



Removal of Ferrous using Citric Acid in Patchouli Oil Purification by Complexometry

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ABSTRACT

This study is aimed to examine citric acids as a potential chelating agent to decrease colloidal impurities in patchouli oil to improve its quality. It covers colour, specific density, refractive index, acid value, iron content, oleoresin oil content, and patchouli alcohol. Complete Randomized Design with factorial design is used with two factors and repeated 3 times. Factors are (1) citric acid concentration consists of 0.25%, 0.5%, 1.0% and 1.50% (w/v), (2) stirring time of 30, 60 and 90 minutes. Further, purified oil by citric acid was compared to purified oil by Ethylene Diamine Tetra Acetate (EDTA). Findings show that the concentration of chelating agents and the time of stirring have an effect on the quality of patchouli oil. The higher the chelating concentration and the more the stirring time, the better the quality of purified patchouli oil in terms of colour, specific density, refractive index, acid value, and iron content. Findings also show that citric acid has almost the same performance as EDTA. The main components in patchouli oil (patchouli alcohol and oleoresin oil) are not affected by treatment. Purified patchouli oil by using citric acid meets Indonesian National Standard (SNI) requirements so citric acid is one of the potential chelating agents.

Keywords: Purification process, essential oil, chelating agent, patchouli oil, iron content.

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Introduction

Patchouli oil is extracted from the Pogostemon patchouli plant or Pogostemon cablin Benth. It is one of the priceless essential oil and until now there has been no other type of essential or synthetic oil that can replace patchouli oil as a fragrance binder (perfume) [1]. Patchouli oil is usually not fractionated into its derivatives such as citronella oil, vetiver, clove and others, because it has very harmonious components and no one component is very prominent and is the most difficult volatile essential oil compared to other essential oils.

Others, so that it can be used to make compositions of a fragrance or perfumery compound as a cosmetic raw material [2]. Patchouli oil has fixative properties, namely the ability of the oil to bind several other odours/perfumes so that the composition forms a unified odour. The smell of patchouli oil is pungent, strong, long-lasting, and musty.

However, in recent years, the price of patchouli oil from local communities has fallen significantly due to the poor quality of patchouli oil (high colloidal impurities). The impurities cover dark brown colour, high acid number, high iron content (ppm) and high oleoresin oil content (%). The nature of patchouli oil

is determined by its chemical compounds. These components can be terpenes, alcohols, aldehydes, acids, esters, ketones and others. In addition, these components may contain saturated or unsaturated bonds so that patchouli oil is easily oxidized, hydrolyzed and polymerized [3].

Most local community farmers in developing countries use metal iron distillers [[4], [5]]. Patchouli oil turns dark in colour due to the influence of Fe_2O_3 which is a sensitizer to double bonds and the compounds contained in the oil [6]. Due to the influence of base and temperature, the resinification reaction in essential oils is accelerated [7]. The reaction between metal ions and acids in the oil will form salts which will make the oil darker and more concentrated. The decline in the quality of patchouli oil can be prevented by using stainless steel distiller. However, it is considered expensive by local community farmers [8].

Patchouli oil purification can be done by vacuum distillation and re-distillation [3], adsorption [9], and using chelating agents [10]. The redistillation purification method has the disadvantage of being relatively expensive, long processing time and charred-smelling oil [[11], [12]]. Purification of dark-coloured patchouli oil by flocculation in principle is to bind the metals contained in the oil by adding a chelating agent to form a complex salt that coagulates and settles. Furthermore, the precipitate formed is separated from patchouli oil through filtration. Chelating agents have been used such as tartrate acid and Ethylene Diamine Tetraacetic acid (EDTA). However, those chelating agents are quite difficult and more expensive for local community farmers. Meanwhile, citric acid has a chelating agent effect, is organic, and has abundant availability [13]. Thus, this study aims to examine citric acids as a potential chelating agent to decrease colloidal impurities in patchouli oil to improve its quality.

Experimental part

Patchouli oil used in this study is patchouli oil obtained from a local community farmer in West Sumatera, Indonesia. The citric acid solution used was a 65% concentration with the addition of 1% ethanol 96%. Then, the amount of patchouli oil was added with a solution of a chelating agent (65%) as much as 0.25%, 0.5%, 1%, and 1.5% by weight of patchouli oil and stirred with a rotation speed of 300 rpm for 30 minutes, 1 hour, and 1.5 hours. CaO 1% of oil is poured into the solution and stirred again for 2 hours with a rotation speed of 500 rpm. The mixture is allowed to stand overnight until a layer of

patchouli oil is formed at the top and a precipitate at the bottom which will then be separated by filtration. The treatment which is a combination of the level of concentration of each chelating agent and the time of stirring is planned in a completely random factorial with 3 repetitions. The purified oil is examined based on Indonesian National Standard (SNI 06-2385-2006). Oil clarity is in Transmittance percentage (%T).

Results and Discussion

Preliminary analysis is carried out to determine the physicochemical properties of patchouli oil before purifying. It is compared with physicochemical properties after purifying and standard SNI 06-2385-2006. Table 1 describes the physicochemical characteristics of patchouli oil before purification.

Table 1 - Physicochemical characteristics of patchouli oil before purification

Characteristics	Standard based on SNI 06-2385-2006	value
Color - Visual - Transmisi,%	Yellowish to dark brown	Dark brown 2.8±0.13
Specific density (25°C)	0.943-0.983	0.988±0.15
refractive index (ND ²⁰)	1.504-1.514	1.512±0.12
Acid value	Max. 5.0	8.04±1.86
Iron content, Fe (ppm)	-	328.04± .98
Patchouli alcohol (%)	Min.30	28.54%

Table 1 shows the physicochemical characteristics of patchouli oil based on standard, before and after purifying. Based on the results of the preliminary analysis of both the physical and chemical characteristics of patchouli oil from local community farmers, some characteristics such as specific density, acid value, oleoresin oil content, and patchouli alcohol do not meet standard SNI 06-2385-2006. Patchouli oil is one of the important characteristics that are below standard.

The dark colour of the oil causes the level the clarity of the oil to be very low, and this is due to its high iron content [[13], [14]] argues that contamination by iron occurs during the distillation process which uses a metal iron distiller. Iron ions

can stimulate oxidation reactions in conjugated double bonds found in patchouli alcohol compounds in patchouli oil to produce colour-forming chromophore compounds from groups $>C=C<$ or $>C=C$ [15]. Patchouli alcohol compound is the main component in patchouli oil. Dark colours cause low clarity. Colour is a parameter that is easily visible, therefore greatly affecting consumer acceptance and can reduce quality [[16], [17]].

In the purification process, the effect of concentration (EDTA, and citric acid) on the patchouli oil clarity is presented in Figure 1.

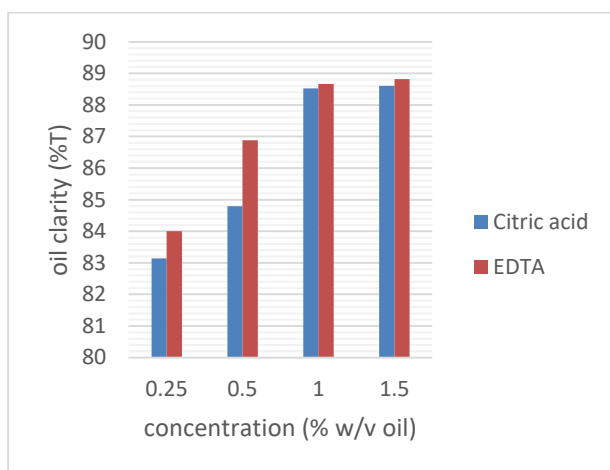


Figure 1 – Effect of concentration on the oil clarity

Findings showed that chelating agent concentration has a significant effect on oil clarity. The oil clarity could be 88.52%T by using 1% citric acid and 88.61%T by using 1.5% citric acid as a chelating agent. The significant oil clarity is increased from a concentration of 0.5% to 1% citric acid (w/v). Meanwhile if using EDTA as a chelating agent, the oil clarity could be 88.67%T by using 1% and 88.82%T by using 1.5% of concentrations. The significant oil clarity is also increased from a concentration of 0.5% to 1% EDTA (w/v). Citric acid has nearly the performance as EDTA in concentrations of 1% and 1.5%.

Fe content in patchouli oil is the cause of the dark colour and low quality of the oil. Thus, the effect of the chelating agent, concentration, and time of stirring on Fe content is presented in Table 2.

Findings showed that the interaction of concentration and stirring time has a significant effect on Fe content. The higher concentrations and the longer the stirring time, the less Fe content. The least Fe content is by using a 1.5% chelating agent

during 90 minutes time of stirring. However, the result of the 1% chelating agent during 60 minutes is not different significantly when compared to 1.5% chelating agent during 90 minutes.

Citric acid has 3 pairs of free electrons meanwhile EDTA has 6 pairs of free electrons from C=O and N atoms. Thus, EDTA has better performance. However, citric acid has almost similar performance to EDTA. The further result before and after purification is presented in Table 3.

Table 2 – Interaction between the chelating agent, concentration, time of stirring and Fe content

Chelating agent	Concentration (%)	Stirring time (minutes)	Fe content (ppm)
EDTA	0.25	30	19.65
		60	17.28
		90	15.34
	0.50	30	18.35
		60	14.13
		90	13.04
	1	30	14.05
		60	13.04
		90	10.34
	1.5	30	11.57
		60	9.05
		90	8.87
Citric acid	0.25	30	19.85
		60	18.08
		90	16.24
	0.50	30	18.05
		60	13.83
		90	13.04
	1	30	15.05
		60	12.54
		90	11.04
	1.5	30	11.92
		60	9.25
		90	9.30

Table 3 showed that after purifying, specific density (25°C), refractive index (ND²⁰), acid value, iron content (ppm), oleoresin oil content, and patchouli alcohol meet the standard. Citric acid as chelating agent has three carboxyl functional group

(-COOH) by setting its concentration and stirring time, H atoms occurred deprotonation [18]. Ion H⁺ replaces Fe²⁺. Thus, it could decrease Fe content and increase clarity of patchouli oil [19].

Patchouli oil from local community farmers is dark brown due to it contains colloidal Fe. The addition of citric acid as a chelating agent reacts with Fe metal to form chelate complex ions [20].

Table 3 - Physicochemical characteristics of patchouli oil before and after purification by using citric acid as a chelating agent

Characteristics	Standard based on SNI 06-2385-2006	Before purification	After purification
Color Visual Transmission, %	Yellowish to dark brown	Dark brown 2.8±0.13	Light yellow 57.3±0.16
Specific density (25°C)	0.943-0.983	0.988±0.15	0.963±0.26
refractive index (ND ²⁰)	1.504-1.514	1.512±0.12	1.509±0.31
Acid value	Max. 5.0	8.04±1.86	0.12±1.63
Iron content, Fe (ppm)	-	328.04±.98	9.3±4.12
Patchouli alcohol (%)	Min.30	28.54%	34.84%

A chelating agent forms a complex salt that binds to the iron occurred in the oil. The chelating agent in forming the complex salt lumps is supported by calcium oxide which also neutralizes the acidity in the oil [21]. The decrease in Fe content significantly from 328.04±3.98 ppm to 9.3±4.12 cause a significant change in the colour of the oil. Patchouli oil changes colour to clear yellow and is the preferred colour in the market.

Conclusion

Purification of patchouli oil by using citric acid as a chelating agent can significantly improve the quality of the oil value. Its performance is almost similar to EDTA in certain conditions. This refining can improve the characteristics of the oil from the aspect of colour, patchouli alcohol, acid number, iron content (ppm) and oleoresin oil content (%). Citric acid can reduce iron content by much as 97.16%. Therefore, citric acid has promising potential to be used in patchouli oil refining for local community farmers.

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Conflicts of interest. On behalf of all authors, the corresponding author states that there is no conflict of interest.

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Комплексометрия бойынша пачули майын тазартуда лимон қышқылын қолдану арқылы темірді жою

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ТҮЙІН

Бұл зерттеу пачули майындағы коллоидты қоспаларды азайту арқылы оның сапасын жақсарту үшін лимон қышқылдарын потенциалды хелатизатор ретінде пайдалануды зерттеуге бағытталған. Зерттеулер түсті, меншікті тығыздықты, сыну көрсеткішін, қышқылдық мәнін, темір құрамын, олеорезин майының мазмұнын және пачули спиртінің қамтиды. Факторлық дизайнмен толық рандомизацияланған дизайн екі фактормен пайдаланылады және 3 рет қайталануы. Факторлар (1) лимон қышқылының концентрациясы 0,25%, 0,5%, 1,0% және 1,50% (салм/көлем) тұрады, (2) араластыру уақыты 30, 60 және 90 минут. Одан әрі лимон қышқылымен тазартылған май этилендиамин тетраацетаты (EDTA) арқылы тазартылған маймен салыстырылды. Нәтижелер пачули майының сапасына хелаттандырушы заттардың концентрациясы мен араластыру уақыты әсер ететінін көрсетеді. Хелатизатордың концентрациясы неғұрлым жоғары болса және араластыру уақыты неғұрлым көп болса, тазартылған пачули майының түсі, меншікті тығыздығы, сыну көрсеткіші, қышқылдық мәні және темір мөлшері бойынша сапасы соғұрлым жақсы болады. Нәтижелер сонымен қатар лимон қышқылы EDTA-мен бірдей сипаттамаларға ие екенін көрсетті. Пачули майындағы негізгі компоненттер (пачули спирті және олеорезин майы) өзгермейді. Лимон қышқылын қолдану арқылы тазартылған пачули майы Индонезия ұлттық стандартының (SNI) талаптарына сәйкес келеді, сондықтан лимон қышқылы потенциалды хелат агенттерінің бірі болып табылады.

Түйін сөздер: Тазарту процесі, эфир майы, хелат агенті, пачули майы, темір құрамы

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Удаление железа с помощью лимонной кислоты при очистке масла пачули по комплексометрии

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АННОТАЦИЯ

Это исследование направлено на изучение использования лимонной кислоты в качестве потенциального хелатирующего агента для улучшения качества масла пачули за счет уменьшения количества коллоидных примесей. Исследования включают цвет, удельный вес, показатель преломления, кислотное число, содержание железа, содержание олеорезинового масла и спирт пачули. Полностью рандомизированный план с факторным планом используется с двумя факторами и повторяется 3 раза. Факторы включают (1) концентрацию лимонной кислоты 0,25%, 0,5%, 1,0% и 1,50% (вес/объем), (2) время перемешивания 30, 60 и 90 минут. Кроме того, масло, рафинированное лимонной кислотой, сравнивали с маслом, рафинированным этилендиаминтетраацетатом (ЭДТА). Результаты показывают, что на качество масла пачули влияет концентрация хелатирующих агентов и время смешивания. Чем выше концентрация хелатора и дольше время смешивания, тем лучше качество рафинированного масла пачули с точки зрения цвета, удельного веса, показателя преломления, кислотного числа и содержания железа. Результаты также показали, что лимонная кислота имеет те же характеристики, что и ЭДТА. Основные компоненты масла пачули (спирт пачули и масло олеорезина) не изменились. Масло пачули, рафинированное с использованием лимонной кислоты, соответствует требованиям Национального стандарта Индонезии (SNI), поэтому лимонная кислота является одним из потенциальных хелатирующих агентов.

	Ключевые слова: процесс очистки, эфирное масло, хелатирующий агент, масло пачули, содержание железа.
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