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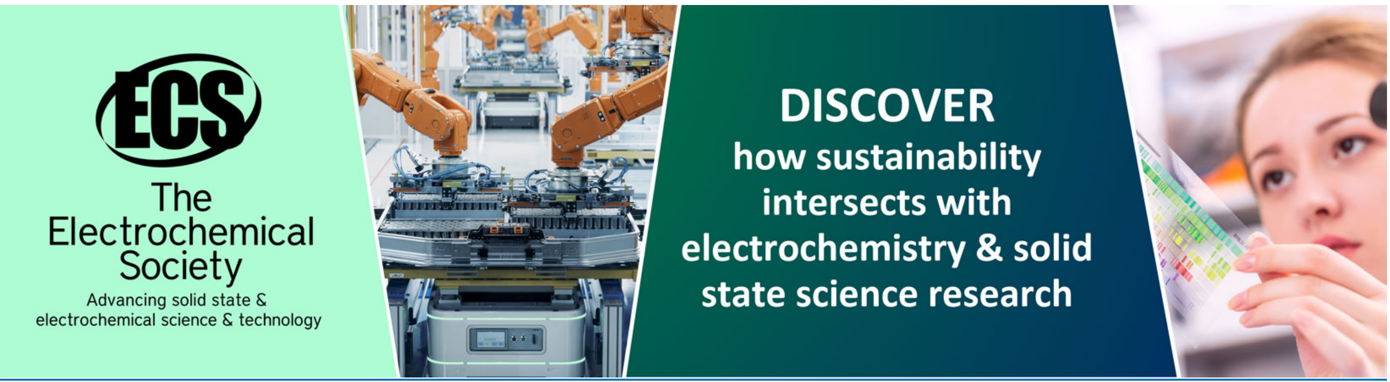

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Optimization and Characterization of Cinnamon Leaves (*Cinnamomum burmannii*) Oleoresin

L.U. Khasanah, Kawiji, P. Prasetyawan, R. Utami, W. Atmaka, G.J. Manuhara, A.P. Sanjaya
Food Science and Technology, Sebelas Maret University, Surakarta

E-mail : liaumikhasanah@yahoo.co.id

Abstract. This research aimed to determine the optimum yield condition on cinnamon leaves oleoresin production at various temperature and contact time during maceration and to find out the characteristics of cinnamon leaves oleoresin such as active compound, cinnamon leaves oil content, and solvent residue levels at optimum yield. This research used the variations of extraction temperature (70, 75 and 80°C) and extraction time (4, 5 and 6 h). Based on Response Surface Methodology (RSM), the equation of cinnamon leaves oleoresin sample optimization as follow: Y = 13 – 1.0167X1- 0.2833X2- 0.6833X12- 0.5833X22- 0.3250 X1X2. The optimum yield of cinnamon leaves oleoresin (13.3790%) was obtained at 77.754°C for 4.9185 h. The characteristics of cinnamon leaves oleoresin that showed the optimum yield were 59.56% eugenol level, 9.50% cinnamon leaves oil content and 22700 ppm solvent residue level.

1. Introduction

Cinnamon is an important export commodity of Indonesia. The product is exported to the United States of America (46 %), the Netherlands (11 %), Germany (4 %), and Singapore (4 %). In 2015, the most exported cinnamon product (95%) was cinnamon bark quill and the other was cinnamon powder [1]. Indonesian cinnamon production reached 45 % of the world's total production, and the volume of Indonesian cinnamon export was the largest in the world (26%) [2]. The main product of cinnamon tree is cinnamon bark. The by-products of cinnamon tree are branches and leaves. However, all parts of the cinnamon tree contain essential oil and oleoresin that mainly found in the bark, stems, and leaves, and only a small portion is found in the wood part [3].

Besides from the cinnamon bark, oil may be distilled from the cinnamon leaves. The oil of C. *zeylanicum* leaf contains eugenol as the main component (80 - 90%), whereas the main content of the oil of C. *burmanii* leaf and C. *cassia* leaf is cinnamaldehyde that same with the content of the bark oil [4].

The main chemical components of the essential oil of cinnamon leaves (*Cinnamomum burmannii*) are cinnamicaldehyde (63.61 %) and eucalyptol (17.27 %) [5]. *Cinnamomum zeylanicum* oleoresin contains an active compound of eugenol, as much as 87.2% [6]. *Cinnamomum tamalla* oleoresin contains an active compound of eugenol, as much as 17.3 % [7].

Oleoresin is a mixture of oil and resin or gum, which is obtained from the extraction, evaporation, and standardization of essential oil (essential oil and non-volatile components of the herbs and spices). The oleoresin characteristics usually are viscous liquid, paste, or solid. Many researchers have studied various kinds of oleoresin such as *Curcuma zanthorrhiza* oleoresin, galangal

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oleoresin, and cinnamon oleoresin. Oleoresin has many advantages. It is more popular and hygienic because its aroma and flavor are stronger than its raw material; it is stable during storage; and it needs less space for storage [8]. Oleoresin has the following characteristics: its tends to have antimicrobial characteristics, it is more hygienic, and it contains natural anti-oxidants, it is enzyme-free, it has a longer shelf life, it allows more economical storage, it has a lighter weight for transport, and it is free from the danger of mildew on spices [9].

Extraction is an important phase of oleoresin production. One-phase extraction (direct extraction) is a direct process of oleoresin production by adding solvent which produces pure oleoresin with the comparison of essential oil and resin as genuinely found in the raw materials. A two-phase extraction is a process of oleoresin-production through distillation to obtain the essential oil, and follow by oleoresin extraction which produces oleoresin that contain essential oil and resin in a certain ratio [8].The previous research stated that at the pilot plant scale, oleoresin produced by one-phase extraction with the ethanol solvent was 35% while oleoresin produced by two-phase extraction was 31.25% [8].

The extraction process is affected by several factors such as size of the raw material, solvent, duration of the extraction process, and extraction temperature [10]. In this research, optimization of extraction temperature and extraction time was performed to investigate the optimum conditions that resulting the highest oleoresin yield.

2. Experimental

The *Cinnamomum burmannii* leaves were chopped (1 cm) and extracted through maceration method. The extraction varied at several temperaturs (70, 75, and 80°C) and contact time (4, 5, and 6h). The solvent was ethanol (70%). The ratio between the material and the solvent was 1:6. Filtering process was performed to separate the dregs and filtrates. The filtrates were then separated with a *rotary vacuum evaporator* to obtain the *Cinnamomum burmannii* leaf oleoresin. The data of oleoresin yields obtained from the treatment were analyzed using the computer software of Matlab Version 7.0 to determine the optimum conditions. The oleoresin of the optimum yields was analyzed to determine the characteristics such as eugenol content using GCMS (SHIMADZU, column: Rastek RX-i-1MS, inert gas: Helium), essential oil content with the distillation method [11], and residual solvent content using GC.

3. Results and Discussion

3.1. Cinnamomum burmannii leaf Oleoresin Yield Optimization

Table 1 showed the *Cinnamomum burmannii* leaf oleoresin yields. The yields were analyzed by using the *Response Surface Methodology* (RSM) and Matlab 7.0 to investigate the optimum yields. The RSM is a statistical technique to find the optimum conditions of a response affected by complex variables. The original concept of this method was introduced by Box and Wilson in 1951 [12].

In this research, the RSM was employed to determine the optimum conditions that would result in the higher *Cinnamomum burmannii* leaf oleoresin yields (% b/b) with two factors which are extraction temperatures (X_1) of 70, 75, and 80 °C, and extraction time (X_2) of 4, 5, and 6h. Polynomial quadratic regression model was made to predict the optimum Y variable (oleoresin yields in the percentage (%) unit). The mathematical model of the formed response is as follows: Ŷ = b0 + b1x1 + ... + bnxn + b11x12 + ... + bnnxn2 +b12x1x2 + ... + bn-1.nxn-1xn

The result of the *Cinnamomum burmannii* leaf oleoresin yield data optimization with the computer software and Mathlab 7.0 and the factorial experiment design resulted the following response function: Y = 13 – 1.0167X1- 0.2833X2- 0.6833X12- 0.5833X22- 0.3250 X1X2.

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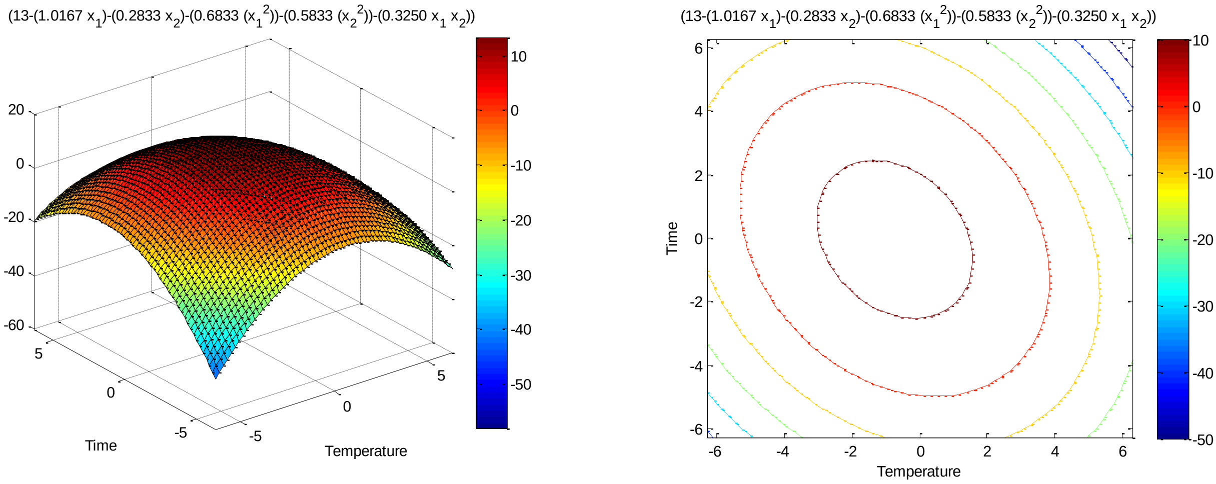
Table 1. The *Cinnamomum burmannii* Leaf Oleoresin Yields

Run	X_1 (Extraction Temperature)	X_2 (Extraction Time)	Yield (%)
1	70 (-1)	4 (-1)	11.5
2	70 (-1)	5 (0)	13.3
3	70 (-1)	6 (1)	12.8
4	75 (0)	4 (-1)	13.3
5	75 (0)	5 (0)	12.8
6	75 (0)	6 (1)	11.5
7	80 (1)	4 (-1)	10.9
8	80 (1)	5 (0)	10.6
9	80 (1)	6 (1)	10.4

Figure 1 showed the optimum response of the effects of extraction temperatures (X1) and time (X2) on the *Cinnamomum burmannii* leaf oleoresin yields at the stationary points of -0.7349 and 0.0381. Of these points, the extraction temperature of 77.754ºC and the extraction time of 4.9185 h gave the optimum *Cinnamomum burmannii* leaf oleoresin yield (13.3790%).



A B

Figure 1. Response Surface Methodology (A) Response surface (B) Response contour

The increase of extraction temperature caused the molecular motion of ethanol as a solvent to be faster but random. In addition, the increase of temperature caused the pores of solids to expand so that the ethanol diffused and entered the materials through the pores and then dissolved the oleoresin. As a result, the oleoresin was interacting greatly and this caused the solute mass transfer from the feed solids to the solvents to be greater [13].

Figure 1 showed that when the temperature was higher and the time was longer, the extraction yield increased. However, when the temperature and time exceeded the optimum points (77.754ºC and 4.9185h), the extraction process became ineffective. The temperature above the solvent's boiling point and the long time of extraction caused the solvents to be wasted. In addition, the volatile components of oleoresin were brought by the evaporated solvents so that the yields decreased. The extraction could be promptly performed at a high temperature. However, in the oleoresin extraction, such a treatment could cause damage to several components found in the spices [14]. The longer the extraction time, the higher the yields obtained until the six-hour limit as the contact time between the materials and the solvents was greater. Above the six-hour contact time, the yields decreased as the solution had already reached the saturation point [15].

3.2. Cinnamomum burmannii leaf Oleoresin Quality Characteristics at Optimum Condition

The optimum *Cinnamomum burmannii* leaf oleoresin yield was obtained at the temperature of 77.754°C and extraction time of 4.9185 h. This treatment became the basis for the production of *Cinnamomum burmannii* leaf oleoresin whose quality characteristics would be tested in terms of yield, active compound content, essential oil content, and residual solvent content. The optimum *Cinnamomum burmannii* leaf oleoresin was then verified. The extraction was performed at 78°C for 4h and 55 mins. The *Cinnamomum burmannii* leaf oleoresin obtained from the verified condition showed the characteristics displayed in Table 2.

Table 2. Quality Characteristics of *Cinnamomum burmannii* leaf Oleoresins

Parameter	Research Result	References
Yield	12.6 %	6.9%[a] – 23.02%[b]
Active compound content	Eugenol (59,56%)	Eugenol (17.3%[c] - 87.2%[a])
Essential oil content	9.5 %	15%[d]
Residual solvent content	22700 ppm	5000 ppm[e]

Notes: [a] [6]; [b] [16]; [c] [7]; [d] [17]; [e] [18]

3.2.1. Yield

The yield of *Cinnamomum burmannii* leaf oleoresin that was extracted at 78°C for 4h and 55 mins was 12.6%. This result was greater than that of *Cinnamomum zeylanicum* oleoresin of 6.9% [6]. The *Cinnamomum zeylanicum* leaf powder was extracted with soxhlet for 2h using the acetone solvent [6]. The *Cinnamomum burmanii* leaf oleoresin yield of this research was smaller than the *Cinnamomum cassia* yield that extracted by using supercritical fluid extraction (23.02%) [16]. The difference between the yields was due to the difference in chemical compositions of different plant materials or the soil characteristics and agro-climatic conditions, including other factors such as the effectiveness of the solvent in dissolving the endogen compounds. There were several factors that affected the yield and quality of oleoresin, such as variety, condition and size of the spice powder, solvent selection, extraction conditions, and the solvent evaporation process [19].

3.2.2. Active Compound Content

Figure 2 showed the result of GCMS analysis. Table 3 showed the chemical components detected in the *Cinnamomum burmannii* leaf oleoresin such as eugenol with the relative content of 59.56%, trans-caryophyllene of 3.96%, 1,4,8-Cycloundecatriene of 1.72%, Glycerol triacetate of 2.02%, dodecane, 4,6-dimethyl of 1.42%, Benzyl benzoate of 7.78%, trans-2-tridicenal of 1.51%, tetradecanamide of 2.41%, and oelic acid amide 19.58%.

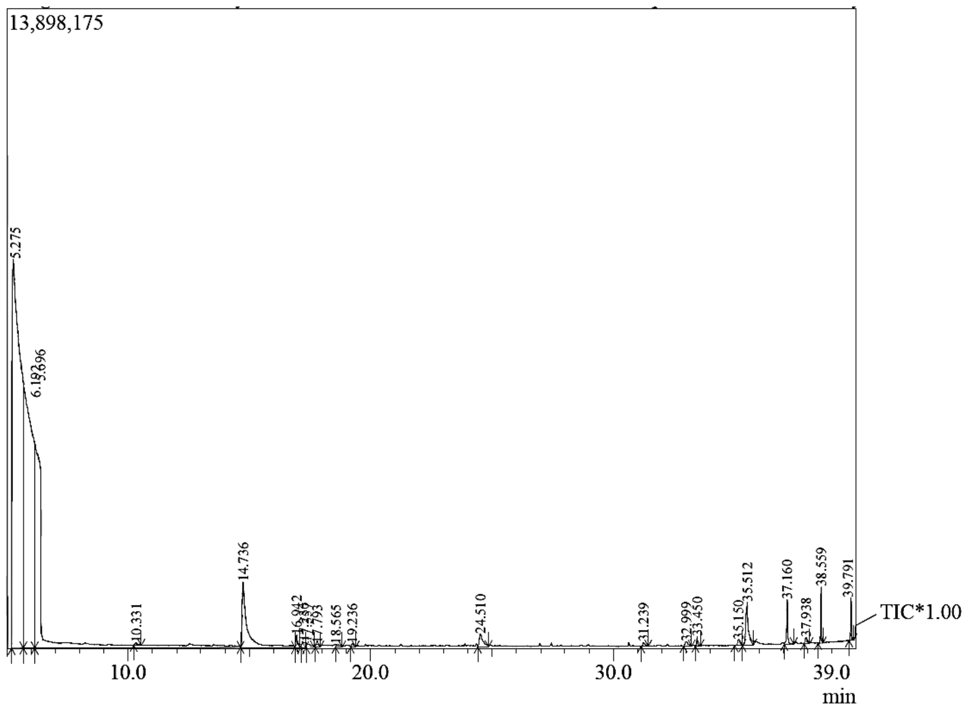


Figure 2. GCMS Spectra

Table 3. The Chemical Components in The *Cinnamomum Burmannii* Leaf Oleoresin

Peak	R.Time (min)	Area	Area (%)	Compound Name
1	14.736	20451277	53.99	Eugenol
2	16.942	1501091	3.96	trans-Caryophyllene
3	17.236	1005380	2.65	Eugenol
4	17.357	1104457	2.91	Eugenol
5	17.793	651937	1.72	1,4,8-Cycloundecatriene
6	18.565	765686	2.02	Glycerol triacetate
7	19.236	540143	1.42	Dodecane, 4,6-dimethyl- (CAS)
8	24.510	2949177	7.78	Benzyl benzoate
9	31.239	574605	1.51	Trans-2-Tridecenal
10	32.999	914525	2.41	Tetradecanamide
11	35.512	7420131	19.58	Oelic Acid Amide

The dominating active compound of the *Cinnamomum burmannii* leaf oleoresin was eugenol with the relative content of 59.56 %. Previous research state that eugenol content of the *Cinnamomum zeylanicum* leaf oleoresin was 87.2% [6] while other reported that the eugenol content of *Cinnamomum tamala* leaf oleoresin was 17.3% [7]. The eugenol content of *Cinnamomum burnanii* leaf oleoresin was higher than that of *Cinnamomum tamala* leaf oleoresin but smaller than that of *Cinnamomum zeylanicum* leaf oleoresin. The extraction of *Cinnamomum zeylanicum* leaf oleoresin used acetone solvent by using a soxhlet extractor for 2h, while the extraction of *Cinnamomum tamala* leaf oleoresin used the CCl_4 solvent for 3h. The different solvents and extraction methods affected the active compound content of oleoresin. In addition, the eugenol content of the materials was also influenced by many factors such as rainfall, altitude, harvest time, drying method, and harvest-produce storing [20].

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Eugenol is slightly soluble in water but easily soluble in organic solvents. In addition to its use as an aroma enhancer, eugenol is also antiseptic, and therefore it is used in soaps, detergents, toothpastes, perfumes, and pharmaceutical products. Eugenol also has antimicrobial properties so it can be used as a natural preservative.

3.2.3. Essential Oil Content of Cinnamomum burmannii Leaf Oleoresin

Essential oil content is one of the most frequently tested qualities of oleoresin of spices because it is used particularly due to its volatile oil that very much determines its flavor. The greater the essential content found in the oleoresin, the better its quality is. In terms of quality standard, the essential oil content applicable in international trade must be greater than 15% [21]. If compared with the distillation-generated essential oil, the essential oil of oleoresin has weaker aroma and smell, but it stays longer and diffuses better [19].

The essential oil content of *Cinnamomum burmannii* leaf oleoresin obtained in this research was 9.50%, which was less than 15% so it did not fulfill the standard of international trade. The low content was probably due to the evaporation process which used the temperature of 80°C, which may have caused the essential oil to evaporate together with the solvent and enter the distillation round bottomed three-neck flask. Evaporation at a high temperature and over a long period could damage the essential oil components found in the oleoresin. In addition, many components of the oleoresin easily evaporated and were brought by the evaporated solvent [19].

Previous research reported that the essential oil content of the *Cinnamomum burmanii* extracted with ethanol solvent ranged from 7.28 - 7.72% [22]. This yield did not fulfill the international trade standard. The other research showed that the essential oil content of the cinnamon bark oleoresin ranged from 1.09% to 2.79% [23]. The longer the extraction time and the higher the temperature, the greater the probability that the essential oil will evaporate.

3.2.4. Residual solvent content

The solvent is an important factor in oleoresin extraction. The solvent used in the extraction process must easily evaporate so that during the evaporation it can evaporate thoroughly or partly. The volume of residual solvent in oleoresin can influence its quality and flavor.

Analysis of residual solvent in oleoresin is useful for its further application in industry and pharmacy, which must be in compliance with the terms and conditions of the FDA on organic residual solvent. If the residual solvent exceeds the determined threshold, the evaporation process must be optimized as it will influence its application in food and pharmacy [22]. Ethanol has bad impacts and effects on human health. It may disrupt liver functions, damages the hearts increases breast cancer risk, and its continued use can eventually be fatal.

The standard used in determining the residual solvent refers to the standards enacted by the FDA (*Food and Drugs Administration*). Based on the result of testing with GC, the residual solvent of *Cinnamomum burmannii* leaf oleoresin reached 2.27% (22700 ppm). According to the standard of the FDA, the threshold of residual solvent in food and drug products is 5000 ppm.

Thus, the result of research showed that the residual solvent of *Cinnamomum burmannii* leaf oleoresin was far above the threshold determined by the FDA. This was because the evaporation process did not work perfectly, and some solvent was trapped in the oleoresin. In addition, the imperfect performance of vacuum during the evaporation process caused the amount of residual solvent in the *Cinnamomum burmannii* leaf oleoresin to be large. The result of research on oil and oleoresin extraction from the cinnamon bark (*Cinnamomum burmanii*) showed a high percentage of ethanol residual solvent (3.18%) [24]. The residual solvent of cassiavera (*Cinnamomum burmanii* BL) oleoresin extracted with ethanol solvent ranged from 5.66% to 11.98% [22]. The residual was fairly high due to the distillation conditions and evaporation of the solvent in the rotary vacuum evaporator. The column distillation and column packing distillation can be used to as methods to reduce residual solvent of *Cinnamomum burmannii* oleoresin [25].

4. Conclusion

The optimum yield of *Cinnamomum burmannii* leaf oleoresins was 13.3790%, which was obtained at an extraction temperature of 77.754°C and with an extraction time of 4.9185 h. The quality characteristics of *Cinnamomum burmannii* leaf oleoresins at the optimum yield condition were 59.56% eugenol level, 9.50% cinnamon leaves oil content and 22700 ppm solvent residue level.

Acknowledgments

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References

[1] Towaha J dan Indriati G 2008 *Warta Penelitian dan Pengembangan Tanaman Industri* **14** 14-16
[2] Zaki I 2011 *Tiga Klon Unggulan Kayu Manis dan Teknik Perbanyakan Secara Vegetatif* (Surabaya: Balai Besar Perbenihan dan Proteksi Tanaman Perkebunan)
[3] Zarfis 2008 *Buletin BIPD*. **16**
[4] Leung A 1980 *Encyclopedia of Natural Ingredients* (New York: John Wiley & Sons)
[5] Tampubolon D F 2011 *Isolasi dan Analisi Komponen Minyak Atsiri Daun Kayu Manis (Cinnamomum burmanii) dengan GCMS* (Medan)
[6] Singh G 2007 *Food and Chem. Toxicol.* **45** 1650-61
[7] Kapoor I P S 2009 *Nat. Product Radiance* **8** 106-116
[8] Sari E 2010 *Jurnal Ekotrans* **10** 108-117
[9] Pruthi J S 1980 *Spices and Condiments, Chemistry, Microbiology, Technology* (New York: Academic Press)
[10] Anam C 2010 *Jurnal Pertanian MAPETA* **12** 71-144
[11] Sundari E 2002 *Pengambilan Minyak Atsiri dan Oleoresin dari Kulit Kayu Manis* (Bandung: Thesis)
[12] Shieh C J 2000 *J. Agric. Food Chem*. **48** 1124-28
[13] Treyball R E 1981 *Mass-Transfer Operations* (New York: Mc Graw-Hill)
[14] Sujarwadi E T 1996 *Kajian Jumlah Pelarut dan Lama Ekstraksi Rimpang Kencur terhadap Rendemen dan Mutu Oleoresin Kencur* (Bogor: Thesis)
[15] Suryandari S 1981 *Pengambilan Oleoresin Jahe dengan cara Solvent extraction* (Bogor: Balai Besar Industri Hasil Perkebunan)
[16] Hong Yang C 2012 *Molecules* **17** 7294-7304
[17] Nurdjanah N 2007 *Teknologi Pengolahan Pala* (Bogor: Balai Besar Penelitian Dan Pengembangan Pascapanen Pertanian)
[18] Food and Drug Administration 2012 *Guidance for Industry Q3C-Tables and List* (US: Department of Health and Human Services)
[19] Lestari W E W 2006 *Pengaruh Nisbah Rimpang dengan Pelarut dan Lama Ekstraksi Terhadap Mutu Oleoresin Jahe Merah (Zingiber officinale var.* rubrum) (Bogor: Thesis)
[20] Nurdjanah N 1988 Buletin Penelitian Tanaman Rempah dan Obat **3** 72-75
[21] Sutianik 1999 *Pengaruh Suhu Pengeringan dan Ukuran Bahan Terhadap Rendemen Dan Mutu Oleoresin Jahe (Zingiber officinale*, Roscoe) (Bogor: Thesis)
[22] Yusmeiarti S and Syarif R 2007 Buletin BIPD **15**
[23] Sulaswaty A 2001 *Kajian Awal Hasil Ekstraksi Minyak dan Oleoresin Dari Kulit Kayu Manis (Cinnamomum burmanii* blume) (Yogyakarta: Proceeding)
[24] Solehudin M 2001 *Ekstraksi Minyak dan Oleoresin dari Kulit Kayu Manis (Cinnamomum burmanii)* (Bogor: Thesis)
[25] Khasanah L U, Anandhito B K, Utami R, Kusumaningrum P, Waningsih, M 2014 *Jurnal Teknologi Industri Pertanian* **24(1)** 53-60