



Simple Vacuum Distillation of Vetiver Oil from Smallholders for Quality Improvement

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Simple Vacuum Distillation of Vetiver Oil from Smallholders for Quality Improvement

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I. INTRODUCTION

Vetiver oil is an essential oil extracted from the roots of *Vetiver grass (Chrysopogon zizanioides, Linn Nash)*, a tropical grass growing wild or cultivated. The extraction can be done via water distillation, steam distillation, solvent extraction or expression (Dowthwaite and Rajani, 2000). As an essential oil, vetiver oil will find wider application due to the increasing tendency towards the use of natural materials. The classical roles of vetiver oil are flavor, fragrance, and aromatherapy (Lavania, 2003). It has been observed also that the vetiver oil has activities such as antimicroorganism, antioxidant, insecticide, and sedative.

Indonesia produces 60 to 75 tons of vetiver oil annually and becomes the main vetiver oil suppliers in the world, besides Haiti and La Réunion. About 90% of Indonesia vetiver oil is produced by smallholders in Garut, a small district about 200 km from Jakarta to the south-east. It is produced by water distillation at 4 to 5 bars in the conventional ways. The main equipment consists of a boiling vessel, a burner, and a condenser. Vetiver roots are packed on grids over a layer of water in the base of the vessel. Saturated steam which is produced by direct firing the vessel extracts the essential oil contained in the roots. Essential oil containing steam is then condensed to obtain vetiver oil.

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Due to primitive equipment and the lack of process control, the quality of most vetiver oil obtained could not satisfy the required standard. Minimum total vetiverol content of 50% is hard to obtain. In addition, the color of the vetiver oil is very dark, from dark brown to black. It is supposed due to overheating and oxidative degradation which results in tar like substances which are hard to separate by physical methods. Standard on vetiver oil requires the color from light yellow to reddish brown. Further treatment, therefore, is necessary to improve such vetiver oils in order to comply with standard qualities.

Many works have been published on vetiver oil. Aggarwal et al. (1998) studied the effects of harvest period, storage period after harvest, roots treatment and distillation time on the yield of extraction by distillation. They found that the extractable oil decreased with harvest delay and storage period. In addition, they found that cutting the roots did not affect oil yield significantly, water distillation gave oil recovery a little bit higher than steam distillation (0.28% vs 0.23%, based on dry root weight). Danh et al. (2010) have studied the extraction by an unconventional method, i.e., an extraction by supercritical CO₂ with ethanol as co-solvent. They found that pressure and ethanol concentration linearly affected oil yield but, in contrary, temperature in the range of 40 to 50 °C did not influence oil yield. It was found also that supercritical fluid extraction did not extract the metals in the vetiver roots giving an advantage for strict applications, such as foods and drugs. The effects of plant environments on the quantity and quality of extracted oil have attracted the attention of researchers. Adams et al. (2004) reported that cleansed vetivers gave lower oil yield with strictly different composition to those of non-cleansed vetivers. They found that the yield of oil from bacteria and fungi free vetivers was almost twenty times lower and contained large amounts of C₁₉-C₂₉ alkanes. The roles of microbes were also reported by Pripdeevech et al. (2006) who obtained larger oil yield and higher content of some low molecular weight volatiles from vetiver plants grown in normal soil with added microbes compared to normal soil (0.27% vs 0.18%). Adams et al. (2008), however, reported no correspondence between *arbuscular mycorrhizal fungi* colonization and oil yield, and postulated that the composition of vetiver oils are majorly controlled by vetiver genes. Massardo et al. (2006) reported variations in oil yield and in the contents of isovalencenol and

khusimol in the obtained oils during growth of roots. The effects of climatic conditions have also been studied by Kotoky et al. (2011) who found that rain-fall played a role on the yield and quality of vetiver oils. Their data indicated that vetiver oil quality is closely related to the metabolism of its root which is influenced by climatic conditions. The complexity of vetiver oil composition has challenged many researchers in developing qualitative and quantitative analysis, among them are Cazzausus et al. (1988), Weyerstahl et al. (200a, 200b), Paillat et al. (2012) and Filippi et al. (2013). The works of Weyerstahl et al. (2000a, 2000b) and Filippi et al. (2013) are very intensive. In recent years, biological functionalities of vetiver oil have much attention. The antibacterial and antifungal activities of vetiver oil have been reported by Prabuseenivasan (2006), Gupta et al. (2012), and Sangeetha and Stella (2012). The insecticidal activities of vetiver oil have been reported by Zhu et al. (2001a, 2001b). The antioxidant, antinociceptive, and anti-inflammatory activities, the sedative effects, and the toxicity of vetiver oil have also been studied (Kim et al., 2005, Lima et al., 2012, Thubthimthed et al., 2003, Sinha et al., 2014). Apart from the many works, no attention has been paid to upgrade the poor quality of crude vetiver oil which commonly found from smallholders.

II. MATERIALS AND METHODS

Fig. 1 shows the schematic of the distillation apparatus. The apparatus mainly consisted of a distillation column with insulation, a condenser and a condensate collector. The apparatus was equipped with a cooling water system with controlled temperature and connected to a vacuum system. The pressure inside the apparatus was controlled by a needle valve and was set at 10 mmHg. Boiling points were measured by using a thermometer. Crude vetiver oil of 300 ml was used for each run. Distillates were collected at every 20% recovery until 80% volume. Three vetiver oil samples from small distillers were used. The parameters observed included density, refractive index, acid number, ester number, ester number after acetylation, and vetiverol content. Density was measured by a piknometer. Refractive index was measured using a refractometer. Acid number was measured by titration. Ester number was measured by saponification. Acetylation was done using acetic anhydride. The content of free alcohol as vetiverol was calculated from ester number of original oil and that of acetilated oil. The parameters observed also included color, odor, and solubility in ethanol. Color was observed visually and also measured by a Lovibond Tintometer. Solubility in ethanol was observed as "soluble" or "not soluble" in ethanol at volume ratio of 1:1. All chemicals for analysis were of pro analysis grade.

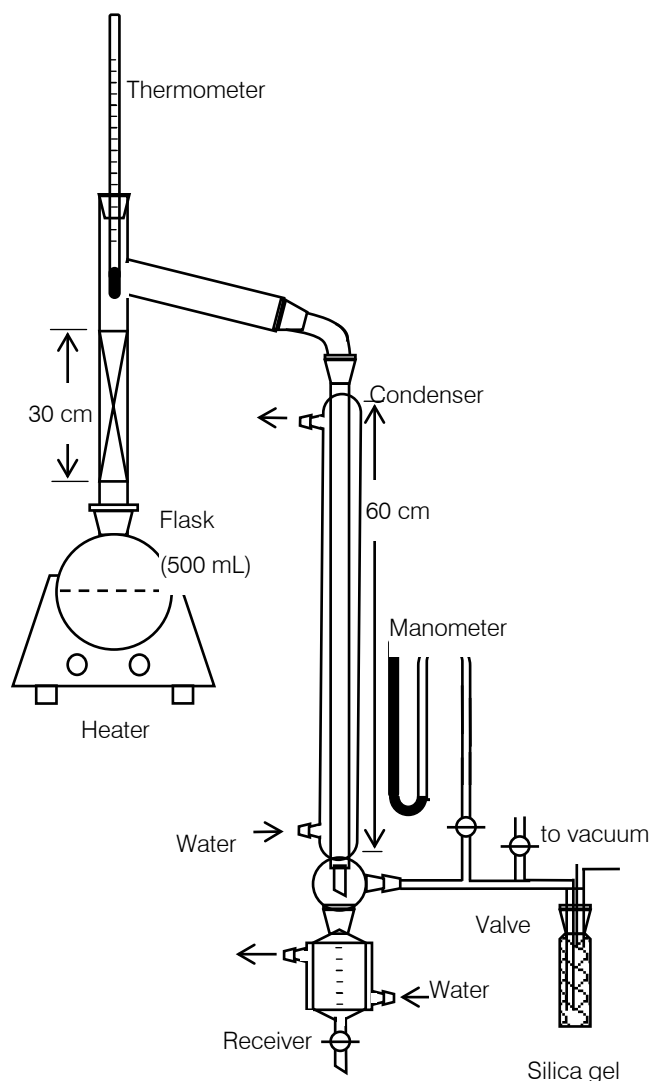


Fig. 1 : Schematic of experimental apparatus

III. RESULTS AND DISCUSSION

a) Vetiver Oil Samples

The characteristics of three vetiver oil samples studied here are given in Table 1, columns 3 to 5. The Indonesia standard quality (SNI, 2006) are shown in the rightmost column. As shown in the table, all three samples had vetiverol contents lower than the required standard. Moreover, they smelt smoky and their color were dark black. These three quality parameters are commonly fail to achieve by most smallholders. It is due to poor distillation operation. Many factors affecting the distillation of vetiver root as discussed by Aggarwal (1998) which are not considered in the field. The smoky odor comes from soil materials which were not removed prior to distillation. The vetiverol content appeared to correlate with density. In this case, lighter vetiver oil exhibited higher vetiverol content. All samples were soluble in ethanol at 1:1 volume ratio.

Table 1 also shows the characteristics of Indonesian vetiver oil samples from Guenter [1950], columns 6 to 9. This author procured four samples of vetiver oils from Indonesia in the past, said to be genuine samples, with physicochemical properties as shown in Table 1. The data was completed with vetiverol contents which were calculated in this work from the

ester numbers. In regards to vetiverol content, the samples used in this work were inferior to those from Guenter [1950]. The samples used here had higher ester numbers but lower ester numbers after acetylation resulting in lower total vetiverols. In contrast to the samples of this work, the genuine samples from Guenter [1950] complied with the required standards.

Table 1 : Characteristics of vetiver oil samples

#	Parameter	Samples in this work			Guenther (1950)†				Standard‡
		A	B	C	I	II	III	IV	
1	Relative density (20/20 °C)	0.987	0.994	1.024	1.009	1.009	1.007	0.991	0.980 to 1.003
2	Refractive index (20 °C)	1.52	1.52	1.52	1.526	1.5271	1.526	1.5258	1.52 to 1.53
3	Acid number (mg KOH/g)	27	30	34	31.1	32.5	30.2	12.5	10 to 35
4	Ester number (mg KOH/g)	24	22	26	2	12.6	1	4	5 to 26
5	Ester number after acetylation (mg KOH/g)	133	124	117	141.5	150	152.1	129	100 to 150
6	Total vetiverol (%)	46	43	39	61*	60*	67*	54*	Minimum 50%
7	Color	Too dark	Too dark	Too dark	NA	NA	NA	NA	Pale yellow to Reddish brown
8	Odor	Smoky	Smoky	Smoky	NA	NA	NA	NA	Vetiver characteristic
9	Solubility in ethanol 95% (1:1)	Clear	Clear	Clear	NA	NA	NA	NA	Clear

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9	Solubility in ethanol 95% (1:1)	Clear	Clear	Clear	NA	NA	NA	NA	Clear

†Genuine samples analyzed by Guenter (1950). *Calculated in this work from ester numbers. ‡National Indonesian Standard. NA: not available

b) Distributions of Physicochemical Properties

Fig. 2 to 7 illustrate the distribution of physicochemical properties at various distillate recovery. The densities of distillates varied in the range of 0.94 to 1.00, as shown in Fig. 2. As can be expected, lighter distillate distillate had lower relative density. All distillates from samples A and B had density lower than the required minimum density, although their initial sample obeyed the required standard density. The density of sample C was higher than the upper limit of standard density. However, its distillates from 60% to 80% recovery were in the range of standard density. As seen in the figure, the range of standard density is narrow. The deviation in densities were actually not significant, only at the

second decimals. The largest was shown by 20% recovery of sample A (a deviation of 0.04 from the lower limit). Fig. 3 shows that the refractive index did not vary significantly. All distillates had refractive index in or close to the range of 1.52 to 1.53, except at 20%-recovery.

In contrast to essential oil in general, vetiver oil exhibited high content of free fatty acid. The contents of free fatty acid as acid numbers are shown in Fig. 4. In general, acid number increased with %-recovery. Lower %-recovery tend to decrease acid number and make the acid number lower than the minimum acid number. The distillation cuts between 40% and 60% and between 60% and 80% had acid numbers significantly larger than the other lighter distillation cuts. This indicated that acid

compounds in vetiver oil concentrate at the heavier fractions.

Fig. 5 and Fig. 6 shows the ester numbers before and after acetylation, respectively. Ester number before acetylation in general increased with %-recovery but ester number after acetylation decreased with %-recovery. Most of distillate obeyed the standard for ester numbers before acetylation. Ester numbers after acetylation of light distillate (20% to 40%) were higher than the maximum allowed numbers. As difference in these two ester numbers indicates vetiverol content, this indicated that the vetiverol content should be higher at the lighter distillate.

Fig. 7 shows the distribution of vetiverol content. Vetiverol contents were found to decrease with distillate recovery. The better the sample, the higher the vetiverol content of the distillate obtained. Minimum vetiverol content of 50% could be obtained from samples A and B until 60% recovery and almost achieved at 80% recovery. It means that the vetiverol content of samples A and B could be improved to very close to the required standard at the expense of 20% oil lost. With sample C, however, more quantity have to be sacrificed to achieve standard content of vetiverol. At 80% recovery, sample C gave distillate with a vetiverol content of 42%. It is significantly lower than the required standard, although the vetiverol contents have improved compared to the initial samples. Minimum vetiverol content of 50% could be achieved until 60% recovery for sample C.

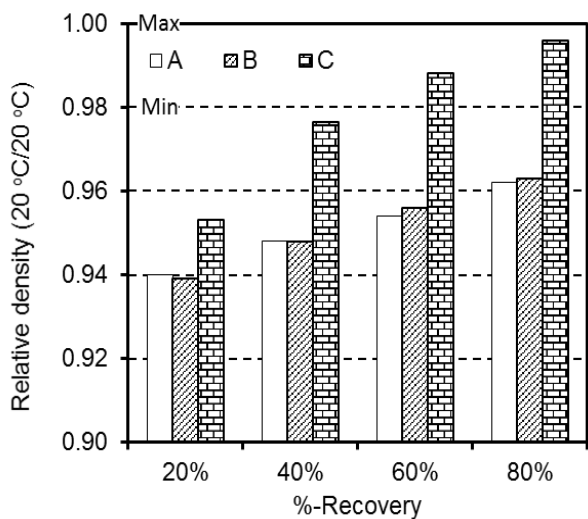


Fig. 2 : Distribution of relative density

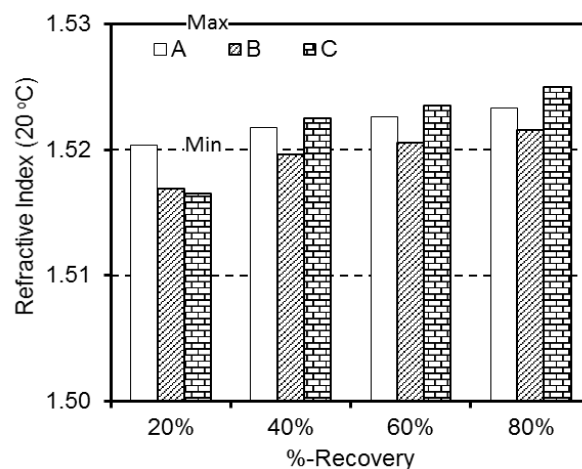


Fig. 3 : Distribution of refractive index

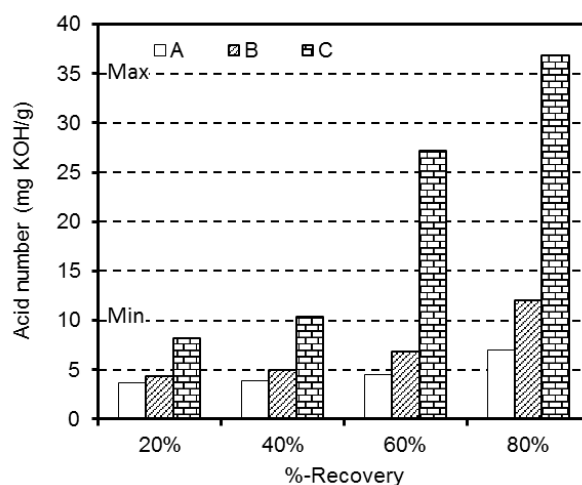


Fig. 4 : Distribution of acid number

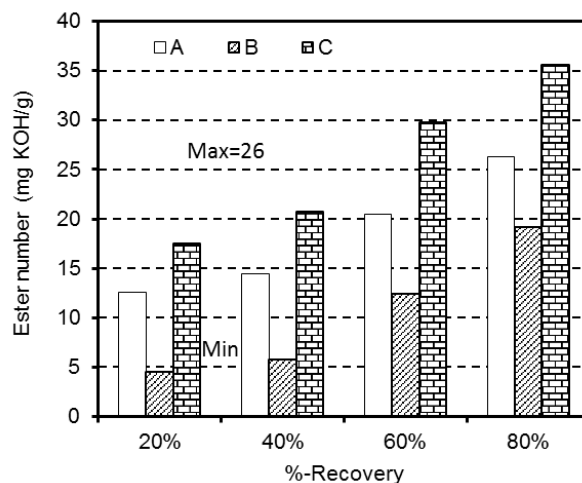


Fig. 5 : Distribution of ester number

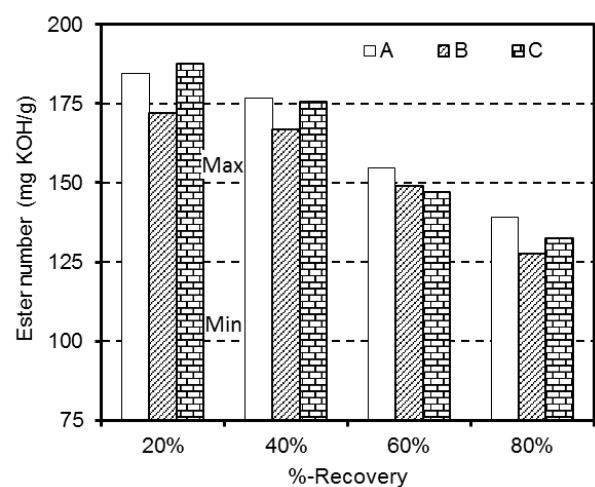


Fig. 6 : Distribution of ester number after acetylation

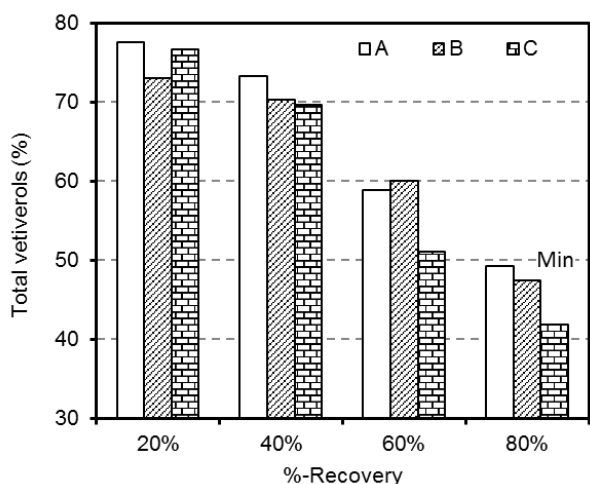


Fig. 7 : Distribution of vetiverol content.

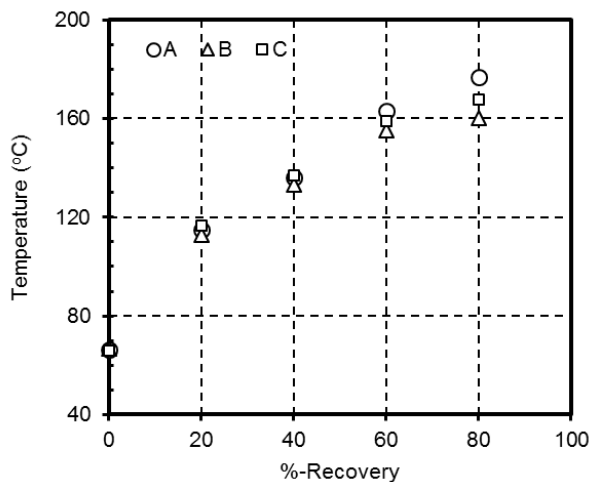


Fig. 8 : Distribution of boiling point

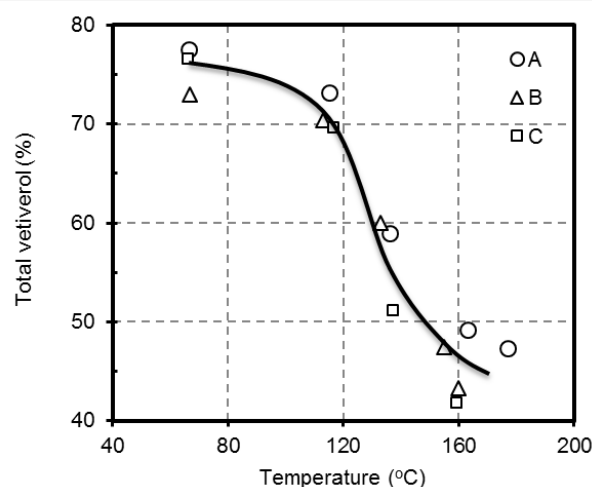


Fig. 9 : Vetiverol content vs boiling point.

Fig. 8 shows the temperature curves of distillation. The boiling temperatures of all samples were close each other, although they were getting away along with temperature. The relationship between vetiverol content and boiling point are shown in Fig. 9. Although the data is rather scattered, they could be approximated by a third order polynomial. More importantly, it clearly shows that lower distillation temperature gave higher vetiverol content. This reveals that the impurities are concentrated at the heavy fractions and distilling the samples under vacuum improved their vetiverol contents.

There are no references for the complete distribution of acid and ester numbers and vetiverol content in respect to vetiver oil distillation cuts. Gildemeister (1913) and Weyerstahl et al. (2000a) presented several distillation cuts of vetiver oils, however, for the purposes of showing the cuts in which individual components are concentrated. The results here showed that all parameters regarding compound groups in vetiver oil were continuously distributed. This indicated that vetiver oil composition is quite complex, consisting of many components with a wide boiling point range. Hundreds of components have been identified (Weyerstahl et al., 2000b and Filippi et al., 2013). However, many components are still unidentified.

c) Color, Odor and Ethanol Solubility

All distillates were soluble in ethanol at volume ratio of 1:1. The Indonesian standard qualitatively requires that vetiver oil has color from pale yellow to reddish brown. None of the vetiver oil samples studied here comply with this color standard. The colors of all samples were dark (almost black). In contrast, all distillates complied with the required qualitative standard. Although there is no quantitative standard for color of vetiver oils, popular color scales for oils and fats or chemicals in general were used in this study to describe the colors of distillates. Sample C was selected to study the color as sample C was the darkest sample.

The colors of distillates from sample C were measured using a Lovibond Tintometer. Table 2 shows the results in the scales of Lovibond RYBN, AOCS RY, FAC, Gardner, and ASTM. Lovibond RYBN scale is based on different densities of red, yellow, blue and neutral. The AOCS-Tintometer Color Scale is a special red and yellow version of Lovibond RYBN. FAC Scale is divided into 5 groups: Scale 1 (1, 3, 5, 7, 9) for lighter colored fats; Scale 2 (11, 11a, 11b, 11c) predominantly for yellow fats; Scale 3 (13, 15, 17, 19) for dark fats (red cast); Scale 5 (31, 33, 35, 37, 39, 41, 43, 45) for very dark fats, predominantly red. The Gardner scale ranges from a pale yellow to a red in shade and is described in terms of the values 1-18. ASTM is a single number, one

dimensional, color scale ranging from a pale straw through to a deep red. The Lovibond RYBN and AOCS RY scales showed that the basic colors of distillates from Sample C were red and yellow. In addition, the densities of red and yellow increased with distillate recovery, causing the appearance of distillate is getting reddish brown as distillate recovery increased. Similarly, considering the FAC, Gardner and ASTM scales, it could be seen that the color of distillate became reddish as the recovery increased. Thus, brown become predominant in 80%-recovery, however, it was still reddish brown. The odor, however, could not be removed. All distillates still smell of smoky, thus, the distillates need further treatment for odor removal.

Table 2 : Color of distillates from Sample C

Recovery	Lovibond RYBN				AOCS RY		FAC	Gardner	ASTM	Visual
	Red	Yellow	Blue	Neutral	Red	Yellow				
20%	2.4	39	0	0.1	2.3	32	11	10.8	2.0	Light yellow
40%	3.0	46	0	0.1	2.9	40	11	11.2	2.1	Light yellow
60%	3.3	57	0	0.1	3.3	50	11	11.6	2.3	Light yellow
80%	10.9	70	0	0.1	9.9	70	33	14.7	3.9	Reddish brown

IV. CONCLUSION

A Simple vacuum distillation has been used to improve the quality of vetiver oils from smallholders. Vetiverol content, which is the difficult quality parameter to achieve could be improved. For all samples, the Indonesian standard on vetiverol content could be obeyed by distilling 60% samples. The better the sample, the less volume of initial oil that have to be sacrificed to achieve the standard vetiverol content. The standard vetiverol content could be achieved from samples A and B, which are better than sample C, until 80% recovery. Although all sample appeared to black and did not meet the standard appearance, standard in color could be achieve by all distillates. The distillate had appearance from yellow to reddish brown, with Gadner scale of color ranging from 10.8 to 14.7. Distillation, however, slightly disturbed the achievement of other parameters including density, acid number and ester number. Lower distillation fraction tend to shift the values of these parameters from their standards. The odor however could not be removed.

V. ACKNOWLEDGMENT

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