

Didekilketon compounds from the leaves of *Artocarpus camansi* Blanco

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Abstract: Research on plant leaves *Artocarpus camansi* (kulu), aims to determine the chemical compounds contained in the hexane extract of the plant leaves. This study begins by isolating the hexane extract, from the leaves of plants *A. camansi*. Subsequently the extracts were characterized by GC-MS, to determine the fragmentation pattern of the compounds contained in leaves of *A. camansi*. Furthermore the hexane extract further fractionated to obtain pure isolates. Pure isolate of the compound as white solid with a melting point of 176-178°C. Characterized to the pure compound with ¹H-NMR, ¹³C-NMR, DEPT and reinforced with HSQC, and HMBC, expressed as didekilketon (C₂₁H₄₂O).

Keywords: *Artocarpus camansi*, pure isolates, didekilketon

Introduction

Artocarpus camansi Blanco, family Moraceae (Mulberry family) is a plant with a height of 10-15 m (33-50 ft) or higher with the main branch along the stem, gummy white on each section. In Indonesia plant *Artocarpus camansi* often referred to as kulu, or kluih, this plant is distributed in the tropics, including the Pacific islands. Plant *Artocarpus camansi*, is very similar to the plant *Artocarpus altilis*, or *A. communis*, so *Artocarpus camansi* often referred to by the name *Artocarpus altilis*, or *A. communis*, and *A. incisa*, but this reference is incorrect, as *Artocarpus camansi* is a different species (Ragone, 2006). However, research on *A. camansi* very less, while research to *A. communis* relatively perfect, both levels of the chemical, as well as its potential as a drug (biological activity).

In this present investigation, we describe the isolation of didekilketon from the hexane extract of the leaves of *Artocarpus camansi* (Rosnani et al, 2013)

Materials and Methods

This research was conducted at the Research Laboratory of the Department of Chemistry Syiah Kuala University in 2011, whereas the spectral characterization was done in Malaysia, GC-MS performed in UPI Bandung.

Plant Material

Leaf of the plant *Artocarpus camansi* (kulu, Aceh name) were collected from Aceh Darussalam. The plant was identified at Department of Biology, University of North Sumatera, Medanense, Medan

Spectroscopic investigation

Melting point was determined by an electrochemical melting point apparatus. Mass spectra were measured with a Shimadzu GC-MS QP 2010 Ultra. The ¹H-NMR (400 MHz) and ¹³C-NMR (125 MHz), HSQC, HMBC, Spectra were recorded on a JEOL in CD₃Cl. Gravity chromatography using Si-gel 60 (Merck), and TLC was performed with silica gel GF₂₅₄ 0.25 mm (Merck). Isolation of secondary metabolites from plant leaves of *Artocarpus camansi* Blanco (Harborne, 1987).

Leaves of *Artocarpus camansi* taken from Banda Aceh, determined in Bogor, leaves of this plant by 1.7kg was macerated with solvent hexane, after evaporated was obtained 45.02g (2.64%) hexane extract. Then 30g of hexane extract separated by gravity column chromatography (KKG) using silica gel 60 (70-230 mesh) as stationary phase, with a mobile phase gradient elution: 100% n-hexane, and n-hexane: ethyl acetate (9:1), long columns used 40cm, with a diameter of 2.5cm, and shelter every 100ml fractions.

Results of fractionation with the KK Gare 59 fractions obtained, fraction groups: A(1-5) as much as 0.029g, B(7-11) by 1.9g, C(12-26) of 1.3g, D(27 -29) of 1.1g, E(30-36) of 2.5g, and F(37-59) as much as 5.6g. D fraction weighing 1.1g, is separated again by gravity chromatography, using hexane solvent and shelter every 10mL fractions, and obtained 12 fractions, namely fractions D1(1-4), 0.02g; Fraction D2(5) ,0.3g; D3(6), 0.02g; D4(7), 0.35g, and D5(8-12), 0.015g. Fraction D2(5), potentially as much as 0.3g of pure isolate is recrystallization with exane and methanol, and washed with exane, yielding pure isolate is D2-1, and from the fraction D4 is D4-1 produced pure isolate.

The pure isolate, is measured its melting point, and test its purity with 3 eluent system, further characterized by instrument: ¹H-NMR, (Nucleus Magnetic Resonance), ¹³C-NMR, DEPT (Distortionless Enhancement by Polarization Transfer), HMBC (¹H-¹³C Heteronuclear Multiple Bond Connectivity), and HSQC (Heteronuclear Single Quantum Correlation).

Results and Discussion

Compound (D2-1)

Based on the existing peaks in the ¹³C-NMR spectrum is known that compounds D2-1, containing 10 C atoms and one C atom bonded to oxygen, based on chemical shift there are 10 peaks, peak at: 14.0978; 22.6722; 24.6745, 29.0527; 29.2251; 29.3401; 29.4167; 29.5700; 31.9076, and 34.0057 ppm and peak 179.6744 ppm for C atom that binds to oxygen atom (C=O). Based on data from its DEPT, that the D2-1 containing compounds: methyl group, methylene group (CH₂), and no group met in (CH). Data on the relationship with the ¹³C-NMR DEPT contained in Table 1.

Based on Table 1, the compound D2-1 is possibility to a straight chain compounds. Because in this compounds there is only one (1) methyl groups (on DEPT, there is only one group that leads to the upper or opposite to CH₂). A compound with a methyl group, chances are straight chain. Furthermore compound D2-1 is characterized by a Mass Spectroscopy to determine its mass by its fragmentation. This determination is based on data obtained from the ¹³C-NMR data and DEPT above. D2-MS spectrum of compound 1 are in Figure 1.

Table 1. ¹³Carbon Chemical Shift-DEPT Data Compound D2-1

Peak No.	¹³ C-NMR Chemical Shifts Position (ppm)	DEPT (ppm)	Position on DEPT
1.	14,0978	14,1987	CH3
2.	22,6722	22,7731	CH2
3.	24,6745	24,7754	CH2
4.	29,0527	29,1536	CH2
5.	29,2251	29,3260	CH2
6.	29,3401	29,4410	CH2
7.	29,4167	29,5176	CH2
8.	29,5700	29,6709	CH2
9.	31,9076	32,0085	CH2
10.	34,0057	34,1066	CH2
11.	179,6744	-	C=O

<< Target >>

Line#:12 R.Time:27.235(Scan#:5448) MassPeaks:324
 RawMode:Averaged 27.230-27.240(5447-5449) BasePeak:57.05(168611)
 BG Mode:Calc. from Peak Group 1 - Event 1

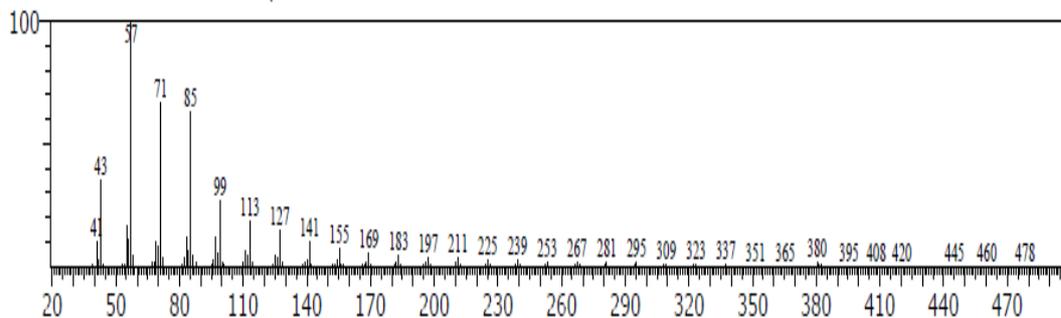
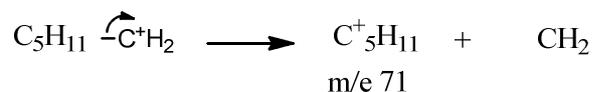
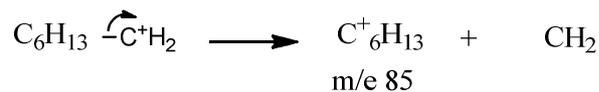
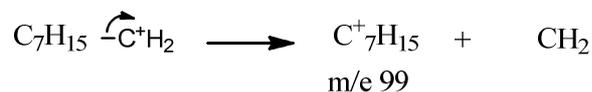
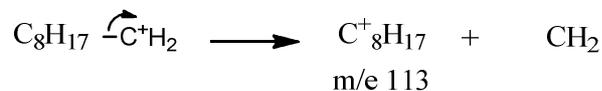
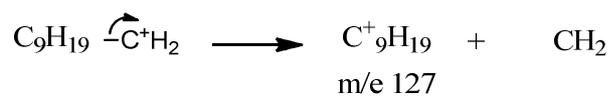
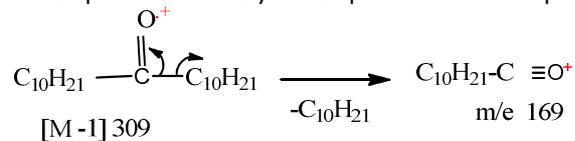


Figure 1. MS Spectrum of D2-1

Fragmentation pattern of compounds D2-1 at the beginning of the mass [MH] is 309. Other peaks are likely other parts of the compound detected. The fragmentation pattern is as follows:



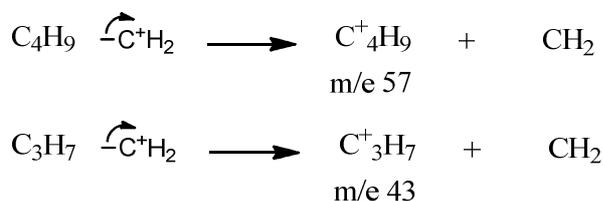
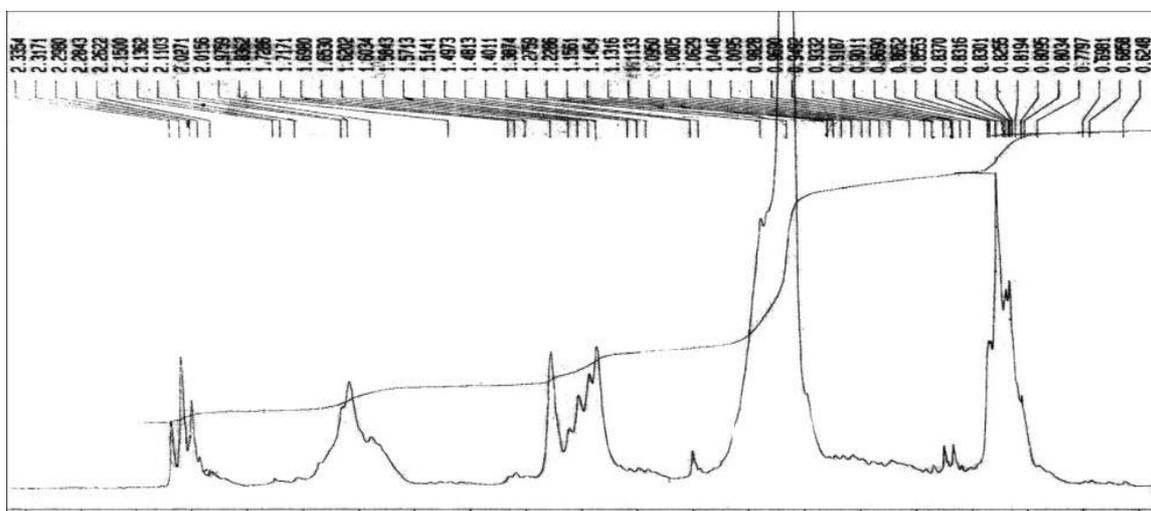


Figure 2. Fragmentation pattern of compound D2-1

The above pattern is a ketone compound fragmentation pattern, the first breaking of oxonium ion from alkyl deky (C₁₀H₂₁). Furthermore oxonium ion would break to be a deky and free CO uncharged. Dekyl group will be break by heterolysis to be other alkyl with m/e smaller and uncharged compounds CH₂, ultimately resulting positively charged alkyl C₃H₇ with m/e 43.

Figure 3. ¹H-NMR Spectrum of D2-1

Based on ¹H-NMR spectrum is known that the chemical shift of methylene protons (CH₂)



ranging from 2.2622 to 2.3354 ppm in the form of a triplet, indicating CH₂ groups which are in addition to the carbonyl group (proton of atom C-2), while the protons of the CH₂ groups that have chemical shift smaller, is the proton of CH₂ that far from the carbonyl group. Methyl groups as triplet and has chemical shift 0.7797 to 0.8652 ppm (figure 3).

HSQC spectrum is known that there is a correlation between the proton H-2 (2.3 ppm) with the atom C-2 (34.007 ppm); correlation between proton H-3 (1.63 ppm) with the atom C-3 (24.6745 ppm); correlation proton H-4 (1.26 ppm) with C-4 atom (31.9076 ppm); correlation proton H-5 (1.22 ppm) with the atom C-5 (22.6722 ppm); correlation between protons 6-10 (1.22 ppm) with atom C-6-10 (29.0527; 29.2251; 29.3401; 29.4167; 29.5700, and the correlation between the proton H-11 (0.78 ppm) with C-atom 11 (14.0978 ppm). Characterization of D2-1 with HMBC (Heteronuclear Multiple Bond Connectivity) is obtained the following relationship.

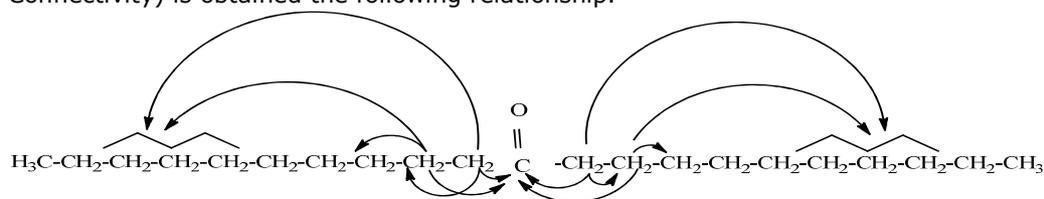
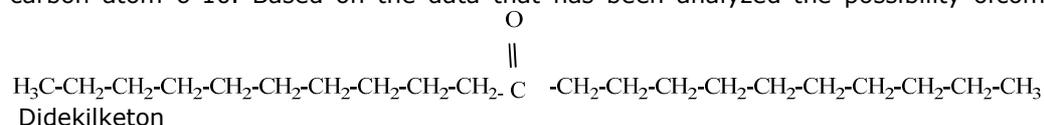


Figure 4. Relationship Proton and carbon atoms in long distance

Correlation in Figure 4, shows the correlation between protons H₂ with carbonyl atom, and correlated too with atom C-3 and with the C atoms 6-10. Then

correlation between protons H³ with carbonyl C atom, and correlated too with the carbon atom 4 and with carbon atom 6-10. Based on the data that has been analyzed the possibility of compounds D2-1 is:



Conclusion

D2-1 compound is possibility to didekilketon, with melting point of 176-178°C.

Acknowledgements

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