EFFECTS OF PRE-TREATMENT CONDITION ON THE YIELD AND QUALITY OF NEEM OIL OBTAINED BY MECHANICAL PRESSING

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ABSTRACT

Neem (*Azadirachta indica A. Juss*) is one of the very few trees known in the Indian subcontinent. Neem seed is a part of Neem tree which has high concentration of oil. Neem oil extraction using mechanical pressing is the cheapest method, and some pretreatment such as heating influenced its yield and physical and chemical characteristics. The quality of Neem oil extracted by mechanical pressing was changed during storage. The results of this experiment showed that the optimum pressure for mechanical pressing of Neem seed was 5000 psi. The pretreatment heating on the Neem seed particles and storage caused the oil quality reduced, therefore room temperature was found to be the recommended temperature for the Neem oil extraction using mechanical process.

Keywords: neem, oil, yield, quality, extraction, pressing.

INTRODUCTION

Neem tree (Azadirachta indica) belongs to the mahogany family, *Meliaceae*. Neem tree is a tropical plant with a good environmental adaptability [1,2]. In Indonesia, neem tree can be found in several regions, such as East Nusa Tenggara, Bali, West Nusa Tenggara, East Java, Central Java, and West Java. All parts of the tree have many beneficial uses for natural insecticide, cosmetics ingredient, health treatment, soap and toothpaste ingredient, etc [1-4]. Even though the Neem tree has many potential applications; the utilization of this tree in Indonesia for industrial purposes still limited, as a canopy tree.

Part of Neem tree which is commonly used is the seed. Neem seed has the largest oil fraction (approximately 40-58.9% weight) compared to the other parts of the tree [1,5]. The kernels yield a greenish to brown-colored oil with strong odor resembling garlic. The refined and purified Neem oil has the following characteristics: specific gravity 0.9087; refractive index 1.4612; iodine value (Wij's) 66.4; saponification value 290.9; and unsaponifiable matter 0.8% [5]. Neem oil has been used in many different industries, especially cosmetics and pharmaceutical industries (as supporting ingredient for medicines) [1-3].

Extraction of oil from Neem seeds can be performed using three different methods: mechanical extraction, solvent extraction, and supercritical fluid extraction. Mechanical extraction is the common method used to extract the Neem oil from the seed [1,6] , since this method is effective for seed contain 30-70% oil [7]. The mechanical extraction has several advantages compared to the other methods, such as simple equipment and low investment, low operating cost, and the oil does not undergo solvent separation process, etc. [6].

Usually the quality and quantity of the oil obtained by mechanical extraction process are affected by various operating conditions such as pretreatment of the Neem seeds, extraction pressure, and storage condition. Effect of extraction condition on quality of oil has been investigated in several studies for wide variations of material, including conophor nut [6], olive [8], jojoba [9], and groundnut [10], and peanut kernel oil [11]. The changes of oil quality during storage also has been investigated for numerous materials, such as soybean [12], peanut kernel [11], sunflower [13], olive [8, 14], and fish oil [15]. The aim of this research was to investigate the effect of heat pretreatment on the yield and physical-chemical characteristics (saponification value, iodine value, acid number and refractive index) of Neem oil during storage.

MATERIALS AND METHODS

Preparation and mechanical extraction

Neem seeds (*Azadirachta indica*) from Bali were used in this experiment. Neem seeds were peeled and washed using water to remove dirt then dried in the open air. Dried kernels were ground into powder using seed grinder PHILIPS HR1701 for 15 seconds, to obtain fine particles without significant loss on seed's oil. Samples were taken for measurement of initial moisture content using moisture determination balance OHAUS MB200, and the result was 7.8%. Seed particles were preheated using oven MEMMERT UM400 at 40, 50, 60, 70, and 80°C until the moisture content constant.

Mechanical extraction of Neem seeds was performed using hydraulic pressing equipment ENERPAC RC-256 and P-39. Untreated seed particles were pressed with various pressures to determine the optimum pressure. Pressure was started at 2000 psi as the oil started to flow out of the seedbed, and stopped at 6000 psi since the oil yield relatively constant at the pressure above 6000 psi. Mechanical extraction was performed for 25 minutes when the oil has stopped flowing out. Oil yield measurement was conducted using mass balance PRECISA 3000D.

Oil analyses

Filtered Neem oil was stored at room temperature (30°C) in dark glass bottles for 14 days. During storage,

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physical-chemical properties of the oil were analyzed on day 0, 1, 7, and 14. Saponification value was determined using titrimetric method 9AOAC Official Method 920.160), iodine value was determined using the Wij's method (AOAC Official Method 920.159), acid number was determined using titrimetric method (AOAC Official Method 940.28) [16]. Refractive index of Neem oil was measured using refractometer ATAGO DTM-1.

RESULTS AND DISCUSSIONS

Effect of pressure and heat pretreatment on the yield of Neem oil

Yield of extracted Neem oil from untreated seed particle showed significant increase from pressure level of 2000 psi to 5000 psi (Figure-1). Similar trend was also obtained in previous studies in mechanical extraction [6, 9, and 10]. Mechanism of oil outflow during mechanical extraction was investigated by Faborode and Favier [17] in the study of oil-point, which is the threshold pressure at which oil emerges from seed kernel during mechanical extraction [17]. Seed particles will deform and more compactly fill up the empty voids at low pressure level. When the pressure increased, the voids will diminished and the seed particles begin to resist the applied pressure through contact points between particles. Further increase in pressure will forced the oil to start flowing out of the particles. In this research, the oil point was appeared to be at 2000 psi. The pressure at 6000 psi give insignificant increase on the oil yield, because empty voids between particles from which the oil could flow out were becoming smaller [10]. Therefore, 5000 psi is the optimum pressure.

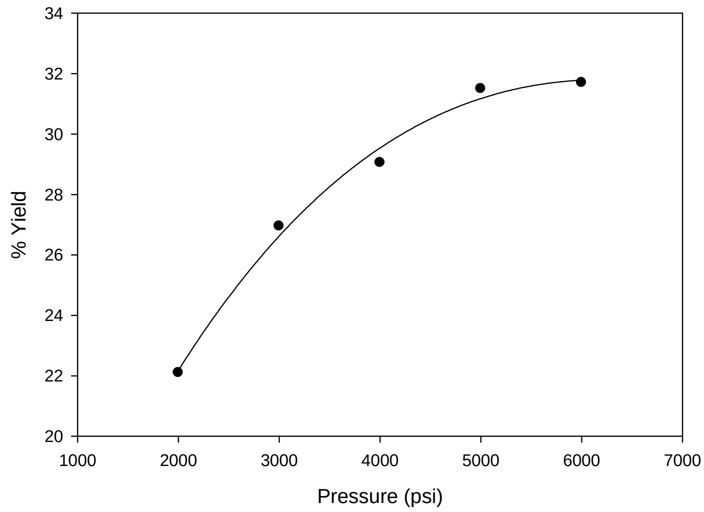
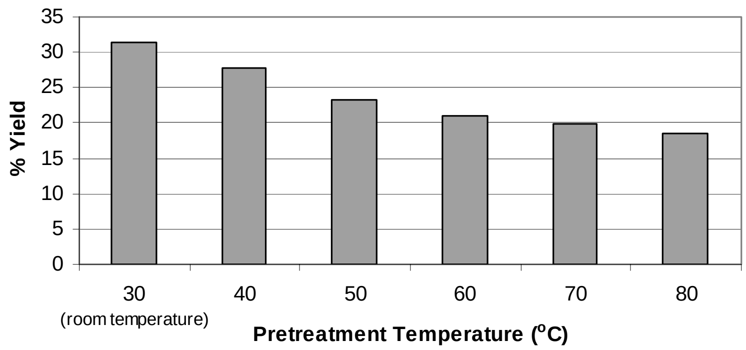


Figure-1. Yield of Neem oil extracted from untreated seed particles at various pressure.

From the plot of polynomial curve in Figure-1 using Sigma Plot 9.0 software, the following equation was obtained:

$$Y = 7.71 + 9.3.10^3 P - 1.139.10^{-6} P^2 + 4.1667.10^{-11} P^3$$

Where R^2 = 0.9926; Y was oil yield (%) and P was pressure level (psi). The equation is valid for the pressure ranged from 2000 to 6000 psi. Similar equation was also obtained in the study of jojoba oil [9]



Figur-2. Yield of extracted Neem oil for various preheating temperature.

Preheating treatment on the seed particle before pressing affected on the lower yield of Neem oil. Figure-2 showed as the preheating temperature increased, the yield of Neem oil decreased from 32% at room temperature (±30°C) to 18 % at 80°C. The decrease in oil yield caused by increasing preheating temperature was mainly related

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to the moisture content and structure of the seeds. With the increase of preheating temperature, there will be a substantial loss in seeds moisture content [6,10,17], which in turn will harden the seed particles, leading to more difficult oil flow [6,10]. In this study, the seeds moisture content after undergo preheating at 40, 50, 60, 70, and 80°C were 4.6, 3.6, 3.2, 2.9, and 2.6%, respectively. Heating could also promote binding between oil and proteins within seed structures, making the oil more attached to the seeds and cannot easily flow out of the seedbed.

Effect of heat pretreatment on the physical-chemical characteristics of Neem oil

Preheating treatment on the need seed particles influenced its chemical characteristic (Table-1). Iodine value and refractive index decreased, while saponification value and acid number increased with increasing pretreatment temperature, indicating the oil quality reduced because of oxidation and hydrolysis. There are several factors that accelerate the oxidation process, such as high temperature and metal content [7,18]. Oxidation occurs when the surface of oil is in contact with oxygen. Oxidation begins with the formation of peroxides and hydroperoxides from triglycerides [7,18,19]. During mechanical extraction, there is possibility that some amount of metal (Fe) from pressing plates were introduced into extracted oil. In this research, the Fe content in the Neem oil was analyzed using Atomic Absorption Spectrophotometer (AAS), and the result was 4.6 ppm. Fe content, along with heat pretreatment, were catalyzing further breakdown of hydroperoxides to form aldehydes, ketones, esters, alcohols, free fatty acids, and other short-chain hydrocarbons [7,18,19].

Table-1. Chemical-physical characteristics of oil at various pretreatment temperatures.

Pretreatment temperature (°C)	**Iodine value** (g/100g)	**Acid number** (mg/g)	**Saponification value** (mg/g)	**Refractive index**
30 (room temperature)	92.9032	19.6283	187.7902	1.4660
40	88.4492	21.6944	193.7209	1.4658
50	83.4609	23.2430	210.6918	1.4655
60	76.1477	25.9087	221.5138	1.4652
70	71.8393	26.9588	224.9813	1.4652
80	68.9791	28.5683	231.5669	1.4650

Hydrolysis process occurring in the oil was mainly caused by water content and lipase enzymes. When seed particles were extracted, water inside the seed is flowing out of the seedbed along with the oil, and then reacted with triglycerides to form free fatty acids and glycerols [7,18,19]. The lipase enzymes produced by several species of fungi inside the oil also accelerate the hydrolysis process [7,20]. Several species of fungi capable of producing lipase enzymes are Aspergillus, Humicola, Fusarium, Penicillium, Mucor, Monilia, Oidium, Rhizopus, and Cladosporium [7,19]. In this experiment, fungi on the surface of Neem seed and inside the extracted Neem oil were observed using OLYMPUS CX31 microscope with 400x magnification (Figure-3).

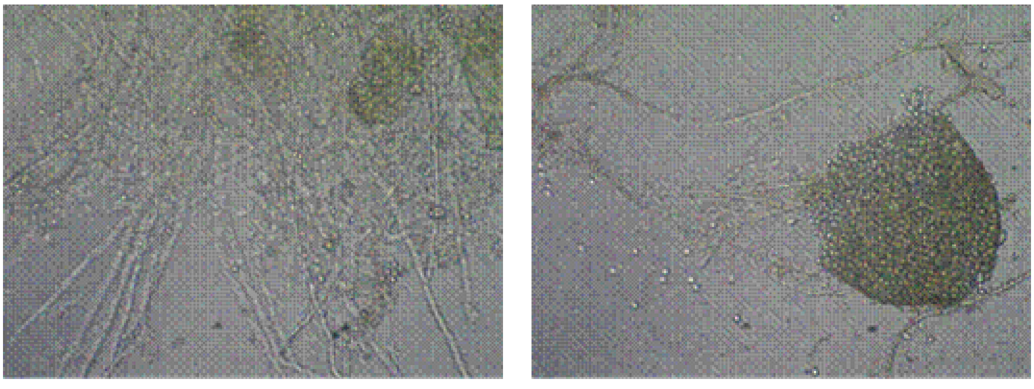

Figure-3. Microorganism that grew in the Neem seed oil (fungi), observed by OLYMPUS CX31 microscope with 400x magnification.

Oxidation and hydrolysis processes were reducing the amount of unsaturated fatty acids in the oil, therefore its iodine value reduced and acid number increased. The formations of aldehydes, ketones, esters, alcohols, free fatty acids and short-chain hydrocarbons decreased the average molecular weight of oil, which is indicated by the increasing of saponification value. Refractive index of the oil decreased as the amount of unsaturated fatty acids and long-chain hydrocarbons reduced.

Effect of storage on the physical-chemical characteristics of Neem oil

Oil quality was decreasing during storage, as indicated by decreasing the iodine value and refractive index and increasing saponification value and acid number (Table-2). These trends were similar with fish oil [15], peanut kernel oil [11] and sunflower oil [13]. Oxidation occur during storage because of the presence of air inside storage bottle and Fe content in the oil, while hydrolysis occurred because of the water content and the activity of lipase enzymes. The effect of light as a catalyst for oxidation during storage can be neglected, since the oil was stored inside dark-colored bottles and kept inside dark container.

Table 2. Chemical-physical characteristics of oil during storage.

Parameter	Day	Pre-treatment temperature					
		30°C	40°C	50°C	60°C	70°C	80°C
Iodine value (g/100g)	0	92.9032	88.4492	83.4609	76.1477	71.8393	68.9791
	1	91.3199	86.5037	81.5587	74.9451	69.1425	67.9410
	7	88.0631	84.8206	79.7397	72.5230	65.4886	61.0710
	14	79.2561	77.0707	74.6099	68.8926	60.7129	59.0132
Acid number (mg/g)	0	19.6283	21.6944	23.2430	25.9087	26.9588	28.5683
	1	19.7213	21.7511	23.7675	26.2161	27.4013	29.2850
	7	20.2508	22.3656	24.0810	26.6454	27.5268	29.6617
	14	20.1177	22.2340	23.9896	26.6893	27.2848	29.4385
Saponification value (mg/g)	0	187.7902	193.7209	210.6918	221.5138	224.9813	231.5669
	1	190.1650	207.7816	222.6497	230.7962	233.8001	237.8933
	7	214.3523	225.5659	231.5593	242.8790	254.5808	265.8126
	14	212.0483	223.0266	228.7287	237.3754	254.3687	260.8314
Refractive index	0	1.4660	1.4658	1.4655	1.4652	1.4652	1.4650
	1	1.4660	1.4658	1.4655	1.4652	1.4652	1.4650
	7	1.4655	1.4655	1.4652	1.4650	1.4650	1.4650
	14	1.4655	1.4655	1.4652	1.4650	1.4650	1.4645

CONCLUSIONS

The effect of pressing pressure on the Neem oil yield shows in the equation of

$$Y = 7.71 + 9.3.10^{3} P - 1.139.10^{-6} P^{2} + 4.1667.10^{-11} P^{3}$$

for 2000-6000 psi. The optimum pressure for Neem oil extraction using mechanical pressure was 5000psi.

The pretreatment heating on the Neem seed particles and storage caused the oil quality reduced, therefore room temperature was found to be the recommended temperature for the Neem oil extraction using mechanical process.

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