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and development note

1**Potential application of waste neem leaves for bleaching of low-quality crude palm oil**

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INTRODUCTION Conversion of crude palm oil to refined edible oil involves removal of the products of hydrolysis and oxidation, the color and the flavor. The refining routes of palm oil are quite identical. There are two routes are taken to process crude oil into refined oil, which are chemical (basic) refining and physical refining. The methods differ basically in the way the fatty acids are removed from the oil. In practice, palm oil bleaching is accomplished by heating the oil in the presence of bleaching agent at certain time until the desired color is reached. The improvement in color is as a result of

7**the removal of organic compounds such as β -carotene,**

xantho- phylls, chlorophyll, etc.[1] Even the acid-activated clays (bleaching earth)

7are efficient and effective adsorbent for bleaching

of crude edible oils,[2 – 4] some attention had been directed to the use of renewable resources such as agro waste materials.[1,5] In this paper, an alternative adsorbent, neem (Azadirachta indica) leaves powder, was used for bleaching of low-quality crude palm oil. The advan- tages of using neem leaves powder for bleaching purpose are environmental friendly and have similar adsorption capacity to activated bleaching earth. In order to enhance the adsorption capacity of the neem leaves powder, the surface modification using acid was conducted. Adsorption mechanism of β -carotene onto active functional group surface of neem leave powder was also proposed. *Correspondence to: Suryadi Ismadji,

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E-mail: suryadiismadji@yahoo.com Curtin University is a trademark of Curtin University of Technology MATERIALS AND METHODS Materials Neem leaves were obtained from Probolinggo, East Java, Indonesia. The neem leaves were repeatedly washed with distilled water in order to remove dust and soluble impurities, and subsequently dried at 60 ◦C under vacuum condition until its moisture content was around 10%. Neem leaves were pulverized using grinder MX-T1106N. After crushing, neem leaves pow- der was shieved using wire mesh screen ASTM, TEST SIEVE, Retsch 5657 HAAN, West Germany to obtain neem leaves powder with particle sizes ranging from 80 to 200 mesh. Characterization of neem leaves powder The surface functional groups available in the sur- face of neem leaves powder were characterized using Fourier transform infrared (FTIR) spectrometer (Shi- madzu 8400s). A qualitative analysis of neem leaves powder was conducted by obtaining FTIR transmis- sion spectra of neem leaves samples by KBr tech- nique. The technique was conducted by placing the KBr powder ground with an agate mortar in the sam- ple cup, and then the powder surface was evened using the attached sample pressing bar. Next, the powder was mounted to the instrument to make a background measurement. After that, the neem leaves powder sample was diluted with the KBr powder with the ratio of 10% and ground with the agate mor- tar until it becomes fine particles to mix both the kinds. Then, the mixed powder was placed in the sample cup and the powder surface was also evened using the sample pressing bar. Finally, the mixed 686 A. ADRIANI LIMAN et al. powder was mounted to the instrument to make a sam- ple measurement in the transmittance %T-mode. Delignification of neem leaves powder Neem leaves powder was soaked in 6 M of sodium hydroxide solution for 24 h. The neem leaves then sep- arated from the solution by centrifugation (Web MLW Medizintechnik Typ T51.1) at 3500 rpm (≈2900 g) for 20 min. Subsequently, neem leaves powder was washed with distilled water until the pH of the solution was con- stant and dried at 60 ◦C under vacuum condition until its moisture content was around 10%. Surface modification of neem leaves powder Neem leaves powder was soaked with hydrochloric acid (37%) for 1 h with impregnation ratio 1 : 0.5; 1 : 1; Asia-Pacific Journal of Chemical Engineering 1 : 1.5; 1 : 2; 1 : 2.5 (w/w). After soaking, neem leaves powder was repeatedly washed with distilled water until the pH of washing solution became constant and subsequently dried at 60 ◦C under vacuum condition for 24 h.

11Bleaching of crude palm oil Bleaching of crude palm oil process was conducted using treated and untreated neem leaves powder. The amount of adsorbent used in the bleaching process was 3% (w/v) of degummed palm oil. Degumming of crude palm oil was conducted before bleach- ing process using 0.2% of 60% phosphoric acid at 90◦C for 30 min. Bleaching process was conducted on degummed palm oil at 110–120◦C for 30 min and followed by vacuum filtration to separate oil and adsorbent. Table 1. R (color removal), FFA (decrease of free fatty acid content), PV (peroxide value) on degummed and bleached palm oil. No. Adsorbent Removal of color (%) FFA (%) PV (meq peroxide/kg oil) 1. Untreated neem leaves (CPO without degumming process) 2. Untreated neem leaves (CPO with degumming process) Without delignification 3. HCl 1 : 0.5 4. HCl 1 : 1 5. HCl 1 : 1.5 6. HCl 1 : 2 7. HCl 1 : 2.5 With delignification 8. HCl 1 : 0.5 9. HCl 1 : 1 10. HCl 1 : 1.5 11. HCl 1 : 2 12. HCl 1 : 2.5 – 5.23 4 6.78 9 5.62 22 5.46 37 5.69 54 5.82 71 5.95 10 5.42 23 5.56 41 5.72 57 5.85 76 5.92 3.2 1.4 0.91 0.82 0.78 0.51 0.35 0.88 0.75 0.62 0.47 0.31 CPO, crude palm oil. Table 2. FTIR spectra evaluation of the adsorbent. Wave number (cm−1) Neem leaves Functional group powder Delignified neem Delignified leaves powder after neem leaves treatment with HCl powder (before adsorption) Delignified neem leaves powder after treatment with HCl (after adsorption) O–H stretching (3590–3650

9**cm−1) C–H stretching (2853–2962 cm−1) C–**

N (2200–2420 cm−1) C–C (1620–1680, 2100–2290 cm−1) C O (1680–1740 cm−1) C C (1620–1680 cm−1) Secondary amides (1510–1550 cm−1) 3643 3649.4 2928 2928.3 2855.5 2854.8 2359.2 2366.9 – – 1681.6 1688.6 1673.9 1666.9 1521.3 1513.6 3606.6 2935.3 2855.5 2330.5 – 1732.7 1652.2 – 3613.6 2943 2870.2 2359.2 1622.8 2155.5 1732.7 1622.8 – Asia-Pacific Journal of Chemical Engineering Oil analyses Total color, %

8**free fatty acid (% FFA)** and **peroxide value (PV)**

were analyzed on degummed

8**palm oil and bleached palm oil.**

The analysis of FFA and PV were conducted according to AOAC Official Method 940.28 and 965.33. Total color of palm oil was analyzed using lovibond tintometer and % removal of color (% R) was calculated using Krishnan equation[6] as follow: Total color of (degummed palm oil %R = −bleached palm oil) × 100% Total color of degummed palm oil RESULTS AND DISCUSSION All adsorbents (Table 1) reduced the color of degummed crude palm oil. The highest color removal was observed with neem leaves powder treated with hydrochloric acid at impregnation ratio 1 : 2.5 (w/w) with delignification process. The FTIR spectral evaluation of the adsorbent is summarized in Table 2. In general, the delignification process provided little effect on the functional groups of neem leaves powder as indicated in Table 2. The shifting of the wave numbers of the functional groups O – H stretching, C – H stretching, C – N, C O, C C, and secondary amides due to the breakdown of lignin structure on the neem leaves. After treatment with HCl the functional group of secondary amides (present in the chlorophyll of neem leaves) become disappear. The breakdown of the chlorophil structure also enhanced the Figure 1. FTIR spectra of neem leaves powder treated with hydrochloric acid at impregnation ratio 1 : 2.5 with delignification process.

1**NEEM LEAVES FOR BLEACHING OF CRUDE PALM OIL**

687 Figure 2. (a) Coordination bonds between hydrogen ion and Lewis site. (b) Adsorption β-carotene on active functional group on neem leaves surface.

2**This figure is available in colour online at www.apjChemEng.com.**

O – H stretching, C O, and C C functional groups indicated by the increase of its intensity (not shown) and shifting of the wave numbers. FTIR spectra of neem leaves powder treated with hydrochloric acid at impregnation ratio of 1 : 2.5 (w/w) with delignification process (before and after bleaching process) are illustrated in Fig. 1. From Fig. 1, it can be seen that the functional groups involve in the adsorption 688 A. ADRIANI LIMAN et al. Figure 3. Adsorption secondary oxidation products on active functional group on neem leaves surface. This figure is available in colour online at www.apjChemEng.com. carotene were O–H stretching (3590–3650 cm−1), C C (1620–1680 cm−1), and C–C (1620–1680, 2100–2290 cm−1). During the impregnation process with HCl, the Lewis sites were created in the sur- face of delignified neem leaves powder. The presence of Lewis sites in the surface of neem leaves pow- der will attract the hydrogen ions from β-carotene to create the coordination bonds between the Lewis sites and the hydrogen ions, leading to the forma- tion of carbonium ions as indicated in Fig. 2a. Sub- sequently, the reaction between carbomium ions with the functional group C C occurred and functional group C–C created during the process as depicted in Fig. 2b. This result is consistent with the FTIR spectra as illustrated in Fig. 1. Here, the intensities of C C decrease and O – H starching and C – C increase after adsorption.

2**Asia-Pacific Journal of Chemical Engineering** The reason for **the**

enhanced carotene adsorption with the increase in the impregnation ratio of HCl due to the increase of Lewis site is therefore that the interaction coordination bonds between the Lewis site and β -carotene also increase. In general, the surface modification of neem leaves using HCl yielded no significant effect on FFA removal during the bleaching process (Table 1). However, a significant decreased in PV value was observed. The decrease of the PV value during the bleaching process was due to the adsorption of peroxide compounds and transformation of peroxide into secondary oxidation products and followed by the adsorption of secondary oxidation products[7] on the surface functional groups of the neem leaves as shown in Fig. 3. CONCLUSION Neem leaves can be used as an alternative adsorbent for color removal of low-quality palm oil. Surface modification of neem leaves powder using hydrochloric acid enhanced the β - carotene removal. REFERENCES [1] C. Agatemor. Food Sci. Technol. Res., 2008; 14, 301 – 305. [2] O.O. James, M.A. Mesubi, F.A. Adekola, E.O. Odebunmi, J.I.D. Adekeye,R.B. Bale. Lat. Am. Appl. Res., 2008; 38, 45 – 49. [3] B.J. Nde-Aga, R. Kamga, J.P. Nguetnkam. J. Appl. Sci., 2007; 7, 2462 – 2467. [4] W. Djoufac, R. Kamga, F. Figueras,D. Njopwouo. Appl. Clay Sci., 2007; 37, 149 – 156. [5] K.Y. Liew, A.H. Yee, M.R. Nordin. J. Am. Oil Chem. Soc., 1993; 70, 539 – 541. [6] E. Srasra, F. Bergaya, H.V. Damme, N.K. Ariguib. Appl. Clay Sci., 1989; 4, 411 – 421. [7] M. Rossi, M. Gianazza, C. Alamprese, F. Stanga. Food Chem., 2003; 82, 291 – 296.

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