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Synthesis of Cardanil Methyl Ether via Etherification Reaction Cardanol Isolated from Cashew Nut Shells (*Anacardium occidentale L.*) with Variations of Molar Methyl Iodide

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ABSTRACT

The synthesis of the Cardanyl methyl ether is carried out through an etherization reaction between Cardanyl seed skin extract (CNSL) isolated from the methyl iodide. Cardanol is isolated with the addition of acetone, $Ca(OH)_2$, NH_4OH 25%, n-hexane: ethyl acetate (98:2), washing with NaOH 2.5%, HCl 5%, aquadest, and Na_2SO4 anhydrate. The yield of the obtained cardanol is 74.39 g (74.39% of the initial weight of the CNSL) and has the following characteristics: acid count 1.2863 mgKOH/g, iodine count 215.6535 g I2/100 g, viscosity 38.7 cP, density 0.8266 g/mL, pH = 6.14. The FT-IR methyl ether Cardanyl spectrum shows an absorption band at 1162.9-1259.8 cm-1 shows a C-O stretching group at a wave number of 1043.6-1148.0 cm^{-1} showing the formation of an asymmetric and symmetrical C-O-C stretching band of the ether compound, the Spectra of the FT - IR methyl ether Cardanyl shows an absorptive band at 1162-1259-125,8 cm^{-1}, showing a C-O stretching Group, at the wave number of 1043.6-1148.0 cm^{-1} indicating the formation, as a result, of an Asymmetric and Symmetric Stretching Group of the ethereal Compound. The Cardanyl methyl ether was synthesized by adding K_2CO_3, the solvent acetone and the iodide-methyl, with a reflux time of 8 hours. As for the parameters carried out in this study, the molar variation of the Cardanyl is the Methyl Iodide (1:1.5, 1:3, and 1:4.5) and yields 3.555 g; 12.084 g; 11.472 g of Cardanyl ether. Methyl ether Cardanyl was obtained in analysis with FT-IR and GC-MS spectroscopic photometers. The results of the analysis of Cardanyl methyl ethers with GC -MS were 63.28%, and the results of analysis of the Cardanyl ether with a variation of the molar ratio Cardanyl: Methyl iodide (1:1.5; 1:3; 1:4.5) with GC-MS obtaining 3.05%, 30.29%, and 26.58%. These results show the maximum percentage of methyl ethers Cardanyl at the reaction conditions of the variation molar ratios Cardanyl iodides (1:3), K_2CO_3 17.3 g, 50 mL of acetone, and 8 hours of reflux time.

Keywords: Cardanol, Cardanyl Methyl Eter, Etherification, Methyl Iodide

ABSTRAK

Sintesis kardanil metil eter dilakukan melalui reaksi eterifikasi antara kardanol hasil isolasi dari ekstrak kulit biji mete (CNSL) dengan metil iodida. Isolasi kardanol dilakukan dengan penambahan Aseton, $Ca(OH)_2$, NH_4OH 25%, n-heksan :etil asetat (98:2), pencucian dengan NaOH 2,5%, HCl 5%, aquadest, serta Na_2SO_4 anhidrat. Rendemen kardanol yang diperoleh sebesar 74,39 g (74,39% dari berat awal CNSL) dan memiliki karakteristik sebagai berikut: bilangan asam 1,2863 mgKOH/g, bilangan iodin 215,6535 g I2/100 g, viskositas 38,7 cP, densitas 0,8266 g/mL, pH = 6,14. Spektrofotometer FT-IR menunjukkan terbentuknya kardanil metil eter yang ditandai dengan adanya penurunan intensitas serapan gugus -OH pada bilangan gelombang sekitar 3000-3400 cm^{-1}. Spektrum FT-IR kardanil metil eter menampilkan pita serapan pada 1162,9-1259,8 cm^{-1} menunjukkan gugus C-O stretching, pada bilangan gelombang 1043,6-1148,0 cm-1 yang menunjukkan terbentuknya gugus C-O-C stretching asymmetrical dan symmetrical daripada senyawa eter, Spektrum FT-IR kardanil metil eter menampilkan pita serapan pada 1162,9-1259,8 cm^{-1} menunjukkan gugus C-O stretching, pada bilangan gelombang 1043,6-1148,0 cm^{-1} yang menunjukkan terbentuknya gugus C-O-C stretching

asymmetrical dan symmetrical daripada senyawa eter, namun terjadi peningkatan intensitas C-O-C pada variasi rasio molar kardanol: metil iodida (1:1,5), hal ini diduga berasal dari adanya gugus -OH yang terdapat pada senyawa lain yang mungkin ikut termetilasi pada saat dilakukan proses sintesis. Kardanil metil eter disintesis melalui penambahan K_2CO_3, pelarut aseton dan metil iodida, dengan waktu refluks 8 jam. Adapun parameter yang dilakukan pada penelitian ini adalah variasi molar kardanol: metil iodida (1:1,5 ; 1:3 ; dan 1:4,5) dan menghasilkan 3,555 g; 12,084 g; 11,472 g kardanil metil eter. Kardanil metil eter yang diperoleh di analisis dengan spektrofotometer FT-IR, dan GC-MS. Hasil analisis kardanol dengan GC-MS dipeorleh sebesar 63,28 %, dan hasil analisis kardanil metil eter dengan variasi rasio molar kardanol: metil iodida (1:1,5 ; 1:3 ; 1:4,5) dengan GC-MS diperoleh sebesar 3,05% , 30,29%, dan 26,58%. Hasil tersebut menunjukkan persentase kardanil metil eter maksimum didapatkan pada kondisi reaksi variasi rasio molar kardanol: metil iodida (1:3), K_2CO_3 17,3 g, 50 mL aseton, dan waktu refluks selama 8 jam.

Keyword: Eterifikasi, Kardanil Metil Eter, Kardanol, Metil Iodida

1. Introduction

The agro-industrial development of Anacardium occidentale can provide greater economic value by processing the yields of mite bulldozers into mite beans and processing mite seed bulls into CNSL (Cashew Nut Shell Liquid). The CNSL compound is a thick brown-coloured fluid extracted from the skin of the pink pepper seed. To date, in Indonesia, the skin is not fully utilized, most of which is still wasted, burned or discarded, causing the production of CNSL to remain very low [1]. CNSL can be reacted to form a wide range of derivatives, including polymer compounds or resins. The biological benefits of CNSl are known as anti-tumour, antioxidant, and antibiotic [2].

Gandhi (2013) used the soxhletation method to obtain CNSL extracts with polar, semi-polar and non-polar solvents in his research. The most abundant amount of CNSL extract produced was with semipolar solvent, namely methyl isobutyl ketone (MIBK) of 8.276 g of CNSL extract. In contrast, the lowest amount of CNSL extraction was with an ethanol solvent of 2.077 g of CNSL extract [3].

CNSL contains four main compounds: anacardic acid, cardanol, cardol, and 2-methyl cardol. The compounds found in CNSL are monohydrate phenols or dihydrates with a side chain of hydrocarbons located at the meta position [4]. The phenolic compounds can also protect against UV-B sunlight and therapeutic agents such as diabetes and cancer [5]. Cardanol is a phenolic compound with a C15 aliphatic ring at the meta position obtained from CNSL isolation.

Cardanol has no stinking smell and can be used on an industrial scale without causing excessive environmental contamination. Cardanol polymerization can be used as phenolic resins such as resins and resins. Cardanol can be modified by reacting with dialkyl sulphate with the help of alkali to produce cardanol dialytic. Another way to produce cardanol ether is to react halogen compounds with CNSL with the aid of alkaline [6].

Cardanol derivatives such as epoxy and polyurethane can be used as anti-corrosive surface coatings, e.g., ethoxy cardanol [7-8]. Cardanyl acrylic can be a reactive lubricant or resin [9]. Cardanyl alkyl ether and linoleate can be used as a thermoset resin [10].

Jia (2017), in his research, has modified the Cardanyl as a mannish base Cardanyl butyl ether to be applied as a substitute for PVC plastic. Cardanyl butyl ether was synthesized using butyl chloride, DMF solvent, and potassium carbonate with reflux method at 120^{o}C for 6 hours [11].

Njuku (2015) in his research used n-hexane solvents to extract CNSL extracts and then modified the cardanol with esterification and etherization processes, each using anhydride acetate acid and methyl iodide pegylation agents. Etherization of the compound cardanol was performed by a simple reflux method for 8 hours and obtained a yield percentage of Cardanyl methyl ether of 52.39 ± 0.01% [12].

Based on the description above, the researchers were interested in synthesizing the Cardanyl methyl ether from an extract of the skin of the pepper seed (*Anacardium occidentale L.*) using methyl iodide as a methylation agent, potassium carbonate as a base and acetone as a solvent through an etherization reaction.

As for the parameters to be studied, the variation of the molar ratio of cardanol: methyl iodide (1:1.5, 1:3, 1:4.5) with a reflux time of 8 hours. The methyl ether Cardanyl obtained was analyzed using Gas Chromatography-Mass Spectra (GC-MS) and function group changes with FT-IR.

2. Materials and Methods

2.1. Equipment

Instruments used in this study include FT-IR spectroscopic photometer, GC-MS spectrophotometer, thermometer, magnetic bar, hotplate stirrer, static and clem, split pipe, neck doughnut 3, dryer, analytical balance, socket tool, oven, universal pH indicator, ball condenser, doughnuts, beaker glass, measuring glass, dripper, rotating dough, rotary evaporator, filament paper, glass pile, drop pipette, mixer rod, spatula, plastic, rubber, pycnometer, Erlenmeyer.

2.2. Materials

The materials used in this study include pink seed skin, acetone, $Ca(OH)_2$, NH_4OH 25%, n-hexane, ethyl acetate, NaOH pellet, HCl 37%, aquadest, Na_2SO_4 anhydrate, K_2CO_3, diethyl ether, $H_2C_2O_4$, KOH, $NA_2S_2O_3$, PP indicator, KI, Chloroform, CH_3I.

2.3. Sample preparation.

The first stage of the seed skin is separated from the peanut butter and then dried at a temperature of 700°C for 12 hours. The next 12 hours are smoothed and blended into powder.

2.4. Mete Seed Skin Liquid Extraction [3].

As much as 100 of fine-tuned peel seeds are inserted into a 17x17 cm sheet of paper and put into Soxhlet. Then, 300 mL of acetone and boiling chips are put into a 500 mL boiling flask. Acetone was heated in the Soxhlet reflux system. The Filtrate was then evaporated using an evaporator device to separate the extract from acetone.

2.5. Cardanol Isolation From Mete Seed Skin Extract [13]

As much as 100 g of mete seed skin extract is poured into a 1000 mL beaker glass, and then 600 mL of acetone. $Ca(OH)_2$ 50 g was slowly added while mixing until dissolved. The mixture is then covered with aluminium foil and heated at 50^{o}C for 3.5 hours. The resultant was then filtered, added with NH_4OH 25% as much as 200 mL, and stirred for 15 minutes. Then partitioned with n-hexane: ethyl acetate with a 98:2 ratio of 3 x 100 mL. Separate the organic layer and wash with a 2.5 % NaOH solution of 200 mL. Then wash with 5% HCl of 100 mL and rinse with a 100 ml aquadest. Added Na_2SO_4 anhydrate to bind the remaining water content. The obtained filter was measured and tested for characteristics such as pH, density, viscosity, acid count, iodine count, and FT-IR and GC-MS characterization.

2.6. Acid count test

A total of 2 g of the sample is inserted into the 250 mL Erlenmeyer. Alcohol 96% is poured into the sample until 50 mL and boiled using a water bath. The boiled mixture was cooled down before adding PP indicators and titrated by KOH 0.1 N until the color of the lymph changed to purple-red.

2.7. Density test

The empty picnometer was weighed three times and recorded its mass. The sample is inserted into the picnometers, weighed three times, and recorded the mass.

2.8. Iodine count test

As much as 0.5 g of sample was weighed, and 20 mL of chloroform and 25 mL of Wij's solution were then shaken until homogeneous. Store in a dark place for 30 minutes at room temperature. Then, 20 mL of 15% KI and 100 mL of distilled water were added. The sample was Titrated with 0.1 N $Na_2S_2O_3$ solution until it was pale yellow. Added 1 mL of amylum indicator into it and titrated again until the blue color disappeared. The same was done for the blank.

2.9. Viscosity test

Liquid paraffin is inserted into a 5 liter beaker glass and heated at 40^{o}C. A thermometer was installed on static and inserted into the beaker glass. The sample was poured into the viscometer until the line marks. The viscometer containing the samples was inserted into the beaker glass in the way the viscometer is hung on

the static. It was sucked to mark the boundary with a pipette balloon and then removed and measured the time of the sample flowing to the bottom line. The result was drawn and repeated three times.

2.10. pH test

The electrode is prepared by rinsing it using aquadest and drying it with fine tissue. Then, the electrodes are immersed into the test sample until the pH meter shows a constant scale reading. The scale readings or figures are recorded on a pH meter screen.

2.11. Synthetic Cardanyl methyl ether

A mount of Cardanyl was inserted into a two-neck flask, acetone and K_2CO_3 anhydrate were added while stirring, and CH_3I slowly was poured. The mixture was refluxed for 8 hours. Aquadest was then poured into the sample. The organic layer is washed with NaOH 2 M, then separated and added Na_2SO_4 anhydrate. The filter was applied to separate the diethyl ether. The same procedure was performed for variations in reflux times of 7 hours and 9 hours. The filter obtained was tested for FT-IR and GC-MS characterization.

2.12. FT-IR analysis (Fourier Transform Infrared Spectroscopy).

The samples to be measured are identified, either atomic or molecular, to determine the chemical bonds in the samples. The infrared rays that acted as a light source were divided into two files; one passed through the sample and the other through the comparison. Then, successively pass the chopper. Once through a prism or grating, the file will fall on the detector and be converted into an electrical signal that the recorder will then record. Next, an amplifier is needed when the generated signal is very weak. The standard used is ASTM E1252. Typical infrared scans are in a range of 650-4000 cm^{-1}.

2.13. GC-MS analysis (Gas Chromatography-Mass Spectroscopy.

The sample is injected into the injector, where the temperature can be regulated. The compounds contained in the sample will evaporate and be carried by the carrier gas to the column for the separation process. Once separated, each component will pass through the ionization chamber and be bombarded by electrons so that ionization occurs. The detector and the mass spectrum will capture the resulting fragments that will be produced. In mass spectrophotometers, only positive ions are detected by the spectrometer and presented as tables or graphs with peak m/z (mass/load).

3. Results and Discussion

3.1. Extracting the Skin of the Pink Pine Seed (Anacardium occidentale L.)

The skin of the chasew seed (*Anacardium occidentale L*.) is from the Road Sisingamangaraja, Harjosari II district, Medan. A hundred g of chasew seed skin was produced by using acetone as a solvent and had a thick brown-coloured solution weighing 38.55 g. 100 g of extracts were obtained from chasew seed paste 260 g, which was extracted in three repetitions using soxhlet. The chasew seed skin produced 38.55 % of the extract, which is higher than other research (33.104%) by Gandhi (2011) [3].

3.2. Cardanool isolation from the skin extract of pink pepper seed

The cardanol obtained from 100 g of the skin extracts of rosé pepper is 74.39 g (74.39% of the initial weight). Based on the spectrum of the test results, the cardanols isolated from the leather extract of brown seed mete have peak absorption characteristics at the wave numbers 3414.2, 3004.2, 2922.2, 1595.3, 1461.1; 1371.7, and 1162.9 cm^{-1} which respectively show the presence of function groups -OH (Vstretching), C-H sp^2 (Vstretching), C-H (Vs) asymmetric (CH_3), C=C aromatic (Vsstretching) C- H sp3 (Vbending), C -H sp3 (V stretching). The FT-IR spectrum of cardanol can be seen in Figure 1 below.

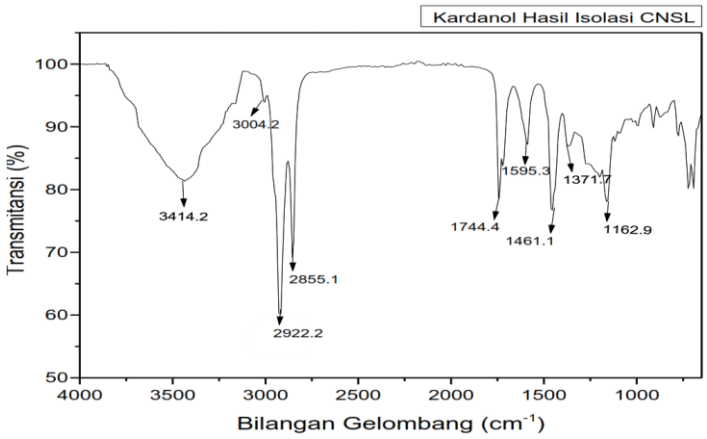

Figure 1: FT-IR Spectrum of Cardanol CNSL Insulation Results

To be more convinced that the compound obtained is cardanol, a GC-MS analysis is carried out.

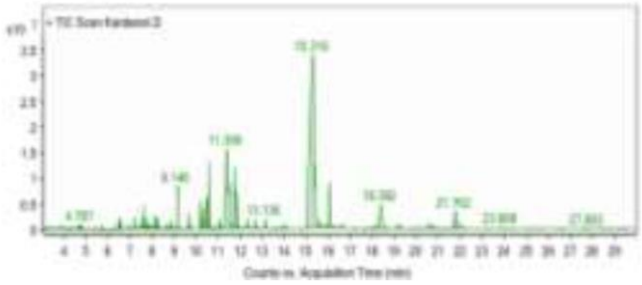

Figure 2: Cardanol Compound Chromatogram CNSL Isolation Results.

Table 1: Compounds resulting from GC-MS Kardanol

RT	Contain (%)	Compound	Compound Formula
7.631	0.63	Butylated Hydroxytoluene	$C_{15}H_{24}O$
9.146	1.27	decane 5 6-bis(2 2-dimethyl propionate)	$C_{20}H_{38}$
10.18	1.4	hexadecan-1-ol	$C_{16}H_{34}O$
10.476	1.23	9 12-octadecadienoyl chloride	$C_{18}H_{31}ClO$
10.605	1.36	hexadecanoic acid methyl ester	$C_{17}H_{34}O$
11.999	14.17	3-(Pentadecylphenol	$C_{21}H_{36}O$
11.75	4.58	9-octadecenoic acid methyl ester	$C_{19}H_{36}O$
11.898	0.9	methyl benzene	$C_6H_5CH_3$
15.316	49.11	3-(Pentadecylphenol	$C_{21}H_{36}O$
16.054	2.97	bis(2-ethylhexyl) phthalate	$C_{24}H_{38}O=$

Based on Figure 2, two main peaks are found. The highest peaks are at retention times 15.316 and 11.399, with percentages of 49,11% and 14,17% respectively. The two peaks indicate the same compound so that the resulting cardanol compounds can be identified as much as 63,28%. Mass spectrum data show the molecular ion peak at m/z 302, where this value corresponds to the cardanol compound's relative molecular weight (Mr) with the molecule formula $C_{21}H_{34}O$ with the relative Molecular Mass 302 g/mol.

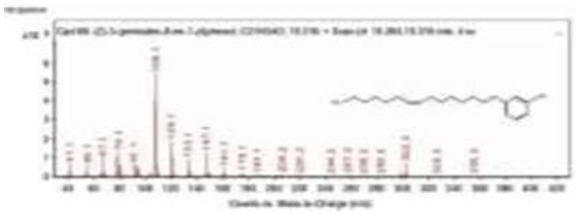

Figure 3: Cardanol mass spectrum at retention time 15.316

3.3. Cardanol Characterization Isolation Outcome Rose Pine Seed Leather Extract

This is the result of the characterization of the compound Cardanol resulting from the isolation of the skin extract of the cardanol seed:

Table 2: Cardanool Characterization Results Isolation Results Skin Extract of Pink Pine Seed

Parameter	Cardanool Standard	Research Result
Acid number	Max 5 mg KOH/g	1.2563 mg KOH/g
Iodine number	Min 210 g I_2/100g	215.6535 g I_2/100g
Viscocity	40-60 Cp	38.7 Cp
Density	0.93-0.95 g/mL	0.8266 g/mL
pH	5.5-6.7	6.14

From the data obtained, the acid count, the iodine count, and the pH have already met the applicable prescribed standards. In contrast, the viscosity and density do not meet the prescriptive standards. (Risfaheri dkk, 2004). The low-density value of the cardanol is associated with the abundance of the detergent contained in the cardanol resulting from the isolation of the skin extract of the rosé seed. According to Risfaheri (2004) [14], the heat treatment of cardanol influences type weight or density. Values of density and viscosity relative to straight. The low viscosity is also affected by heat treatment during the cardanol extraction and isolation process, which leads to the loss of hydrogen bonds so that the composing molecules are more rigid, resulting in a lower intermolecular style [15].

3.4. Results of FT-IR Cardanyl Methyl Eter Spectrophotometer Analysis

The Cardanyl methyl ether is the result of an etherization reaction between the Cardanyl from the skin of the brown seed and the methyl iodide in the variation of the molar ratio of Cardanyl iodides (1:1,5; 1:3; 1:4,5), where the results of the synthesis are analyzed using the FT-IR spectroscopic photometer. The methyl ether Cardanyl synthesis results are in Figures 4, 5, and 6.

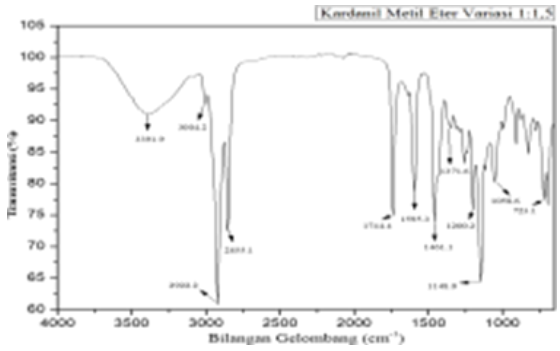

Figure 4: FT-IR spectrum Cardanyl methyl ether variation 1:1.5

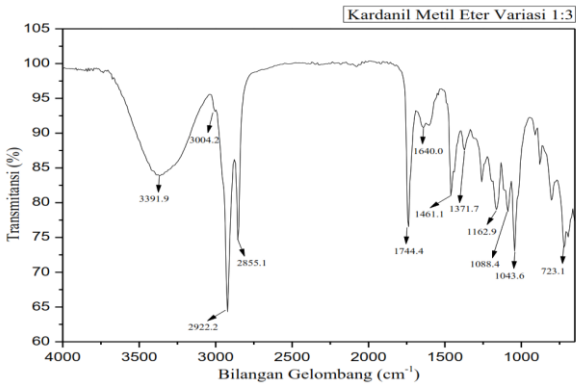

Figure 5: FT-IR spectrum Cardanyl methyl ether variation 1:3

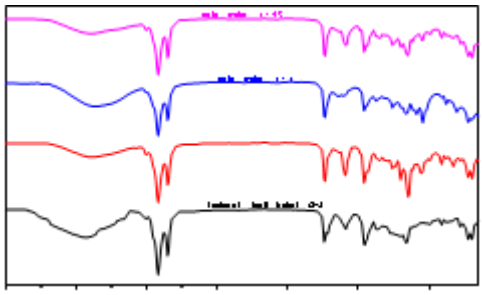

Figure 7: FT-IR spectrum of cardanol and cardanoyl methyl ether

The FT-IR spectrum above indicates that cardanol has a functional group characteristic - OH phenolic compound (stretching vibration) at a wave number of 3000-3400 cm^{-1}, C-H sp3 (stretching vibrations) at the wave count of 2922.2 cm^{-1}, C-H sp^2 (stretching vibrations), at the number of waves of 3004.2 cm^{-1}, C=C (bending vibration), which is a ring bond of aromatic compounds at a wavelength of 1595.3 cm-1 and a wage number of 1461.1 cm^{-1}, which is the bond of C- [12].

According to the FT-IR results of the Cardanyl methyl ether compound, it can be observed from the presence of a decrease in the intensity of the absorption of the group -OH at several waves of about 3000-3400 cm^{-1}. The spectrum of the C-O-C stretching is asymmetric and symmetrical of the compounds of the ether. Still, there is an increase in the intention of C-O-C in the variation of the molar ratio of Cardanyl: iodide (1,5:1). It is believed to be from the existence of the -OH group that is present in other compounds that may have been methylated at the time of the synthesis process.

3.5. GC-MS Cardanil Methyl Eter Spectrophotometer Analysis Results

GC-MS was performed to determine the concentration of the resultant compound concentration Cardanyl methyl ether of kardanol. Chromatogram of Cardanyl ether methyl ether with Cardanyl: methyl iodide variation molar ratio (1:1,5;1:3; 1:4,5) was seen in Figure 8, Figure 9 and Figure 10.

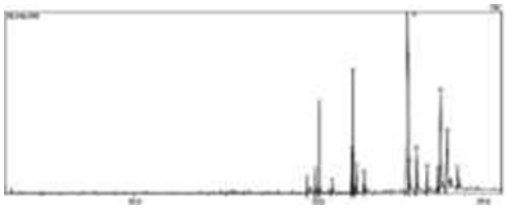

Figure 8: Methyl ether Cardanyl chromatogram variation 1:1.5

Figure 8 shows that cardanyl methyl ether from cardanol molar ratio (1:1.5) has peak retention times of 24.969 minutes and 25.530 minutes with 3.05% Cardanyl methyl ether.

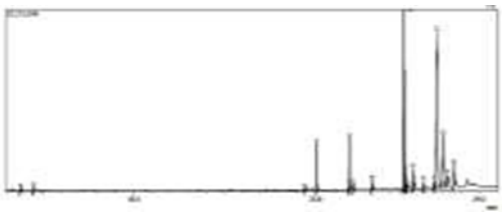

Figure 9: Methyl ether Cardanyl chromatogram variation 1:3

Figure 9 shows that cardanyl methyl ether from cardanol molar ratio (1:3) has peak retention times of 24.862 minutes, 24.962 minutes, and 26.532 minutes with 30.29% Cardanyl methyl ether.

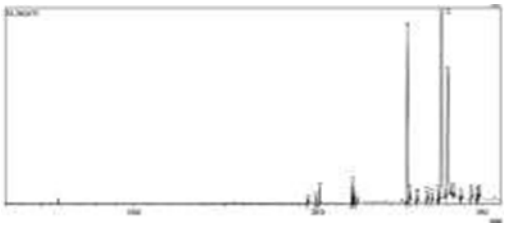

Figure 10: Methyl ether Cardanyl chromatogram variation 1:4.5

Figure 10 shows that cardanyl methyl ether from cardanol molar ratio (1: 1.5) has peak retention times of 24.969 minutes and 25.530 minutes with 3.05 % Cardanyl methyl ether.

4. Conclusion

Cardanyl methyl ether was successfully synthesized from Chasew seed skin extract (Anacardium occidentale L.) through cardanol and methyl iodide reaction. The cardanol obtained from 100 g chasew seed skin extract is 74.39 g (74.39% of the initial weight). It has an acid count of 1.2863 mg KOH/g, iodine count of 215.6535 g I2/100 g, viscosity of 38.7 Cp, density of 0.8266 g/mL and pH 6.14. The ratio of Cardanool toward iodide (1:3) with 8 hours reflux time showed the highest yield percentage of cardanyl methyl ether (30.29%).

5. Acknowledgments

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6. Conflict of Interest

The authors declare no conflicts of interest.

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