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Isolation of Methyl Gallate from the Fruit of *Toona Sureni* and Its Utilization in Preventing Damage the Vegetables Oil During Storage

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ABSTRACT

A study to assess acid value (AV) in palm oil and coconut oil during storage was conducted. The study was conducted three stages: extraction methyl gallate from the surian fruits, oil extraction, and oil quality testing by determining the acid value (AV). To test the quality of the oil, the samples were added of methyl gallate and kept at temperature 40°C for 30 days. As a comparison used synthetic antioxidant, butylated hydroxytoluene (BHT), tert-butyl hydroquinone (TBHQ). Analysis of acid value (AV) is performed every 5 days, from day 0 to day 30. The results showed that methyl gallate derived from of surian fruit extract could prevent the increasing of acid value of palm oil and coconut oil during storage at 40°C.

Keywords: Acid value, Antioxidant, Coconut oil, Methyl gallate, Palm oil

INTRODUCTION

Methyl gallate is the main component of the phenolic compounds contained in *Toona sureni*. The compound is often found on the leave, stem and bark of Aceraceae and Meliaceae families. The literatures show that methyl gallate has many bioactivities, such as cytotoxic [1], antiviral [2], antioxidant [3-4], and antibacterial [5-8].

Utilization of synthetic antioxidants in food products have the potential to cause toxic in the body [9] and the toxic effect can lead to swollen of the liver and affect the activity of liver enzymes [10].

Currently, the tendency to isolate antioxidants derived from natural materials increase. Natural antioxidants can be used for food preservation by preventing lipid damage, and also be used for medicinal purposes. Antioxidants from natural materials have some advantages because these compounds have no side effect for human.

In this paper, we report utilization methyl gallate isolated from *Toona sureni* to prevent the damage the vegetables oil during storage.

MATERIALS AND METHODS

Materials

The plant materials were collected in Padang and identified in the Herbarium of the Andalas University (ANDA), Padang, with specimen M. Taufik Eka Prasada, 0107 (ANDA.Fr).

Silica gels for column chromatography and thin layer chromatography were obtained from Merck. All solvents were redistilled before used.

Isolation of methyl gallate

1.5 kg of air-dried and powdered fruits were macerated with 2 × 6 L of *n*-hexane for 2 days at room temperature. The extract was filtrated and the residu was macerated with 3 × 4 L of acetone for 3 days and 2 × 4 L of methanol for 5 days. Each fractions was concentrated with rotary evaporator. Acetone extract (15 g) was eluted by silica gel in column chromatography by increasing percentage of ethyl acetate in *n*-hexane (100:0 up to 0:100 with 300 mL of each of them) and ethyl acetate:methanol (90:10 (300

mL)). Fraction with the same R_f on TLC were combined and rechromatographed on the silica gel column by increasing percentage of ethyl acetate in *n*-hexane, and then recrystallized from *n*-hexane to produce white needle crystals.

Extraction of coconut oil

1 kg of grated coconut was placed in a plastic pot then added 1 Liter of water. Furthermore the grated coconut was squeezed to form coconut milk. The coconut milk was separated from coconut pulp using cloth filter. Let the milk for 2 hours to form two layers, the lower layer (water) and the top layer (cream) containing coconut oil. The coconut oil was separated and heated at a temperature 60°C to 70°C for one hour and stored to further processing.

Extraction of palm oil

10 kg of palm fruits was heated using steam for 3-4 hours. Separated fiber from the seeds and squeeze the fiber to produce orange palm oil. Let the palm oil one day to settle the dirt. Furthermore evaporated the water content in palm oil using a rotary evaporator. Stored the palm oil for further processing.

Determination of acid value

25 mL the oil was added by methyl gallate isolated from the fruit of *Toona sureni* with five treatment concentrations (25 mg/L, 50 mg/L, 100 mg/L, 150 mg/L, and 200 mg/L). The oil also was added by synthetic antioxidant (BHT and TBHQ). The concentration of syntetic antioxidants were used by the maximum allowed for the addition of synthetic antioxidants in vegetable oils. During analysis, samples were stored at temperatures of 40°C for 30 days. Analysis Acid Value (AV) was performed every 5 days starting on day 0 up to the 30th day.

To determinate Acid Value, weigh approximately 3 g sample in erlenmeyer and add 50 mL neutral alcohol. Homogenize the solution completely by heating it above the hotplate until no bubbles, add 3 drops of phenolphthalein indicator and titrate with 0.1 N KOH until until the resulting purple colors that are stable at least 30 seconds. Acid Value was calculated by formula below:

$$AV = \frac{V \times N \times 56.1}{W}$$

AV=Acid value

V=Volume of KOH (mL)

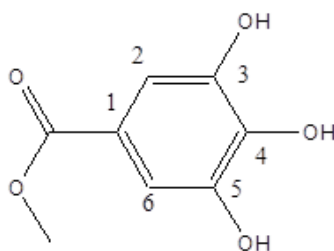
N=Concentration of KOH solution (N)

W=Weight of sample (g)

56.1=Potassium hydroxide molar mass, g/mol

RESULTS AND DISCUSSION

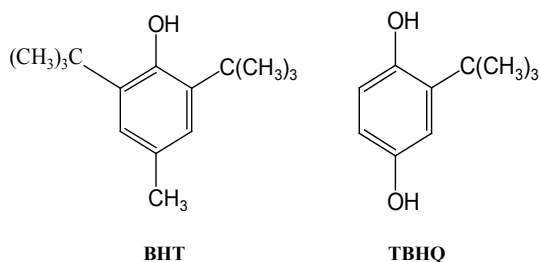
Extract in acetone (15 g) was subjected to column chromatography of silica gel and eluted with an increasing percentage of ethyl acetate in *n*-hexane. Fractions with the same R_f on TLC were combined and rechromatographed on the silica gel column and eluted with an increasing percentage of ethyl acetate in *n*-hexane and was further recrystallized from *n*-hexane to give white crystals 19 mg. Methyl 3,4,5-trihydroxybenzoate or methyl gallate was obtained as white crystals, m.p 188-189°C. The structural elucidations of the compound have been reported [4]. The molecular formula was determined to be $C_8H_8O_5$.



Methyl gallate

The mean acid value of palm and coconut oil containing different concentration of methyl gallate as affected storage period is showed in Tables 1 and 2. Data showing that mean acid value of palm oil and coconut oil containing different concentration of methyl gallate are lower than palm oil and coconut oil without containing methyl gallate after 30th day. The decreasing effect of mean acid value of palm and coconut oil during storage at 40°C is showed in Figures 1 and 2.

The main acid value of palm oil dan coconut oil containing antioxidant type (methyl gallate, BHT, and TBHQ) and without antioxidant is showed in Tables 3 and 4. Effect of antioxidant type on acid value of palm oil and coconut oil during storage period at 40°C is showed in Figure 3 and 4. The results showed that methyl gallate derived from of surian fruit extract could prevent the increasing of acid value of coconut oil and palm oil during storage at 40°C.

**Table 1: Mean acid values of palm oil containing different concentrations of methyl gallate**

Storage period in days	Concentrations of Methyl gallate (mg/Kg)					Control
	25	50	100	150	200	
0	6.10	6.10	6.10	6.10	610	610
5	5.31	4.04	6.62	4.28	4.48	640
10	4.00	3.51	3.60	3.71	3.96	4.48
15	4.12	4.21	4.89	5.11	4.83	5.15
20	3.54	3.75	3.25	3.62	3.84	3.95
25	4.07	4.29	4.04	4.26	4.40	4.69
30	3.69	3.61	3.39	3.32	3.56	3.94

Table 2: Mean acid values of coconut oil containing different concentrations of methyl gallate

Storage period in days	Concentrations of Methyl gallate (mg/kg)					Control
	22	44	88	132	176	
0	1.02	1.02	1.02	1.02	1.02	1.02
5	0.99	0.73	1.14	0.90	0.98	0.80
10	1.07	0.82	0.65	0.74	0.98	0.57
15	1.05	1.06	0.74	0.97	1.15	0.79
20	1.22	0.98	1.23	1.05	1.13	1.02
25	1.06	1.07	1.22	1.14	0.90	1.22
30	0.98	0.98	0.90	0.97	1.06	1.47

Table 3: Mean acid values of palm oil containing different of antioxidant type as affected by storage period

Storage period in days	A	B	C	D
0	6.10	6.10	6.10	6.10
5	5.62	4.75	4.87	6.40
10	3.60	4.01	4.06	4.48
15	4.89	4.29	4.15	5.15
20	3.25	3.26	3.46	3.95
25	4.04	4.22	4.43	4.48
30	3.39	3.84	3.88	3.94

A=Concentrations of methyl gallate (100 mg/Kg)

B=Concentrations of BHT (100 mg/Kg)

C=Concentration of methyl TBHQ (150 mg/Kg)

D=Without antioxidant

Table 4: Mean acid values coconut oil containing different of antioxidant type as affected by storage period

Storage period in days	A	B	C	D
0	1.02	1.02	1.02	1.02
5	0.73	0.74	0.52	0.80
10	0.82	0.90	0.82	0.57
15	1.06	0.98	1.14	0.79
20	0.98	1.31	1.22	1.47
25	1.07	0.73	1.22	1.22
30	0.89	0.90	0.97	1.47

A=Concentrations of methyl gallate (44 mg/Kg)

B=Concentrations of BHT (62 mg/Kg)

C=Concentration of methyl TBHQ (113 mg/Kg)

D=Without antioxidant

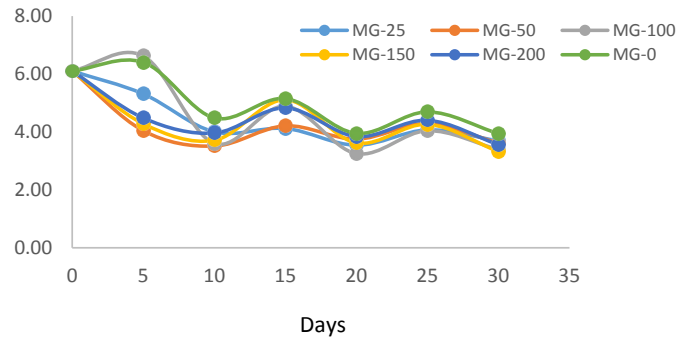


Figure 1: Effect of methyl gallate on acid value of palm oil during storage at 40°C

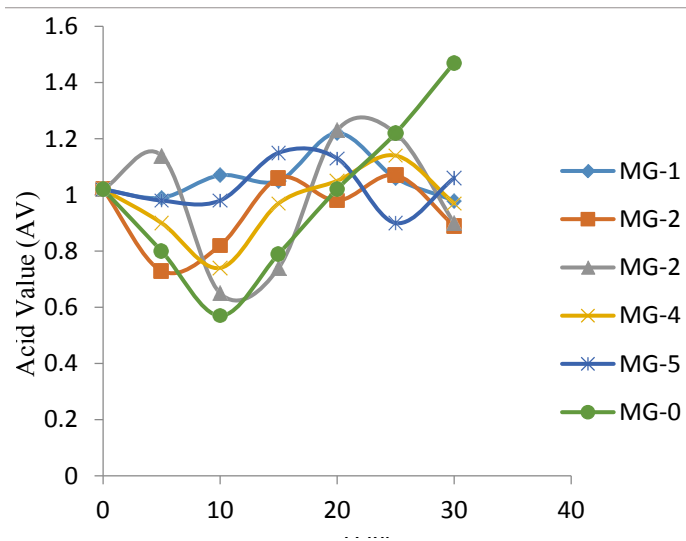


Figure 2: Effect of methyl gallate on acid value of coconut oil during storage at 40°C

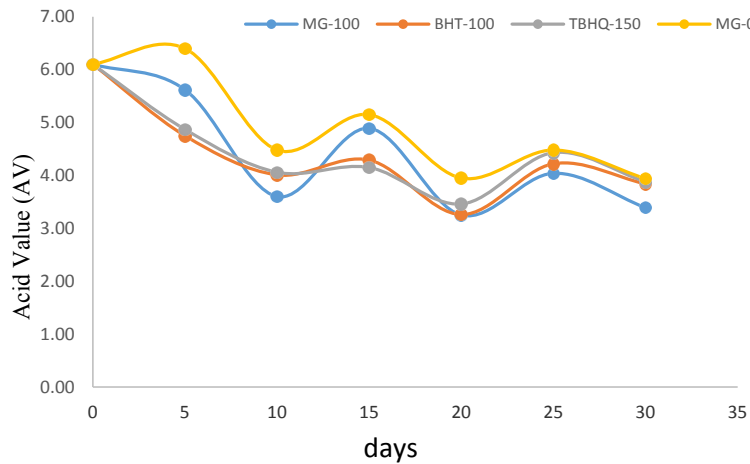


Figure 3: Effect of antioxidant type on acid value of palm oil during storage at 40°C

CONCLUSION

Methyl 3,4,5-trihydroxybenzoate (methyl gallate) derived from of surian fruit extract could prevent the increasing of acid value of palm oil and coconut oil during storage at 40°C.

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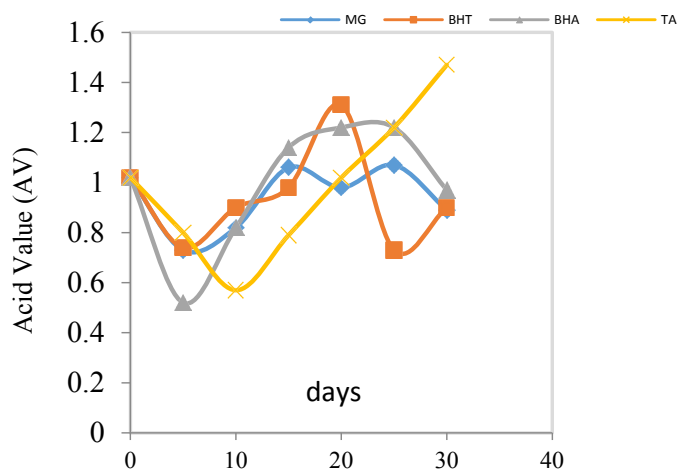


Figure 4: Effect of antioxidant type on acid value of coconut oil during storage at 40°C.

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