviations of triplicate samples should be reported and analyzed for the statistical difference. It would be more concrete if characterization of the BPAC is conducted such as bulk density, porosity and microscopic analysis for microstructure and compared with commercial activated carbon. Discussion of results should also be included. The quality of UOC after being treated with BPAC should be compared to the standard for cooking oil.

**Research sample:**
- What type of oils were collected (palm, coconut, etc.)? Which processing/cooking method were these UCO samples subjected to?

**Oil quality testing:**
- Why was only the UCO sample treated with BPAC sample prepared at 600°C for 3 hours tested for colour? What about the colour of other treated UCO samples?

**Results and discussion:**
- Table 1: The distribution of score should be presented.
- Figure 1: Data should be presented in the form of a Table.
- Saponification number: This measurement does not appear in the Methods part.

**Is the work clearly and accurately presented and does it cite the current literature?**
Yes

**Is the study design appropriate and is the work technically sound?**
Partly

**Are sufficient details of methods and analysis provided to allow replication by others?**
Yes

**If applicable, is the statistical analysis and its interpretation appropriate?**
No

**Are all the source data underlying the results available to ensure full reproducibility?**
Yes

**Are the conclusions drawn adequately supported by the results?**
Partly

**Competing Interests:** No competing interests were disclosed.

**Reviewer Expertise:** food lipids, food waste utilization and recovery, food material characterization

I confirm that I have read this submission and believe that I have an appropriate level of expertise to confirm that it is of an acceptable scientific standard, however I have significant reservations, as outlined above.

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Author Response 04 Apr 2019

**Sulistyo Prabowo,** Mulawarman University, Samarinda, Indonesia

Thank you very much for this great review to improve our research. This is ongoing research and we still working on it. We acknowledge many limitations in this short communication and will do the suggestion.

**Competing Interests:** No competing interests were disclosed.
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