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**Research Article**

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# Isolation and characterization Flavonoids from The Bark of *Toona Sureni* (Blume) *Merr*

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**ABSTRACT**

*Toona Sureni (blume) Merr is one of species in the Meliaceae family. Phytochemical screening of the bark of Toona Sureni showed the presence of flavonoids, phenolics, tannins, terpenoids and saponins. According to preliminary examination, this research has been conducted on isolation and characterization flavonoids from the bark of Toona sureni. Isolation was done by macerated with n-hexane, ethyl acetate and methanol. Each of extracts was eluted in column chromatography (silica gel) to get a pure extract. Based on spectrum analysis of UV-vis, IR, NMR and mass spectroscopy were known that flavonoids compound in ethyl acetate extract was catechin.*

**Key words**: *Toona sureni*, phytochemical, flavonoids, catechin.

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## INTRODUCTION

Indonesia is a largest archipelago in the world with a total number of secondary metabolites more than ten thousands from variety plants. The rich flora of Indonesia includes many unique varieties of tropical plant life in various forms. One of million species that had been researched is family of *Meliaceae*, i.e. *Toona sureni* that had essential oil from their barks and fruits [1].

*Toona sureni* had been reported contained tetranortriterpenoids [2-3], carotenoids [4-6], triterpenoids [7], and methyl gallate [8]. All of that compounds were founded in the leaves of *Toona sureni*. Another genus, *Toona sinensis*, showed the presence of phenolic compounds, flavonoids and terpenoids in their bark [9-10].

In this research, we studied on isolation and characterization another secondary metabolite in the bark of *Toona sureni*.

## EXPERIMENTAL SECTION

Plant material
The bark of *Toona sureni* that were used in this reasearch were collected in Belimbing Padang, and were identified at Andalas University Herbarium, Padang (Anda. Fr).

Chemicals
Chemicals that were used are: TLC plats (silica gel 60 F254 Merck), silica gel E.Merck 7733, n-hexane, ethyl acetate, methanol, alcohol.

Procedure
One point five kilograms (1.5 kg) air-dried and powdered barks of *Toona Sureni* were macerated with 4 x 12 L of n-hexane for 4 days at room temperature. After filtration, the residu was macerated with 6 x 15 L of ethyl acetate for 4 days and 10 x 21 L of methanol for 5 days. Each fractions was concentrated with rotary evaporator.

Ethyl acetate extract (12 g) was eluted in column chromatography (silica gel 0.2-0.5 mm and 0.063-0.2 mm) by using Step Gradient Polarity (SGP) with n-hexane : ethyl acetate : methanol. Fraction with the same Rf on TLC were combined and rechromatographed on the silica gel column, and then recrystalized from n-hexane to produce white needle crystals. The identification of the products were carried out by means of melting points apparatus, UV-Vis, IR, NMR and mass spectroscopy.

## RESULTS AND DISCUSSION

Product of isolation the bark of *Toona sureni* were 8.03 g, 46.81 g, and 92.14 g respectively in n-hexane, ethyl acetate and methanol. Its means that polar compounds were dominantly in the bark of *Toona sureni*. After rechromatography of extract in ethyl acetate, we got 151.4 mg of white needle crystal with only one spot at Rf = 0.37. That crystal has m.p. 102 – 103 ºC. This informed that it was the pure solid crystal.

Uv-vis spectroscopy spectra initiates 2 absorption with $\lambda_{max}$ at 342.0 nm (band 1) and 280.4 nm (band 2). These indicate that compound has conjugated double bond, and 2 characteristic peaks of flavonoids. According to shift reagent that were used (NaOH, $AlCl_3$, $AlCl_3$ + HCl, NaOAc, and NaOAc + $H_2BO_3$), absorption at 280 nm shows the presence of phenolic chromophore from flavonoids. Then, addition of alkalis made a bathochromic shift identifies of hydroxyl substituent in aromatic compounds [11].

Infrared spectra of the product shows the product contain hydroxyl group. This is proved by existence of an absorption at 3401.21 $cm^{-1}$ due to O – H bending vibrations. Strong absorption intensities appearing at 3401.21 $cm^{-1}$ and at 1031.89 $cm^{-1}$ refers to hydroxyl and ether group existence in the product (C – O bending). The presence of two strong absorption at 1628.50 $cm^{-1}$ and 1522.49 $cm^{-1}$ strengthens aromatic absorption system. Furthermore, absorption bands due to aromatic C – H bending vibrations in out of plane region i.e. 983.04 – 668.75 $cm^{-1}$ indicates that aromatic ring was subtituted. Interpretation of infrared spectra analysis informs that the product has all of fungsional groups on flavonoids.

Analysis of $^{13}$C-NMR product appears 15 signals that shows how much carbons are. For simplified the kind of carbons that it has, $^{13}$C-NMR DEPT 135 ºC spectra indicates that the product has seven of quaternary carbons, seven of tertiary carbons and one of secondary carbon.

The $^{1}$H-NMR of the product has nine of protons with three types of magnetic environment of the protons. There are, aromatic cyclic tertiary protons that showed at chemical shift 5.85 – 6.83 ppm. Nonaromatic cyclic proton appeared at chemical shift 3.96 – 4.57 ppm. The last, the chemical shift at 2.49 – 2.85 ppm indicates the secondary protons of methylene (-$CH_2$-).

**Table 1. Chemical shift of the product and catechin from literature**

| C | $^{13}$C-NMR (ppm) | | H | $^{1}$H-NMR (ppm) | |
|---|---|---|---|---|---|
| | Isolated compound | Catechin from literature | | Isolated compound | Catechin from literature |
| 2 | 82.9108 | 82.7066 | 2 | 4.56 d | 4.5738 d |
| 3 | 68.8703 | 68.34 | 3 | 3.97 m | 3.9508 t |
| 4 | 28.5899 | 29.7102 | 4α | 2.40 dd | 2.5433 t |
| 10 | 100.8810 | 100.6497 | 4β | 2.84 dd | 2.9246 dd |
| 5 | 157.6437 | 157.2079 | | | |
| 6 | 96.3408 | 96.1496 | 6 | 5.92 d | 6.0305 d |
| 7 | 157.8821 | 156.9111 | | | |
| 8 | 95.5586 | 95.4602 | 8 | 5.85 d | 5.8850 d |
| 9 | 156.9760 | 157.7249 | | | |
| 1’ | 132.2526 | 132.1986 | | | |
| 2’ | 115.3125 | 120.0674 | 2’ | 6.84 d | 6.9020 d |
| 3’ | 146.2960 | 145.7086 | | | |
| 4’ | 146.3121 | 145.6320 | | | |
| 5’ | 116.1614 | 115.7204 | 5’ | 6.76 d | 6.8050 d |
| 6’ | 120.1294 | 115.2417 | 6’ | 6.75 dd | 6.7680 dd |

According to HMQC spectra, well known that H-3 proton at δ 3,97 ppm in C ring of flavonoids has a correlation with C-3 carbon at δ 68.8 ppm, and H-2 at δ 4.56 ppm with C-2 at δ 82.9 ppm. The correlation between carbon and proton in A ring occurs with H-6 (δ 5.93 ppm) to C-6 (δ 96.3 ppm), and H-8 (δ 5.85 ppm) to C-8 (δ 95.5 ppm). In the same ways, it be done in B ring e.g. H-6’ with C-6’, H-2’ with C-2’, and H-5’ with C-5’. All of the spectra of HMQC showed that C ring has proton on C-2 and C-3, A ring on C-6 and C-8. Beside that, B ring has a proton on C-2’, C-5’ and C-6’.

Considerating of spectra $^{13}$C-NMR and $^{1}$H-NMR of isolated compound with similar product, catechin, that had been isolated by Santoni from *Toona sinensis* are viewed at Table 1.

According to IR, $^{13}$C-NMR, $^{1}$H-NMR analysis and correlation with catechin isolated by Santoni, the structure of product that had been isolated from the bark of *Toona sureni* is shown in Figure 1.

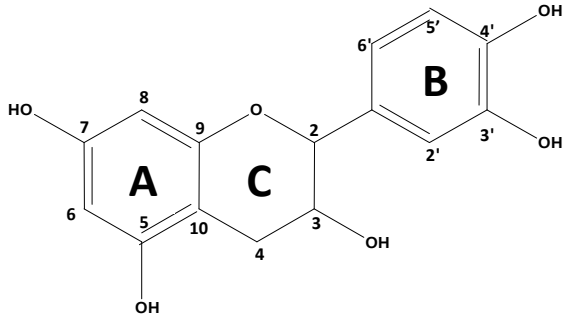


**Figure 1. Chemical structure of catechin**

HMBC spectra analysis and correlation with the structure can be seen in Figure 2. It showed correlation of H-2 with C-2, C-3, C-9, C-1', C-2' and C-6'. This correlation indicated proton and the adjacent carbons.

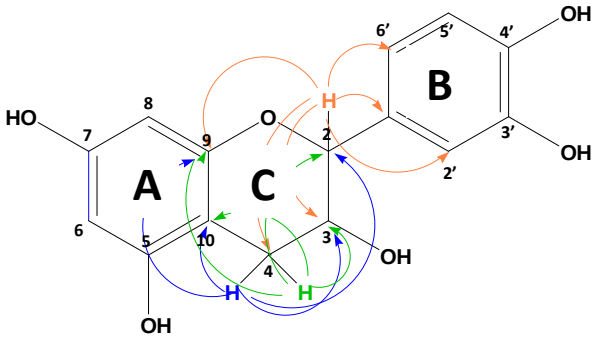


**Figure 2. correlation of HMBC in isolated compound**

$^{1}$H – $^{1}$H Correlated Spectroscopy (COSY) was used to identifies the correlation between proton and another proton in adjacent carbon. It shows that H-2 has the correlation with H-3. In other side, H-3 correlates with H-4. It means H-3 closed with H-2 and H-4. The correlation of protons in structure are viewed in Figure 3.

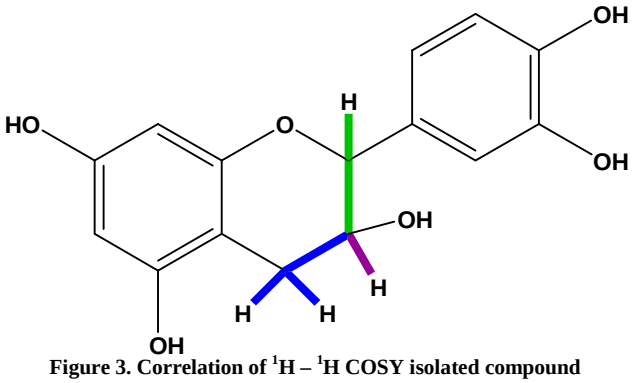


**Figure 3. Correlation of $^{1}$H – $^{1}$H COSY isolated compound**

The LC-MS spectra of the product are shown in Figure 4. In this spectra appears only one peak with retention time 1.55. The molecular ion ($M^{+}$) of the spectra informing the molecular mass of the product is 291.24. This molecular mass is suitable with the molecular mass of catechin.

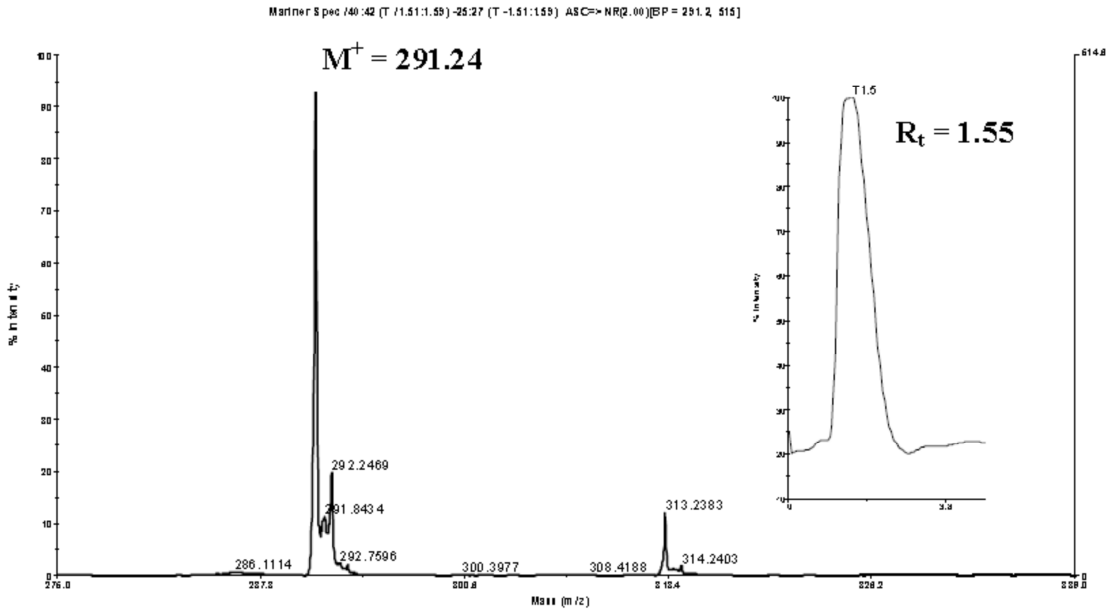


**Figure 4. LC-MS spectra of isolated compound**

## CONCLUSION

The conclusion of this research are:
1. Isolated compound of the bark of Toona sureni are catechin according to $^{1}$H-NMR, $^{13}$C-NMR, HSQC, HSQC, FT-IT, and mass spectroscopy.
2. This research produced 151.4 mg of white needle pure of ethyl acetate extract from isolated compound.

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