Journal of
Food and Pharmaceutical Sciences

Original Article

The Effects of Activated Carbon and Bentonite on Physicochemical Characterization of Red Fruit Oil (*Pandanus conoideus* Lam)

Mustika Erlinaningrum[1,2], Abdul Rohman[3,4]*, and Agustina Ari Murti Budi Hastuti[3,4]

[1]Master in Pharmaceutical Sciences, Faculty of Pharmacy, Universitas Gadjah Mada, Jl. Sekip Utara, Sleman, Yogyakarta 55281, Indonesia; mustikaerlinaningrum@mail.ugm.ac.id
[2]Indonesian Food and Drug Authority, District of Manokwari, Papua Barat 98312, Indonesia; mustikaerlinaningrum@mail.ugm.ac.id
[3]Center of Excellence Institute for Halal Industry & Systems, Universitas Gadjah Mada, Yogyakarta 55281, Indonesia; abdulkimfar@gmail.com
[4]Department of Pharmaceutical Chemistry, Faculty of Pharmacy, Universitas Gadjah Mada, Yogyakarta 55281, Indonesia; agustina.ari.m.b.h@mail.ugm.ac.id
*Corresponding author: Abdul Rohman | Email: abdulkimfar@gmail.com

Received: 18 March 2024; Revised: 20 May 2024; Accepted: 21 May 2024; Published: 23 August 2024

Abstract: This research aims to see the quality of red fruit oil (RFO) before and after being treated with activated carbon and bentonite. The use of activated carbon and bentonite as adsorbents is because they are relatively cheap and widely available. The quality of red fruit oil from wet extraction can be seen from the analysis of physicochemical characterization determination, which includes the determination of peroxide value (PV), acid value (AV), saponification value (SV), p-anisidine value (PAV), and iodine value (IV). In this study, AV RFO ranged from 9.22 – 10.23 mg KOH/g, PV RFO ranged from 5.57 – 7.56 meq/kg, SV RFO ranged from 96.57 – 110.24 mgKOH/g, IV RFO ranged from 70.00 – 70.83 $gI_2/100g$, and PAV RFO ranged from 5.97 – 7.00 meq/kg. The purification of RFO using activated carbon and bentonite has a significant effect ($p < 0.05$) affected the physicochemical characteristics of the oil, where the process succeeded in improving the quality of RFO by increasing the iodine value, but reducing the peroxide value, acid value, saponification value, and p-anisidine value.

Keywords: Activated carbon, Adsorbent, Bentonite, Red fruit, Wet extraction

1. INTRODUCTION

Papua is one of the island that has red fruit (*Pandanus conoideus* Lam)[1]. Red fruit contains many active components such as tocopherols, carotene, cryptoxanthin, oleic acid, and palmitate acid[2], [3], [4], [5]. The Papuan people widely utilize red fruit as a source of edible oil and as an antioxidant[2], [3]. Traditionally, Papuans use a wet extraction method to produce red fruit oil. The wet extraction method requires adding a certain amount of water during the extraction process[6].

Free fatty acids (FFA) may be present in extracted red fruit oil (RFO), lowering its quality. The quality of RFO can be seen by looking at its characterization, namely by determining peroxide value (PV), acid value (AV), iodine value (IV), saponification value (SV), and p-anisidine value (PAV). The PV indicates the degree of oil deterioration. The amount of FFA in the oil is indicated by the AV. The IV indicates the unsaturation of the fatty acids that make up the fat. The SV indicates the molecular weight of the oil. The anisidine value shows the number of secondary oxidation products in the oil[6], [7]. To improve the quality of RFO, RFO needs to be purified by adding adsorbents that aim to absorb impurities in oil such as pigments, components, and FFA. Bentonite, attalpugite, chitosan, and activated carbon are examples of adsorbent used in oil refining. [8]. In this study, RFO purification was carried out using bentonite and activated carbon as adsorbents because they are relatively cheap and easily found in everyday life. Previous studies reported that the use of the bentonite and activated carbon can improve the quality of hazelnut oil and used cooking oil [9], [10]. This study aims to see the quality of RFO before and after being given bentonite and activated carbon adsorbents. The addition of bentonite and activated carbon will likely improve the quality of RFO.

2. MATERIALS AND METHODS

2.1. Materials

Reagents used were ethanol p.a, chloroform, Wijs reagent, glacial acetic acid (Supelco, Germany), sodium thiosulfate, hydrochloric acid (HCl), anhydrous sodium sulfate (Na_2SO_4), potassium hydroxide (KOH), potassium iodide (KI) (Merck, Germany), and distilled water. The adsorbents used were activated carbon and bentonite. The red fruit used is collected from Papua, Indonesia.

2.1.1. Preparation of RFO

Oil from red fruits was prepared using the wet extraction method. Red fruits were peeled to separate the drupes from the pedicels using a knife and mixed with water (1:2 by weight/volume). The mixture was then heated for 20 minutes at 103°C and filtered. The paste obtained was then reheated for 60 minutes at 103°C. Next, centrifuge the paste for 10 minutes at 3000 rpm to separate the RFO from water and solid residue[11], [12]. Next, the oil was put into a centrifuge tube and anhydrous sodium sulfate (Na_2SO_4) was added as much as 10% of the sample weight, then homogenized with a vortex for 5 minutes and continued with centrifugation for 15 minutes at 5000 rpm. The precipitate and oil were separated and the oil was put into a new flask. After that, add the adsorbent previously activated by heating 250°C for 1 hour as much as 2% of the weight of the oil. Furthermore, the oil was stirred for 30 minutes at 500 rpm while heated at 40°C to improve adsorption kinetics. The oil was separated from the sediment and put into a dark-bottle[13].

2.2. Characterization of RFO

2.2.1. Determination of PV, AV, IV, and SV

Determination of PV, AV, IV, and SV in RFO according to the standard methods for the analysis of fats and oils in the official analytical methods of AOAC International[14].

2.2.2. Determination of PAV

Determination of PAV in RFO samples using IUPAC standard method (1987) using UV-Vis spectrophotometer[15].

2.3. Statistical analysis

One-way ANOVA from Minitab (version 21) software was used to examine the RFO characterization results, with a significance level of $p < 0.05$. The Tukey test is used to discover significant differences in the analysis if the data from the results show a significant difference.

3. RESULTS AND DISCUSSION

Red fruit extracted using the wet extraction method to produce oil is added with anhydrous sodium sulfate (Na_2SO_4) to reduce the water content in the oil extract because water can affect the quality and stability of oil[16]. RFO is then purified by adding adsorbents. The utilized adsorbents consists of activated carbon and bentonite. Bentonite is a clay containing the main mineral montmorillonite and is used as a vegetable oil bleach[17]. Activated carbon is charcoal that is activated to have a high absorption of odor, color, toxic substances, and other chemical substances used for food oil purification[18]. Before being added, bentonite and activated carbon are activated to enlarge the pores so that the surface increases size and affects adsorption[19]. The appearance of red fruit oil before and after the addition of bentonite and activated carbon is visually not much different (Figure 1).

Figure 1. Visual appearance of crude RFO (a), RFO applied to activated carbon treatment (b), and RFO applied to bentonite treatment (c).

All RFOs were characterized to determine the peroxide value (PV), acid value (AV), iodine value (IV), saponification value (SV), and p-anisidine value (PAV). The RFO characterization results can be seen in Table 1. These charaterization parameters provide an overview ogf the quality and grade of the red fruit oil. Unfortunately, there is no Indonesia National Standard (SNI) for red fruit oil to date.

Table 1. Peroxide value, acid value, iodine value, saponification value and p-anisidine value of crude RFO and RFO purified with activated carbon and bentonite

Parameters	Crude RFO	RFO treated with activated carbon	RFO treated with bentonite	p-Value
Acid value (mgKOH/g)	10.23 ± 0.03^a	9.73 ± 0.03^b	9.22 ± 0.03^c	0.000
Peroxide value (meq/kg)	$7.56 \pm 0,00^a$	6.57 ± 0.00^b	5.57 ± 0.00^c	0.000
Saponification value (mgKOH/g)	$110.24 \pm 0,78^a$	103.89 ± 0.79^b	96.57 ± 0.83^c	0.000
Iodine value (g I_2/100g)	70.00 ± 0.22^b	70.40 ± 0.25^{ab}	70.83 ± 0.22^a	0.013
p-anisidine value (meq/kg)	7.00 ± 0.04^a	6.56 ± 0.01^b	5.97 ± 0.01^c	0.000

* Significant differences ($p < 0.05$) are indicated by the presence of different letters in the same row.

The data in Table 1 shows that RFO refining using activated carbon and bentonite has a significant effect ($p < 0.05$) on AV, PV, IV, SV, and PAV. The refining process using bentonite and activated carbon can improve the physicochemical characteristics of oil by adsorbing oxidation products including peroxides, ketones, FFA, aldehydes, and phosphatides in oil [20].

The application of activated carbon and bentonite to RFO has successfully reduced AV, PV, SV, PAV, and increased IV. The AV is a measure of the quantify of FFA in the oil that are the result of the hydrolysis process of the oil during the extraction and storage process[6], [7], [21]. In this study, the AV of crude RFO was 1023 ± 0.03 mgKOH/g, RFO treated with activated carbon was 9.73 ± 0.03 mgKOH/g, and RFO treated with bentonite was 9.22 ± 0.03 mgKOH/g. The high acid value of RFO is due to the hydrolysis reaction in the extraction process, which uses water and high temperature heating, thus increasing the oil's free fatty acid content [22]. The AV of RFO with the addition of bentonite has better quality. This is because the ability of bentonite to absorb free fatty acids is more remarkable due to its small particle size. The smaller the particle size, the greater the diffusion rate of solute molecules into the pores of the adsorbent[8]. In addition, the ability of bentonite to absorb free fatty acid components in oil is due to the silanol group (Si-OH) formed in the SiO_2 compound in bentonit during activation. Silanol groups are polar and able to bind to other polar compounds, such as free fatty acids. Hydrogen atoms from silanol groups on bentonite bind to oxygen-carbonyl groups (-C=O) on free fatty acids through hydrogen bonds so that free fatty acids are bound to the bentonite surface and can reduce the acid value in oil. This bonding occurs because hydrogen atoms tend to bond with oxygen atoms that are more electronegative [10], [23]. Peroxide value indicates the degree of oil damage[6], [7]. In this study, the PV of crude RFO was 7.56 ± 0.00 meq/kg, RFO treated with activated carbon was 6.57 ± 0.00 meq/kg, and RFO treated with bentonite was 5.57 ± 0.00 meq/kg. RFO PV with the addition of bentonite has better quality. This is because bentonite formed silanol groups (Si-OH) contained in the SiO_2 compound during activation. Silanol groups have polar properties and can bind to other compounds with specific functional groups, such as peroxide groups in peroxide compounds formed in oxidized oil. The silanol group on bentonite binds to the peroxide group in the peroxide compound through hydrogen bonds, which causes the peroxide molecule to bond to the bentonite surface so that the oxydative stability of the oil increases and the PV of the oil is reduced as the amount of peroxide in the oil decreases. This hydrogen bond occurs between the silanol group's hydrogen atom and the peroxide group's oxygen atom[10], [23].

The SV indicates the molecular weight of the oil[6], [7]. In this study, the SV of crude RFO was 110.24 ± 0.78 mgKOH/g, RFO treated with activated carbon 103.89 ± 0.79 mgKOH/g, and RFO treated with bentonite 96.57 ± 0.83 mgKOH/g. The saponification value of RFO in this study is in accordance with the report of Murtiningrum (2005), which ranged from 88.83 – 212.59 mgKOH/g. The molecular weight of fatty acids in the oil decreases as the SV increases. The addition of bentonite and activated carbon to RFO can reduce saponification value because bentonite and activated carbon adsorb impurities such as FFA and short-chain triglycerides in oil[24], [25], [26]. The unsaturation of the fatty acids that comprise up the fat is indicated by the IV[6], [7]. In this study, the IV of crude RFO was 70.00 ± 0.22 gI_2/100g, RFO treated with activated carbon 70.40 ± 0.25 gI^2/100g, and RFO treated with bentonite 70.83 ± 0.22 gI_2/100g. The provision of activated carbon and bentonite can increase the IV in RFO by absorbing unwanted components, such as saturated compounds so that the concentration of double bonds in the oil increases[27]. As the IV increases, the oil's degree of unsaturation increases, and the quality improves[13]. The PAV indicates the number of secondary oxidation products in the oil[6], [7]. In this study, the PAV of crude RFO was

7.00 ± 0.04 meq/kg, RFO treated with activated carbon was 6.56 ± 0.01 meq/kg, and RFO treated with bentonite was 5.97 ± 0.01 meq/kg. Activated carbon and bentonite can inhibit the formation of secondary oxidation products to reduce the PAV [28].

4. CONCLUSION

The purification of red fruit oil using activated carbon and bentonite significantly ($p < 0.05$) affected the physicochemical characteristics of the oil, such as PV, AV, SV, IV, and PAV. The process successfully improved the quality of RFO by decreasing acid, peroxide, saponification, and p-anisidine and increasing iodine value. Providing activated carbon and bentonite can be an alternative to improve RFO quality effectively and economically.

Funding: The research was funded by the Indonesian Food and Drug Authority (Badan POM) Foundation of The Republic of Indonesia through a Badan POM Scholarships 2022/2024.

Acknowledgments: M.E. is grateful to the Indonesian Food and Drug Authority (Badan POM) Foundation of The Republic of Indonesia for a Master's Studentship through the Badan POM Scholarships 2022/2024.

Conflicts of interest: The authors declare no conflict of interest.

References

[1] E. B. Walujo, A. P. Keim, and M. J. S., "Kajian etnotaksonomi Pandanus conoideus Lamarck untuk menjembatani pengetahuan lokal dan ilmiah," *Berita Biologi*, vol. 8, no. 5, pp. 391–404, Agustus 2007.

[2] A. Rohman, S. Riyanto, N. Yuniarti, W. R. Saputra, R. Utami, and W. Mulatsih, "Antioxidant activity, total phenolic, and total flavaonoid of extracts and fractions of red fruit (Pandanus conoideus Lam)," *International Food Research Journal*, vol. 17, pp. 97–106, 2010.

[3] Z. L. Sarungallo, P. Hariyadi, N. Andarwulan, and E. H. Purnomo, "Characterization of Chemical Properties, Lipid Profile, Total Phenol and Tocopherol Content of Oils Extracted from Nine Clones of Red Fruit (Pandanus conoideus)," *Kasetsart J. (Nat. Sci.)*, vol. 49, pp. 237–250, 2015.

[4] E. Fitria, P. Hariyadi, and H. Wijaya, "Identifikasi dan fraksinasi karotenoid pada minyak buah merah (Pandanus conoideus)," *Journal of Agro-based Industry*, vol. 37, no. 1, pp. 7–19, Mei 2020.

[5] A. W. A. Renyaan, E. Suprayitno, Aulani'am, and A. M. Hariati, "Nutrient content and endemic red fruit oil (Pandanus austrosinensis) fatty acid profile in Papua Indonesia," *J. Phys.: Conf. Ser.*, vol. 1665, no. 1, p. 012019, Oct. 2020, doi: 10.1088/1742-6596/1665/1/012019.

[6] H. Mulyani and A. Sujarwanta, *Lemak dan minyak*, 1st ed. Kota Metro: Lembaga Penelitian UM Metro, 2018.

[7] A. Rohman, S. Riyanto, A. Windarsih, Irnawati, Khadijah, and A. R. Putri, *Karakterisasi minyak dan lemak*, 1st ed. Indonesia: Pustaka Pelajar, 2020.

[8] R. T. Rosmalina, W. Kosasih, S. Priatni, and Research Unit for Clean Technology, Indonesian Institute of Sciences (LIPI), Bandung, "The effects of adsorbent materials on the lipid quality of Lemuru fish oil and the enrichment of omega-3 using lipase," *jtip*, vol. 32, no. 1, pp. 16–26, Jun. 2021, doi: 10.6066/jtip.2021.32.1.16.

[9] S. Darmawan, "Pembuatan minyak kemiri dan pemurniannya dengan arang aktif dan bentonit," *Jurnal Penelitian Hasil Hutan*, vol. 24, no. 5, pp. 413–423, Oktober 2006.

[10] J. P. Hidayat, A. Hariyadi, and F. Chosta, “Unjuk kinerja adsorpsi bentonit dan arang aktif terhadap karakteristik minyak jelantah,” *Jurnal Sains dan Teknologi Pangan*, vol. 7, no. 6, pp. 5600–5614, 2022.

[11] Z. L. Sarungallo, P. Hariyadi, A. Andarwulan, and E. H. Purnomo, “Effect of heat treatment prior to extraction on the yield and quality of red fruit (Pandanus conoideus) oil,” *Food Res.*, vol. 4, no. 3, pp. 659–665, Jan. 2020, doi: 10.26656/fr.2017.4(3).281.

[12] E. P. Susanti, A. Rohman, and W. Setyaningsih, “Dual response optimization of ultrasound-assisted oil extraction from red fruit (Pandanus conoideus): recovery and total phenolic compounds,” *Agronomy*, vol. 12, no. 2, p. 523, Feb. 2022, doi: 10.3390/agronomy12020523.

[13] Indah, A. Rohman, and L. A. Lestari, “Physicochemical characterization refined patin fish oil (Pangasius micronea) using bentonite and activated Carbon,” *Journal of Food and Pharmaceutical Sciences*, vol. 10, no. 2, pp. 626–633, 2022.

[14] AOAC, *Official methods of analysis of AOAC International*. 2000.

[15] IUPAC, *Standard Methods for the Analysis of Oils. Fats and Derivatives*, 7th ed. International Union of Pure and Applied Chemistry Blackwell Scientific Publications, 1987.

[16] H. Soetjipto, Y. A. Putra, and A. I. Kristijanto, “Pengaruh Pemurnian Terhadap Kualitas dan Kandungan Skualen Minyak Biji Kemangi Hutan (Ocimum gratissimum L.),” *ALCHEMY J.Pen.Kim*, vol. 16, no. 2, pp. 190–198, Sep. 2020, doi: 10.20961/alchemy.16.2.41110.190-198.

[17] Badan Standardisasi Nasional, “Bentonite untuk pemucatan minyak nabati SNI 13-6336-2000.” Badan Standardisasi Nasional (BSN), 2000.

[18] Badan Standardisasi Nasional, “Arang aktif untuk pemurnian minyak makan SNI 06-4262-1996.” Badan Standardisasi Nasional (BSN), 1996.

[19] L. E. Laos, M. Masturi, and I. Yulianti, “Pengaruh Suhu Aktivasi Terhadap Daya Serap Karbon AKtif Kulit Kemiri,” in *Prosiding Seminar Nasional Fisika (E-Journal) SNF2016 UNJ*, Pendidikan Fisika dan Fisika FMIPA UNJ, 2016, pp. SNF2016-MPS-135-SNF2016-MPS-140. doi: 10.21009/0305020226.

[20] D. F. Ayu, A. Diharmi, and A. Ali, “Karakteristik minyak ikan dari lemak abdomen hasil samping pengasapan ikan patin (Pangasius hypophthalmus),” *Jurnal Pengolahan Hasil Perikanan Indonesia*, vol. 22, no. 1, pp. 187–197, 2019.

[21] Z. L. Sarungallo, P. Hariyadi, N. Andarwulan, and E. H. Purnomo, “Pengaruh metode ekstraksi terhadap mutu kimia dan komposisi asam lemak minyak buah merah (Pandanus conoideus),” *Jurnal Teknologi Industri Pertanian*, vol. 24, no. 3, pp. 209–217, 2014.

[22] Z. Khoirunnisa, A. S. Wardana, and R. Rauf, “Angka Asam dan Peroksida Minyak jelantah dari penggorengan Lele secara berulang,” *JK*, vol. 12, no. 2, pp. 81–90, 2019, doi: 10.23917/jk.v12i2.9764.

[23] L. H. Rahayu and S. Purnavita, “Pengaruh suhu dan waktu adsorpsi terhadap sifat kimia-fisika minyak goreng bekas hasil pemurnian menggunakan adsorben ampas pati aren dan bentonit,” *Momentum*, vol. 10, no. 2, pp. 35–41, 2014.

[24] N. P. Kurnianingsih, M. Maherawati, and T. Rahayuni, “Pemurnian Minyak Kelapa Tradisional Menggunakan Adsorben Arang aktif Tempurung Kelapa,” *AGS*, vol. 4, no. 1, p. 37, Jun. 2020, doi: 10.32585/ags.v4i1.848.

[25] D. A. Putra, M. Meriatna, S. Suryati, and Z. Zulmiardi, “Pembuatan Zat Emulsifier dari Minyak Pliek U dengan Katalis NaOH,” *j. Teknologi Kimia Unimal*, vol. 11, no. 1, p. 22, May 2022, doi: 10.29103/jtku.v11i1.7246.

[26] Murtiningrum, S. Ketaren, Suprihatin, and Kaseno, “Ekstraksi minyak dengan metode wet rendering dari buah pandan,” *Jurnal Teknologi Industri Pertanian*, vol. 15, no. 1, pp. 28–33, 2005.

[27] Z. L. Sarungallo, Murtiningrum, and S. N. P. Paiki, "Sifat Fisikokimia Minyak Kasar dan Hasil Degumming dari Buah Merah (Pandanus conoideus L.) yang diekstrak secara Tradisional Merdey," *Jurnal Agrotek*, vol. 1, no. 6, pp. 9–15, 2011.

[28] L. Sembiring, M. Ilza, and A. Diharmi, "Characteristics of Pure Oils from Belly Fat (Pangasius hypophthalmus) with Bentonite Purification," *Jurnal PHPI*, vol. 21, no. 3, p. 549, Dec. 2018, doi: 10.17844/jphpi.v21i3.24742.