

Analysis of Alcohol Content in A Herbal Medicine of Noni Using Gas Chromatography Method

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ABSTRACT

Noni (*Morinda citrifolia* L) has long been used for herbal medicine to treat various medical conditions, such as arthritis, diabetes, hypertension, arteriosclerosis, heart disease, blood vessel problems, tumour and cancer. One of the compound in some fruit is ethanol. Ethanol in the pharmaceutical industry is often used as a solvent. Regulation of the Head of the National Agency of Drug and Food Control of the Republic of Indonesia (BPOM RI) Number HK.03.1.23.06.10.5166 Year 2010 states that alcohol-containing drugs, traditional medicines, and supplements must declare the alcohol content (in percentage) on the label. There are many available noni herbal medicines on Indonesia's market, either with or without a distribution license. This research aims to analyse alcohol content, product type, distribution licence, and label of alcohol content on noni herbal medicines from herbal drugstores or online shops in Jakarta. Determination of alcohol content in this study was performed using Gas Chromatography Headspace method. The result showed that 13 out of 20 samples contained alcohol ranging between 0.04 - 1.07%. None of the samples with detected alcohol had a label "contains alcohol in per cent (%)". Eighteen samples were categorised as "beverages" and two samples were categorised as "traditional medicines or herbal medicines". These two samples contain 0.21 and 0.07% alcohol. In accordance with the Fatwa of the Indonesian Council of Ulama, these herbal medicines are "allowed" to be consumed with certain conditions: the medicines do not endanger the health and do not encourage substance abuse, their use must follow the recommended dosage and they should not be used intentionally to get drunk. Out of 18 samples within the "beverage" category, only 12 samples were halal; the other 6 were haram.

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1. Introduction

Ethanol, or ethyl alcohol, is an alcoholic compound found naturally in fruits. People often juice fruits which also contain a considerable amount of alcohol compounds. (Roswiem, 2015). If alcohol is forbidden, should fruit juice also be banned? However, it turned out that the Prophet Muhammad still drank fruit juices that had been stored for three days. However, after more than three days of storage, he did not consume it anymore.

The law that applies to the fruit juice's shelf life is based on the Prophet Muhammad's hadith as follows:

"Drink it (juice) while it's not hard yet!" Friends ask: How long has he been hard? It (juice) becomes hard in 3 days. (H.R. Ahmad narrated from Abdullah bin Umar). (Taymiyah, 1987)

Noni (*Morinda citrifolia* L) is a tropical plant that has been used as food and herbal medicine. The herbal medicine from the noni fruit is available in liquid medicinal preparations known as noni juice, extract or noni fruit's fermentation product. If we analyse the content of noni herbal medicine, alcohol will be detected.

Based on the National Agency of Drug and Food Control of the Republic of Indonesia regulation (BPOM RI, 2010), alcohol-containing medicinal products or drinks must put a label with the words "containing alcohol" accompanied by its amount in per cent (%) units. Apart from that, the Fatwa Commission of the Indonesian Council of Ulama has made the following decree on Alcohol for Medicinal Substances, using alcohol from non-khamr industry alcohol for manufacturing liquid or non liquid medicines is permitted with certain conditions. The drugs must not be harmful to health, not misused, safe, used per appropriate dosage and not used intentionally to get drunk (Babar et. Al.,2020).

To ensure the halal status, manufacturers could obtain a halal certificate of medicinal products from a trusted Halal Certification Agency (Nirwandar, 2021; Maryati, 2020). Many noni juice herbal medicines are traded in herbal medicine stores or online sellers. Some herbal medicines are distributed legally, and some are illegal. Besides, some products do not include label regarding their alcohol content. Therefore, this study's questions are: do herbal medicines made from noni juice, extract or fermented noni fruit sold in herbal medicine shops or online shops in Jakarta contain alcohol? If yes, what is the alcohol percentage in them? This study aimed to analyse the alcohol content, product type, distribution licence, and the inclusion of alcohol content in the labels of noni herbal medicines traded in herbal medicine shops or online shops in Jakarta.

2. Method

Delta State shares similar climatic features with other states in the Niger-delta; its vegetation is characterized by mangrove forests, brackish swamp forests and rain forests. It experiences a tropical climate consisting of a rainy season (April to November) and a dry season (December to March). The Warri River stretches within latitude $5^{\circ} 21^1 - 6^{\circ} 00^1$ N and longitude $5^{\circ} 24^1 - 6^{\circ} 2^1$ E and has its source around Utakwa Uno (Aghoghovwia, 2011). Samples were collected in the dry months: January (early dry season) and March (late dry season) and wet months: May (early rainy season) and October (late rainy season) from three different locations along the River Warri.

2.1 Materials

The materials used were samples of noni herbal medicines, absolute ethanol, DMF and distilled water. The equipment included glass tools, micropipettes 100 μ L and 1000 μ L, Gas Chromatography (GC) Head Space.

2.2 Alcohol Analysis Method

The study applied Gas Chromatography Headspace to determine the alcohol content in herbal medicine products.

2.2.1 Sample Preparation

The method refers to the United States Pharmacopeia (2016). One gram or one ml sample was taken and placed in the headspace vial. Four ml of 50% DMF was added into the headspace vial. The vial was locked with a 20 mm Chimper. This solution was shaken slowly. The solution should not come

into contact with the vial cap. After the mixture is homogenous, it is ready to be injected into Gas Chromatography.

2.2.2 Making Alcohol Standard Curve

The alcohol stock solution was prepared by diluting the standard alcohol 99.9%. The final concentration of the stock solution was 77,638.78 mg/L. Standard ethanol solutions were prepared by serial dilution of the stock solution (0.01 ml to 1 ml) in 50% DMF.

2.2.3 Instrument Set up

Analysis of alcohol in samples and standards was performed using the headspace method, followed by the Gas Chromatography method. The Headspace method is used to separate volatile compounds, such as alcohol, in a sample. Before use, the headspace was left to stand for 15 minutes. Headspace was set up with the following parameters: needle temperature = 85 °C, transfer line temperature = 100 °C, oven temperature = 85 °C, pressure time = 1.0 min, injection time = 0.04 min, withdraw = 0.5 min, Thermostat (Oven time) = 40 min, GC Cycle Time = 24 mins. The column and detector used for the Gas Chromatography method was Capillary Column DB-624 and FID, respectively. The instrument was adjusted at inlet Injection mode = Split 2.5: 1, injection temperature = 140 °C, constant velocity = 25 cm/sec, oven initial temperature detector (FID) = 40 °C, and hold = 5 min.

2.2.4 Result Interpretation

Calculation of the residual solvent in the sample was performed using a standard calibration curve with a line equation: $Y = bx + a$, and the following formula:

$$\text{Level of residual solvent} = \frac{(\text{Area}-a)}{\frac{b}{W \text{ sample}}}$$

Note:

a = Intercept of a standard calibration curve

b = Slope of a standard calibration curve

W sample = sample weight (g)

3. Result

3.1 Linearity of Alcohol Standard Curves

Linearity is the analytical method's ability to respond proportionally to the analyte concentration in the sample. Linearity is usually expressed in terms of the variance around the regression line's direction, which is calculated based on the mathematical equation obtained from various analyte concentrations in the samples. The linearity of the method can describe the accuracy of the analysis indicated by the coefficient of determination (R^2) with the condition that the value of $R^2 > 0.997$. The results showed that the standard curve of alcohol has the equation $Y = 24.2309 X - 2731.2336$ with R^2 of 0.9993 (Figure 1).

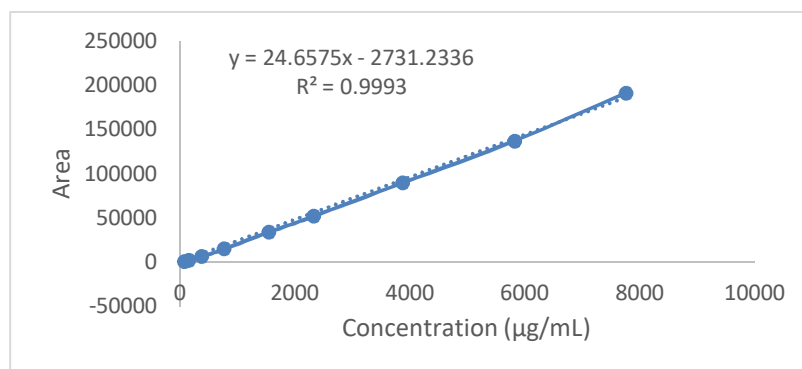


Fig 1. Standard Curve of Alcohol

3.2 Alcohol Content in Samples

There were 20 samples of extract, juice and fermented or distilled product of noni fruit. The alcohol percentage of the samples is given in Table 1.

Table 1. Analysis of alcohol content in noni herbal medicines

No	Sampel Code	Alcohol Content (%)	Note			
			Halal Logo	Distribution Permit	Origin of Materials / Production Process	Color
1	HRC 1	0,21	MUI	POM TI	Noni extract	Blacks brownish
2	HRC 2	0,84	-	Dep Kes PIRT	Noni Fermentation	Brownish black
3	HRC 3	ttd	-	-	Noni Juice	Clear
4	HRC 4	0,68	-	PIRT	From the juice of noni fruit	Brownish black
5	HRC 5	0,59	MUI	-	100 % Noni Juice	Brown
6	HRC 6	ttd	Halal Logo	-	Fermentasi mengkudu	Blacks brownish
7	HRC 7	ttd	MUI	PIRT	100 % Noni Juice	Brownish black
8	HRC 8	ttd	-	-	Fermentation Noni	Blacks brownish
9	HRC 9	0,34	MUI	-	Extract Noni (Result Destilation Noni)	Brownish black
10	HRC 10	ttd	-	-	Extract Noni	Blacks brownish
11	HRC 11	0,91	-	PIRT MD 313	Extract Noni Fruit	Brownish black
12	HRC 12	0,06	-	PIRT	Healthy Drink	Black
13	HRC 13	0,65	-	-	Healthy Drink	Brownish black
14	HRC 14	0,88	-	-	Fermentation	Blacks brownish
15	HRC 15	1,07	MUI	-	100 % Fermentation of Noni Fruit	Blacks
16	HRC 16	0,04	-	PIRT	100 % Noni Juice	Brownish black
17	HRC 17	ttd	-	PIRT	Noni Extret + Species	Yellowish-brown
18	HRC 18	0,07	MUI	-	Noni Extract + flower sugar + Habbatusauda	Blacks brownish
19	HRC 19	0,66	Logo Halal (not Halal MUI)	BPOM RI. MD 16573100	100 % Noni Juice	Blacks brownish
20	HRC 20	ttd	-	-	100 % Fermentation of Noni Fruit	Reddish black

Note: ttd: undetected

4. Discussion

Table 1 showed that The noni herbal medicines in this study were in liquid form with ingredients from noni juice, extract and noni fruit fermentation. Some samples obtained a distribution licence of BPOM MD, BPOM TI or PIRT; others do not have a distribution licence. BPOM MD is the registration code for the food products manufactured in Indonesia. While BPOM TI is for imported traditional medicines. P-IRT is specific for the foods manufactured by home industry or small to medium enterprises (SME). Based on the source of raw materials and the production process of the

samples, they can be categorised into "beverage", "khamr" and "herbal medicine" products. "Beverage" products are made of noni juice or extract. The products generated from noni fruit fermentation are considered as "khamr". While the "herbal" products were sourced from fruit extract plus other ingredients derived from plants. For instance, habatussaudah and flower sugar, which also have bioactivities as herbal medicines. These products have received a distribution licence from BPOM RI and a halal certificate from MUI.

- Regarding the alcohol content:

- a. The alcohol content in seven samples (HRC 3, HRC 6, HRC 7, HRC 8, HRC 10, HRC 17, and HRC 20) is undetected.
- b. Alcohol was detected in thirteen samples (HRC 1, HRC 2, HRC 4, HRC 5, HRC 9, HRC 11, HRC 12, HRC 13, HRC 14, HRC 15, HRC 16, HRC 18, and HRC 19) with the percentage ranging from 0.04 – 1.07%.

Undetected alcohol in a sample does not always mean the sample does not contain alcohol. It could also be because the alcohol content in the sample is lower than the detection limit of Gas Chromatography Head Space tool. The gas chromatograph used in this study had a detection limit (LOD) value for alcohol of 2.8 mg/L or 2.212×10^{-4} %v/v. If the tested sample contains alcohol below the LOD concentration, the alcohol content could not be detected. Regarding the status of seven samples with undetected alcohol content, four are allowed to consume, and the rest is not allowed (especially for Moslems), based on the government regulations regarding the alcohol content in beverages and the MUI Fatwa regarding alcohol content in beverages and medicines (Table 2).

Table 2. Halal Status of Noni Herbal Medicines with Undetected Alcohol Content

No	Code of Sample	Halal Status	Result
1	HRC 3	√	Allowed (halal) for consumption as beverage because it is made from noni juice / extract with an alcohol content lower than 0,5 % and the alcohol comes naturally from noni fruit
2	HRC 10	√	Allowed (halal) for consumption as beverage because it is made from noni juice / extract with an alcohol content lower than 0,5 % and the alcohol comes naturally from noni fruit
3	HRC 7	√	Allowed (halal) for consumption as beverage because it obtained a distribution licence of PIRT and have MUI halal logo
4	HRC 17	√	Allowed (halal) for consumption as beverage because it obtained a distribution licence of PIRT
5	HRC 6	⊖	Haram for consumption, because the product generated from noni fruit fermentation and it considered as khamr
6	HRC 8	⊖	Haram for consumption, because the product generated from noni fruit fermentation and it considered as khamr
7	HRC 20	⊖	Haram for consumption, because the product generated from noni fruit fermentation and it considered as khamr

Note: √ allowed to consume
⊖ not allowed to consume

There were thirteen samples containing alcohol (HRC 1, HRC 2, HRC 4, HRC 5, HRC 9, HRC 11, HRC 12, HRC 13, HRC 14, HRC 15, HRC 16, HRC 18, and HRC 19). Some are categorised as "beverage" products and some are categorised as "traditional/herbal medicines". Samples categorised as a beverage were: HRC 9, HRC 11, HRC 13, HRC 19, and HRC 20, but the, samples categorised as beverages with the alcohol content greater than 0.5% (HRC 2, HRC 4, HRC 5, HRC 13 and HRC 19) is "haram" for Moslem's consumptions, based on MUI fatwa. There were two samples categorised as herbal or traditional medicines that contain alcohol: HRC 1 and HRC 18. Based on MUI Fatwa, their usage is "allowed" with certain conditions, such as : not endangering health, not

misused, safe and in accordance with the dose, and not used intentionally to make drunk (Hassan et al., 2020; Abdel-Wahhab et al., 2020).

5. Conclusion

Based on our results, we concluded that thirteen out of twenty samples of noni herbal medicine were detected with an alcohol content of 0.04–1.07%. Based on the source of raw materials, distribution licence, and Halal Certificate or MUI Halal Logo, 18 samples were categorised as "beverage" and 2 samples were categorised as "traditional medicine or herbal medicine". There were 2 samples categorised as "traditional medicine or herbal medicine", with ethanol or alcohol content of 0.21 and 0.07%. These medicines are "allowed" to consume with the following conditions: The drugs must not be harmful to health, not misused, safe, used per appropriate dosage and not used intentionally to get drunk. Within the "drink" category, only 12 samples were "halal" for consumption, and 6 samples were "haram" for consumption. All samples with detected alcohol content do not state "contains alcohol" in the label, along with alcohol content in per cent (%).

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