

Improving the Quality of Patchouli Oil Using Biomass Adsorbent

Bastian Arifin^{*1}, Rosnani Nasution², Sofyana¹

¹Department of Chemical Engineering, Syiah Kuala University, Banda Aceh 23111, Indonesia

²Department of Chemistry, Faculty of Mathematic and Natural Sciences, Syiah Kuala University, Banda Aceh 23111, Indonesia

Abstract : Patchouli oil were contacted with an adsorbent dregs of coffee powder and fly ash have better quality than the initial quality of patchouli oil (before adsorbed). Among the two adsorbents, the dregs of the coffee powder is better than the fly ash to absorb iron, and absorbing the smaller components in patchouli oil. The content of iron before absorption was 75.936 ppm, and the iron is absorbed by the dregs of the coffee powder active as much as 34.306 ppm or as much as 44.77%, while the fly ash can absorb iron (Fe), which is 20.854 ppm or 26.33%. Adsorbent most effective in increasing the percentage of patchouli alcohol, or absorb other components, so amend the composition of patchouli oil is the fly ash, which led to the composition of patchouli alcohol changed and raise the percentage of patchouli alcohol became 48.71% from, 37.76%, rise up to 10.95%, while dregs of coffee powder led to change the composition of patchouli alcohol and raise the percentage of patchouli alcohol to 45.98% from, 37.76%, rise up to 4.56%. These observations indicate that the stirring of patchouli oil with an adsorbent for 15 minutes, the amount of adsorbent 1 gram/50 mL patchouli oil, showed that the specific gravity and refractive index that meets national standards of Indonesia (SNI). The longer the stirring time, increasing the amount of adsorbent, causing increases the specific gravity and refractive index of patchouli oil. Patchouli oil that has been absorbed by the dregs of coffee powder and fly ash can be completely soluble in alcohol 90%, with a ratio of 1 ml of patchouli oil with 10 ml of 90% ethanol. Transmittance of beginning of patchouli oil obtained by distillation using drums is 2.3682%, after absorption increased to 50%, the amount of adsorbent much as 4 g/50 mL patchouli oil, and a contact time of 45 minutes.

Keywords : patchouli oil, patchouli alcohol, adsorption, The Indonesian National Standard (SNI).

Introduction

Patchouli oil quality is influenced by the content of patchouli alcohol. The content of patchouli alcohol in patchouli oil, in international trade, minimum required is 30%. However, the content of patchouli alcohol in patchouli oil produced from farmers patchouli, in Aceh, Indonesia, in general, is still less than 30% (around 28%). In addition, the quality of patchouli oil is also influenced by metal content of Fe, patchouli oil of Aceh still contain high levels of iron (Fe), which is derived from the distillation drum [1].

In the research, that has been done, to reduce levels of iron (Fe) primarily is to brighten the dark color that can improve the quality of patchouli oil using adsorption method, one of the adsorbent used to overcome

this problem is to use activated charcoal from dregs of coffee powder. The advantage of using this adsorbent as an absorber is able to be made from waste of coffee powder residue, so it will be able to minimize the costs to be incurred.

Waste dregs of coffee powder can adsorb dye, known of its ability to absorb methylene blue (MB) in certain circumstances [2], as well as coffee grounds are bound to clay can absorb heavy metal ions from solution [3], while Namane [4], research on coffee grounds that are enabled to absorb the acid and phenol.

Waste coffee grounds are activated by heating the adsorbent which has been researched and are able to act as activated carbon that can remove dye [2]) and reduce the heavy metals [5,6]. In addition to coffee powder waste, fly ash is used as well as adsorbents.

Waste from palm oil mill, issued in the form of boiler ash (fly ash and bottom ash). According Viraraghavan [7], the ash obtained from the combustion of organic materials can be used as adsorbent for dyes contained in the solution.

One of the utilization of fly ash is used as an adsorbent to remove heavy metal compounds in water / wastewater. According to Sharma, as stated by Viraraghavan [7], coal ash is able to act as an adsorbent alternative to some of the heavy metals contained in water. Adsorption of Fe^{2+} compounds using fly ash which is filled into the column as high as 5 cm, a greater percentage reduction in the Fe ions ranging between 24% and up to 89% untuk Cr. .

Patchouli oil is an oil produced by distillation of leaves of patchouli (*Pogostemon cablin* Benth). Patchouli included in the Labiatae family, originally from India, Malaysia and Indonesia, and became famous for its fragrant aroma and distinctive, so that many substances used as perfume and fragrance fixative agent (binder), [8. 9].

Patchouli oil is one of the essential oil which contains sesquiterpene, which causes the alcohol has a patchouli aroma (flavor). In addition norpatchoulenol and nortetrapatcoulol although the levels are respectively 1% and 0.001%, but is able to produce a strong fragrance [10].

Material and Method

Material

Patchouli oil has been obtained from the patchouli farmers in West Aceh and South Aceh, Indonesia. The dregs of coffee powder Arabica taken from stalls selling drinks coffee in Aceh, and fly ash from waste of palm shells, taken from Singkil.

Quality of Patchouli Oil, Measurement and Equipment

Measurement of quality of patchouli oil is measured with a pycnometer to determine the specific gravity, refractometer to determine the refractive index, a spectrophotometer to determine color, AAS to determine levels of Fe, and GC-MS to determine the composition of patchouli oil. Measurement of quality both before adsorption and after adsorption with adsorbent waste coffee grounds and fly ash, to level Fe and composition of patchouli oil.

Preparation Adsorbent Dregs Of Coffee Powder And Fly Ash .

Dregs of coffee powder obtained from a coffee shop Nufana Banda Aceh Darussalam, washed with warm water until the washing water colored clear. Then dried in an oven at a temperature of $100^{\circ}C$, to obtain a stable weight. Furthermore dregs of coffee powder is crushed with a ball mill, and screened. Dregs of coffee powder is then activated with hydrochloric acid 0.1 N. Subsequently dried at $100^{\circ}C$. The dregs of the coffee powder ready for use.

Fly Ash (Taken from the Singkil plant and ready for use)

Measurement of parameters in patchouli oil

Measurement of initial parameters of patchouli oil is done in accordance with the parameters that we want to know (Specific gravity, refractive index, solubility in Ethanol 90%, color, and Fe content in patchouli oil in accordance with the way the Standard method. Likewise, patchouli oil composition is characterized by gas chromatography-mass spectrometry GC-MS).

Measurement of (Fe content and the composition of patchouli oil), done again after adsorpted with dregs of coffee powder, and fly ash, Likewise, patchouli oil composition characterized again by gas chromatography-mass spectrometry (GC-MS).

Results and Discussion

Specific gravity

Specific gravity of patchouli oil which was adsorbed by the adsorbent of dregs of coffee powder and fly ash are fulfilling SNI 06-2385-2006. This can be seen in the image below.

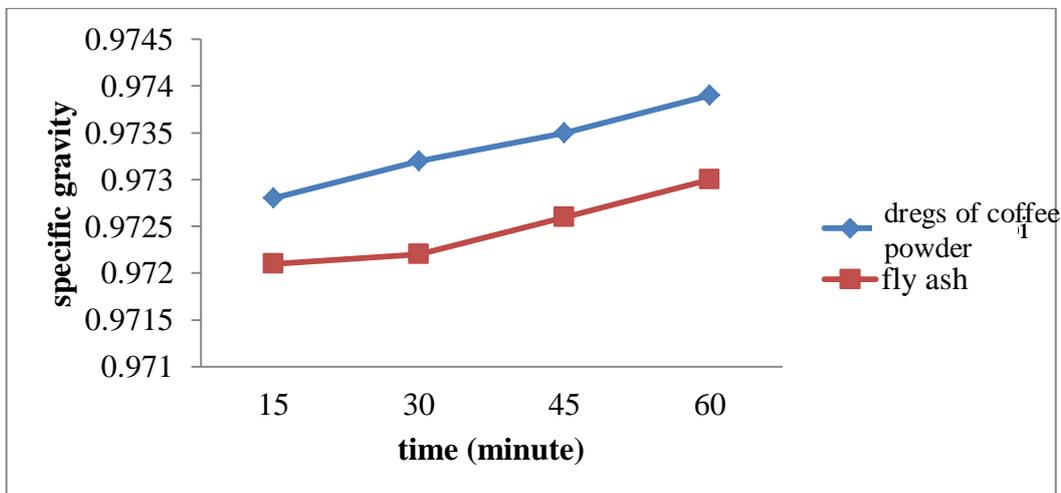


Figure 1. The relationship between adsorption time with specific gravity of patchouli oil with a mass of 1 gram of adsorbent.

In Figure 1 above it can be seen that at 15 minutes of contact time between patchouli and adsorbent, specific gravity of patchouli oil obtained is 0.9728, with adsorbent from dregs of coffee powder and specific gravity was 0.9721 for fly ash. Specific gravity fulfill SNI.

Measurement of specific gravity for the contact time between patchouli and adsorbents to 60 minutes causes the specific gravity of patchouli oil produced rose to 0.9739 with dregs of coffee powder and fly ash adsorbent is 0.9730.

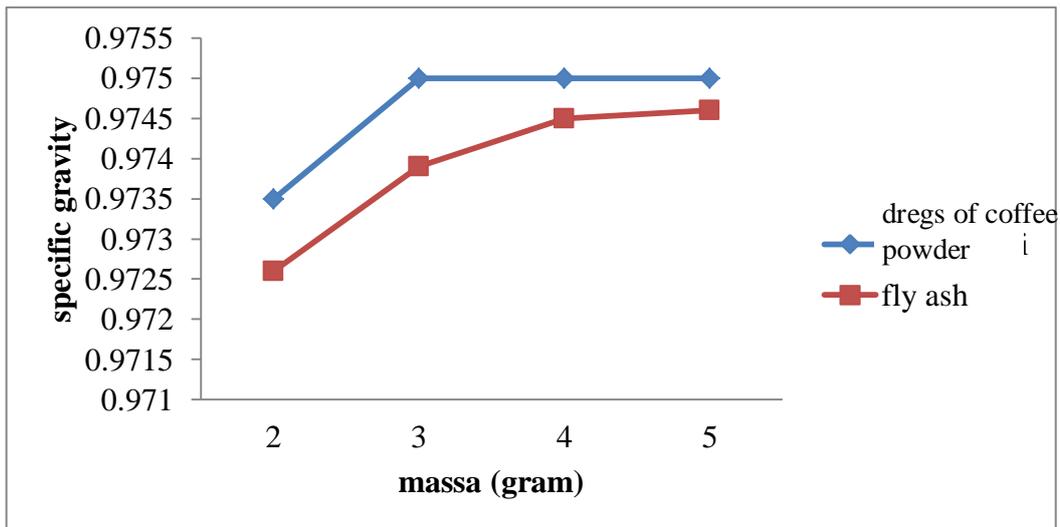


Figure 2. Relationship between adsorbent mass against gravity of patchouli oil in 45 minutes

In Figure 2 above shows that the weight of the adsorbent 2 g was obtained gravity of 0.9735 to 0.9725 for the dregs of coffee powder and fly ash. The specific gravity that meet SNI (0.943 to 0.983). Measurements of density made up to 4 grams cause the resulting density increased to 0.9749 for the dregs of coffee powder and and 0.9745 for fly ash.

These observations indicate that the stirring time between adsorbent and patchouli oil for 15 minutes and 1 gram of adsorbent, showed a specific gravity that meet SNI.

Density is one of the important criteria in determining the quality and purity of essential oils. Essential oil specific gravity value is defined as the ratio between the weight of oil with weight of water at a volume of water equal to the volume of oil. Specific gravity often associated with weight fraction of the components contained therein.

Refractive Index

The results of this study demonstrate that the refractive index of patchouli oil in accordance with SNI 06-2385-2006 ranging from 1.504 to 1.514. The more the water content, the smaller the refractive index value. This is because the nature of the water that is easy to refract incoming light.

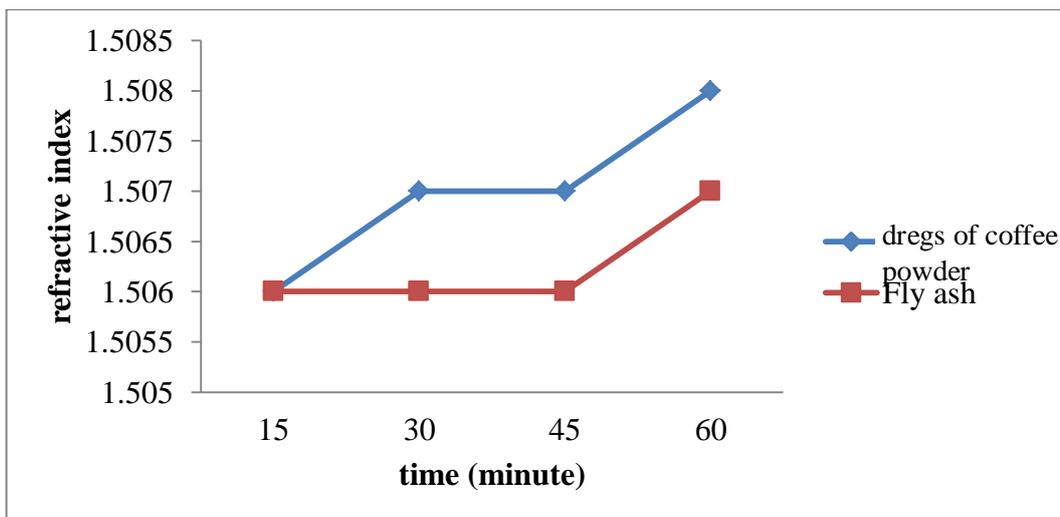


Figure 3. The relationship between the mass of adsorbent to the refractive index of patchouli oil with a mass of 1 gram of adsorbent

In Figure 3 above it can be seen that the dregs of coffee powder and fly ash with a mass of 1 gram and contact time 15 minutes with patchouli oil, resulted in a refractive index of 1.506, which meets the ISO standard (1.506 to 1.516).

From the picture above we can see that the refractive index produced the longer the contact time between the dregs of coffee powder with patchouli oil, higher refractive of index value. The higher the value of the refractive index is produced, the better the quality of patchouli oil (in range standard). The relationship between adsorption to the refractive index of patchouli oil in 45 minutes in figure 4.

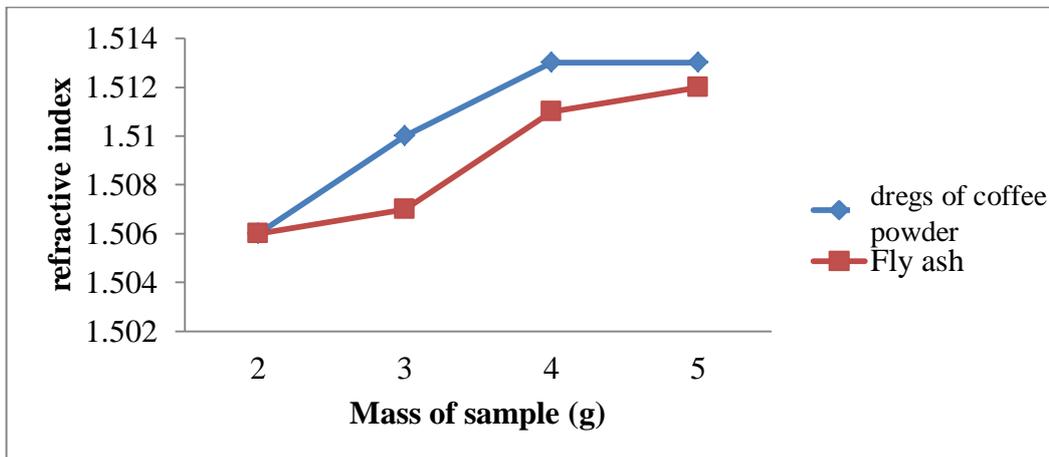


Figure 4. The relationship between adsorption to the refractive index of patchouli oil in 45 minutes

In Figure 4 above can be seen in coffee grounds and fly ash to the mass of adsorbent 2 grams, at 45 minutes, the refractive index is 1.506, meet ISO standards (1.506 to 1.516), whereas the mass of 5 grams obtained refractive index of 0.513 for coffee grounds and fly ash at 0.512. The images can be seen on the refractive index of the resulting higher.

These observations indicate that the stirring time between adsorbent and patchouli oil for 15 minutes and 1 gram of adsorbent, shows the index bias that meet SNI.

Metal Fe

The content of the high iron content in patchouli oil, cause the color of the patchouli oil becomes reddish brown. Iron ions dissolved in patchouli oil is derived from the tool flute made of iron during the distillation process takes place. Ferrous metal content in patchouli oil can be seen in the image below.

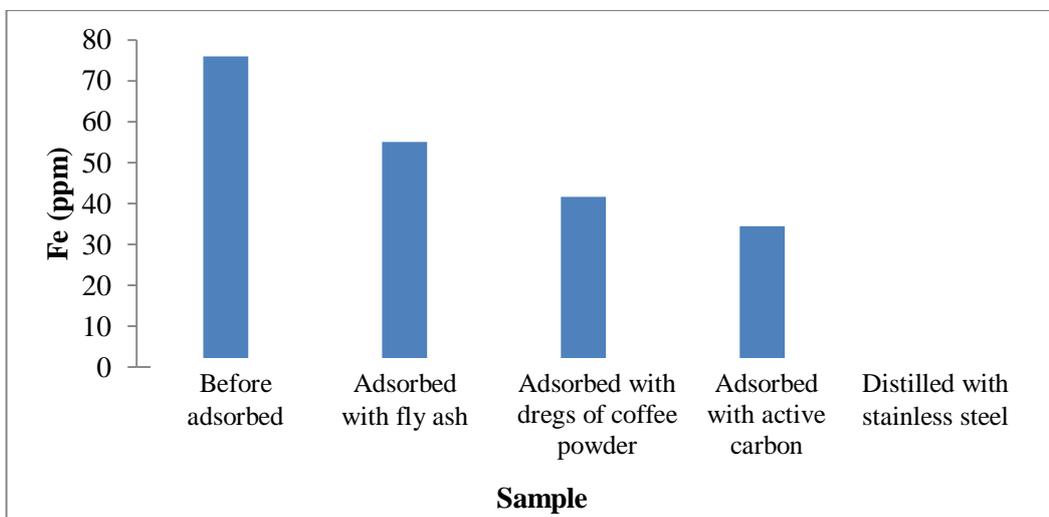


Figure 5. The content of iron in patchouli oil

Figure 5, explaining that the content of the iron content of patchouli oil before the adsorbed (distilled in the drum) is 75.936 ppm, whereas the levels of iron content of patchouli oil is 0.6675 ppm (distilled in stainless steel)

Patchouli oil that contains high levels of iron absorbed by using adsorption methods with dregs of coffee powder and fly ash as adsorbent. After adsorption for 15 minutes with the amount of adsorbent as much as 1 gram and 80 rpm stirrer rotation, volume of patchouli oil is 50 ml. Adsorbent of the dregs of coffee powder can reduce levels of iron (the start is 75.936 ppm) as much as 34.306 ppm (44.77%), and by using fly ash adsorbent can reduce the iron content as much as 20.854 ppm (26.33%) .

Gas chromatography-mass Spectrophotometer (GC-MS)

The results of GC-MS analysis of the components of essential oil of patchouli can be obtained in chromatogram as in Figure 6 below.

a. Analysis of Patchouli Oil

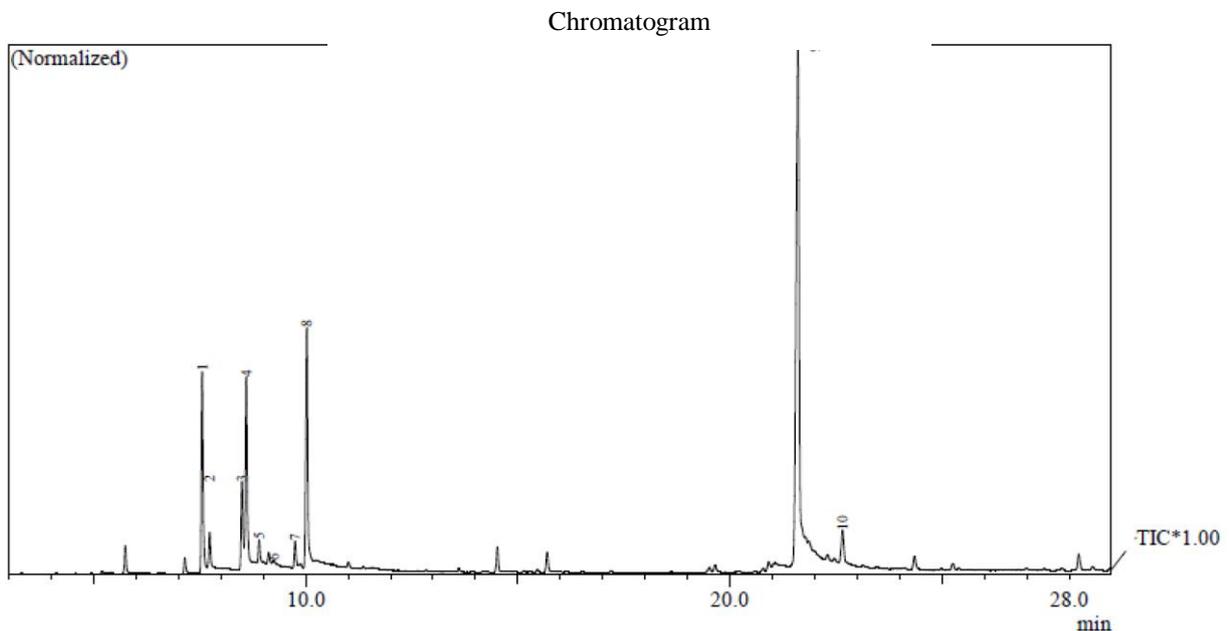


Figure 6. The initial patchouli oil composition (GC)

Furthermore, patchouli oil is absorbed by the dregs of coffee powder and fly ash, patchouli oil produced further characterized again by GC-MS.

Composition of chemical compounds in the early patchouli oil, and the chemical composition after adsorbed by the dregs of coffee powder and the fly ash can be seen in Table 1 below.

Table 1. Composition of constituent chemical compounds of patchouli oil before and after adsorbed

No	Composition	Nilam oil before absorbed	Nilam oil after absorbed/using with:		
			stainless steel	adsorben fly ash	adsorben dregs of coffee powder
1	Azulene	9.9	7.1	8.95	9.24
2	Caryophyllene	3.25	2.27	4.8	4.9
3	Alpa-Patcoulene	3.58	3.52	2.98	3.18
4	Alpa-Guene	2.09	20.67	21.21	12.76
5	Patchouli alcohol	37.76	52.01	48.71	45.98
6	Seychellene	7.76	8.89	6.66	6.9

Based on data from patchouli oil composition of the gas chromatography - mass spectrometry in the above can be seen that with adsorben of dregs of coffee powder, causing the composition of patchouli oil is changed, which resulted in higher percentage of patchouli alcohol became 45.98% from 37.76% initial composition, rise up to 4.56%. Adsorben of fly ash, causing the proportion changed, raising the percentage of patchouli alcohol became 48.71% from 37.76%, rise up to 10.95% . Composition change occurs, due to the absorption of other compounds that cause a rise in the percentage of patchouli alcohol, the amount of the patchouli alcohol is fixed.

The results of chemical analysis of the chromatogram of patchouli oil, have shown that the main compound contained is patchouli alcohol. In international trade levels of patchouli alcohol is the main parameters that distinguish the price even be a password to get into flovour fragrance industry, whereas the trade area (Aceh, Indonesia) content of patchouli alcohol does not affect the selling price of patchouli oil, only the interest of buyers is reduced for patchouli oil levels patchouli lower alcohol.

In this study was also conducted characterization of patchouli oil which was refined by stainless stell, with GC-MS. The result of this patchouli oil containing patchouli alcohol in it is 52.01%.

Solubility in Ethanol 90%

Patchouli oil after absorbed for 45 minutes with fly ash and dregs of coffee powder (4 g) can be completely soluble in alcohol 90%. At each addition of 1ml of patchouli oil with 10 ml of 90% ethanol can dissolved completely. Greater solubility of patchouli oil in alcohol, the better the quality of patchouli oil, can be seen in Figure 7 below..

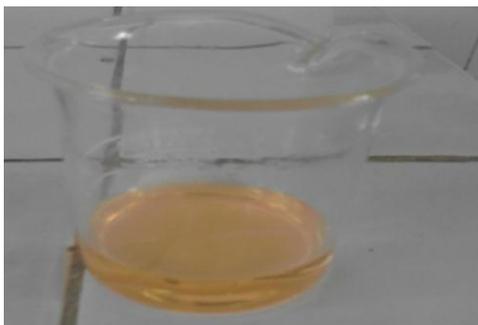


Figure 7. patchouli oil soluble in ethanol 90%

Color

Analysis of color with a spectrophotometer, in this study patchouli oil with original reddish-brown color after the adsorption process, becoming pale yellow in accordance with SNI 06-2385-2006

Tranmittance of beginning of patchouli oil which was distilled using a drum is 2.3682%, with very low transmittance values can be known that the patchouli oil before adsorbed have extremely dark colors. In the picture below can be seen the results of the analysis of absorbance and transmittance of patchouli oil after adsorption.

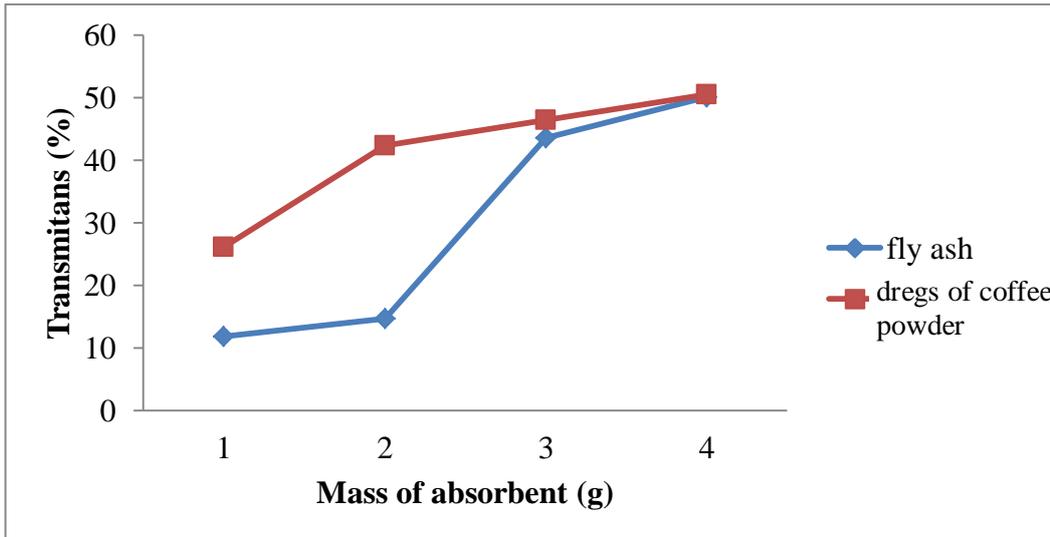


Figure 8. analysis transmittance of patchouli oil with a spectrophotometer after absorbed for 45 minutes with fly ash and dregs of coffee powder (1s/d4 g).

In Figure 8 above it can be seen that the much more the amount of adsorbent used, the higher the value transmittance (light transmitted), meaning that patchouli oil after adsorbed getting brighter. By using the adsorbent of the dregs of coffee powder transmitted light is higher than the use of fly ash . After absorption by the dregs of the dregs of coffee powder and fly ash, transmittan be 50%.

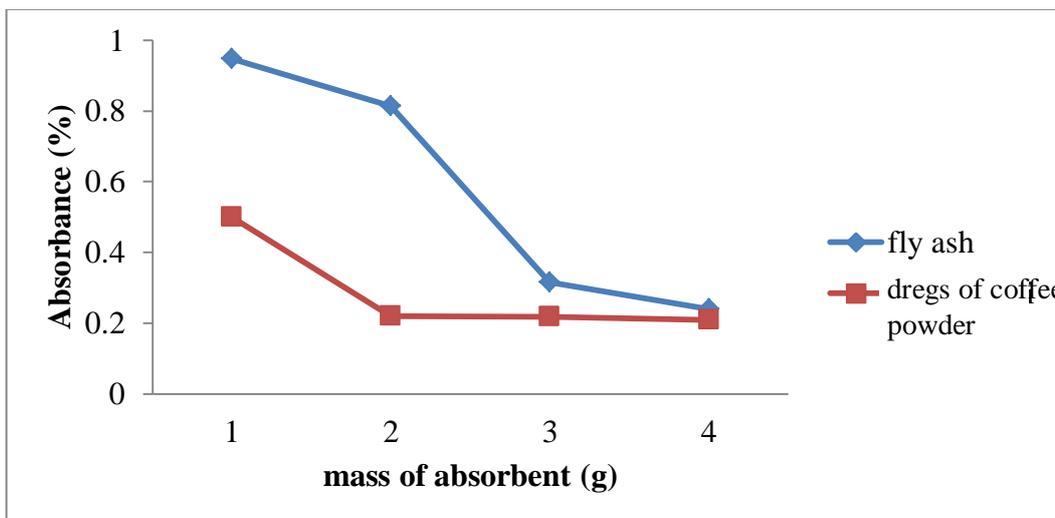


Figure 9. analysis absorbance of patchouli oil with a spectrophotometer after adsorbed for 45 minutes.

Absorbance (the amount of light that is absorbed), the more clear patchouli oil, the absorbance is getting smaller.

a. Analysis visually

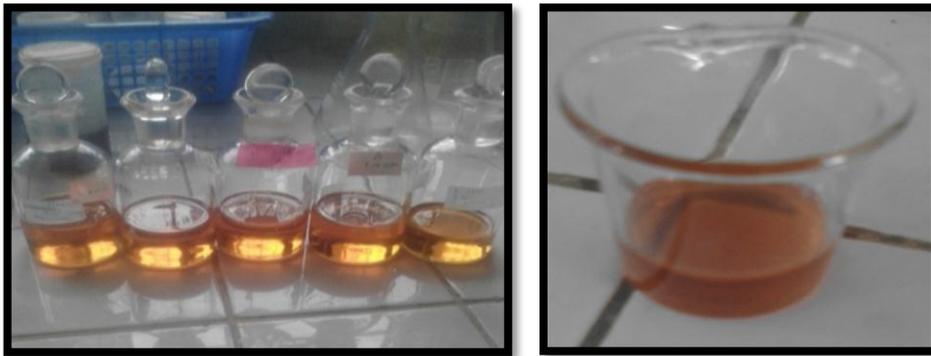


Figure 10. patchouli oil in adsorbed using coffee grounds adsorbent 2 grams

Figure 10 above can be seen visually the difference in color that occurs after adsorbed using the dregs of coffee powder. Patchouli oil that was originally yellow-brown into bright yellow color. This indicates that the dregs of coffee powder are used as adsorbent is able to absorb the dye in patchouli oil. In this study, patchouli oil obtained from adsorption in accordance with SNI 06-2385-2006 that is light yellow.

Conclusion

These observations indicate that the stirring time between adsorbent and patchouli oil (50 mL) for 15 minutes and 1 gram of adsorbent, shows the specific gravity, and refractive index that meet SNI.

Adsorbent of the dregs of coffee powder can reduce levels of iron as much as 34.306 ppm or 44.77%, (the start is 75.936 ppm) by using fly ash adsorbent can reduce the iron content as much as 20.854 ppm (26.33%).

Patchouli oil adsorption results can be completely soluble in alcohol 90%. In the comparison of 1 ml patchouli oil: 10 ml of 90% ethanol.

Transmittance of Patchouli oil from distillation using drums is 2.3682%, after adsorption increased to 50%, the number of adsorbent much as 4 g, and a contact time of 45 minutes, conversely the absorbance becomes small.

Absorbent most effective in increasing the percentage of patchouli alcohol, is the fly ash, from, 37.76% to 48.71%, rise up to 10.95%.

Acknowledgments

The author would like to thank the Universitas Syiah Kuala University, Ministries Of Education And Culture, which funded the research.

References

1. Alfian, Z, 2003. Analisis Kadar Logam Besi (Fe) dari Minyak Nilam (Patchouly Oil) yang diperoleh dari Penyulingan dengan Menggunakan Wadah Kaca, Stainless Steel dan Drum Bekas secara Spektrofotometri Serapan Atom, Vol. 7. No.2, 2003: 55-58.
2. Franca A, Leandro S, Oliveira L S, Ferreira, 2009. Kinetic and equilibrium studies of methylene blue adsorption by spent coffee ground, *Desalination*, Vol. 249.
3. Virote B, Chiyon C, Tanthapanichakoon W, and Jarudilokkul S, 2004. Removal of heavy metals by adsorbent prepared from pyrolyzed coffee separation and purification technology Vol. 36, p 11-22.

4. Namane, A., A. Mekarzia, K Benrachedi, N. Belhaneche- Bensemra, A .Hellal, 2005. Determination of the adsorption capacity of activated carbon made from coffee grounds by chemical activation with $ZnCl_2$ and H_3PO_4 , *Journal of Hazardous Materials*, B119, pp 189- 194,
5. Ondokuz Mayıs, 1993. The removal of heavy metals by using agricultural wastes, *water sciences & technology* Vol 28 No. 2.
6. Amarasinghe, U. A.; Shah, T.; Turrall, H.; Anand, B. K., 2007. *India's water future to 2025-2050: Business-as-usual scenario and deviations*. Colombo, Sri Lanka: International Water Management Institute. 47p. (IWMI Research Report 123),
7. Viraraghavan, T., S. Tanjore, 1994. *The Use of Inexpensive Adsorbents To Remove Pollutants From Waste Water*, Editor, Environmental System Reviews, ENSIC –AIT, Bangkok, 52-64.
8. Guenther, E, 1949. *The Essential Oils*, volume I, Van Nostrand Reinhold Company, New York, 87-226.
9. Lawrence, M.B., R.J, 1990. *Progress in Essential Oils*, Perfumer & Flavorist. Vol. 15.
10. Akhila, A. and R. Tewari, 1984. Chemistry of Patchouli : A Review, *Current Rest. Aromat. Plants*, 6 (1), 38-54.
