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Effect of Various Drying Methods against Biopesticide Product from Noni Leaves Extraction

Budi Santoso*, Sri Haryati, Rizky Risno Santoso, M. Fahri Abdan Syakura

Chemical Engineering Department, Engineering Faculty, Sriwijaya University, South Sumatera, Indonesia
budisantosokimia@unsri.ac.id

The frequent use of synthetic pesticides can negatively affect the environment due to the produced residues that are difficult to decompose. Biopesticide can be an alternative material to the use of chemical pesticides as it is easy to decompose in nature. The main raw materials for biopesticides are plants containing active compounds that can be toxic to pests, for example, Noni leaves. Noni leaves can be used as an feedstock for biopesticides due to their content of alkaloid compounds that can kill pests. One of the most important processes in manufacturing biopesticide is drying the raw material which aims to remove the water content as the high water content will interfere with the extraction process. This study aims to compare the content of noni leaves dried using a solar dryer and oven. As solar dryer has fluctuating heating, it is important to review the quality of the biopesticide produced. The results of testing the quality of biopesticide between the use of the solar dryer and the oven using the GCMS analysis showed that the biopesticide content was not too much different. However, the biopesticides produced by solar dryer drying had the highest contents of amine group compounds, namely Ethylhydrazine, while the biopesticides produced by oven had the highest content of carboxylic acid and ester group compounds.

1. Introduction

Many plants have problems and one of them are pests which can be harmful to many parts of plant, leaves, fruit peel, and stems. To overcome these problems, synthetic or chemical pesticides control pests (Jun Liu and Xuewei Wang, 2020). However, synthetic or chemical pesticides have harmful side effects on the environment, plants, non-animals, and even humans (Fang et al, 2019). Therefore, other alternative materials that are more environmentally friendly are needed to address the issue of pests.

Biopesticides are certain types of pecticides derived from such natural materials as animals, plants, and bacteria (Kachhawa, 2017). Biopesticides will be used as alternative product for reducing chemical or synthetic pesticides consumptions as they are easy to decompose in nature (Oguh et al, 2019). Thus, they do not cause water and environmental pollution and are not toxic to livestock, pets, or humans (N Thakur et al., 2020). Biopesticides are obtained from the extraction of plants such as dried leaves, roots, stems, and fruits (Goncalves, 2021). Noni leaves can be used as raw material for biopesticides as they contain active chemical compounds such as flavonoids, alkaloids, steroids, terpenoids, quinones, saponins, and tannins which are toxic to insect stomachs (Rahayu et al, 2021).

Natural-based pest control can be an alternative and prospective measure that has become an important component of environmentally-friendly pest control (Glare et al., 2012). Biopesticides are natural pesticides derived from organic compounds (plants, animals, or microorganisms) as raw materials for manufacture in order to inhibit growth or development and even kill pests or disease-causing organisms in plants. Lengai et al. (2018) discussed that plants and microorganisms are the most widely used raw materials in the manufacture of biopesticides as they have high bioactive components and act as antimicrobials. The active components in plants used for biopesticides include phenols, alkaloids, steroids, tannins, flavonoids, and saponins (Pedrin et al, 2011). Biopesticides can kill or interfere pests and diseases in plants by destroying the growth of eggs and larvae, inhibiting the reproduction of female insects, disrupting the respiratory and digestive systems of insects, and inhibiting the development of plant pathogens (Lengai et al, 2019).

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The use of biopesticide made from vegetable ingredients are suitable as a preventive measure against plant pests and diseases due to its antimicrobial activity that can inhibit the growth of disease-causing microorganisms in plants (Suteu et al., 2020).

Noni is the Hawaiian name for the fruit of *Morinda citrifolia L*. or known as Indian mulberry (Blanco et al., 2007). Noni plant is a tropical and wild plant that grows to an altitude of 1500 m asl (above sea level) both on fertile and marginal lands. Noni tea has been widely used in traditional medicine by the Polynesians for more than 200 years (Zin et al, 2004). Noni leaves are large, thick, single, oval in shape, and measuring between 10-40 cm in length and 15-17 cm in width. The color of the leaves is glossy green with a quite rough wavy surface.

Noni fruit has been used as a food ingredient and in traditional medicine. Studies concerning the non-volatile chemicals in Noni leaves are limited (Pino et al, 2009). Philips et al. (2020) showed that noni leaves contain anthraquinone compounds in the form of aloin and traquinone using the thin-layer chromatography method. By Haruna et al. (2020) revealed that the components of bioactive compounds of noni leaves covered tannins, steroids, saponins, flavonoids, and alkaloids. The most bioactive compound in noni leaves is tannin (Trieu ly et al, 2020). TKPI (Indonesian Food Composition Table) data published by the Ministry of Health in 2019 showed that every 100 grams of fresh noni leaves contain.9 grams of water.

Solar energy can be a potential alternative with minimum impact on the environment as it is a clean and renewable energy source (Coriolano et al, 2017). The working principle of the solar dryer is that the dried material will not be exposed directly to sunlight or is called a Controlled Sun Dryer (CSD). Plant drying using natural convection performs poorly due to low ventilation in the dryer (Afriye et al., 2011). Heat energy from sunlight will be absorbed or captured by the coating of the solar dryer which is made of glass or clear fiber plastic. The absorbed solar heat will be collected in the solar dryer and the temperature in the drying chamber is maintained. Therefore, the solar dryer works by capturing electromagnetic radiation from the sun and preventing convection (Hou et al., 2020). Drying process aims to remove the water content which can later interfere with the extraction process and to reduce the particle size which can expand contact surface area when extraction process (Liu et al., 2019).

Extraction is a separation process that consists of the separation process of a substance from a matrix (Haryati et al, 2021). Maceration is one of the extraction methods by immersing the material in organic solvents according to the active compounds which will later be taken through a low-temperature heating process or without heating (Jha and Sit, 2022). Factors affecting extraction are time, temperature, type of solvent, and particle size.

This study aims to test the quality of the biopesticides produced by using two different drying techniques, namely using a solar dryer and a laboratory oven by analyzing the compounds content of biopesticide using Gas Chromatography-Mass Spectrometry (GC-MS) analysis test.

2. Methods

Noni leaves as a raw material for producing biopesticides are obtained from Palembang City, South Sumatra Province. This study was conducted at the Process Engineering Laboratory, Chemical Industry Products, Chemical Engineering Department, Sriwijaya University. First, a total of 50 grams of Noni leaves were dried using a solar dryer for 10 hours, then the other 50 grams of noni leaves were dried using an oven for 15 hours. The tools used in this study were Memmert ovens, rotary evaporators, digital scales, solar dryers, beakers, measuring cups, spatulas, blenders, spray bottles, analytical balances, 5 ml pycnometers, vacuum pumps, and Erlenmeyer. While the materials used in this study were 1 kg of noni leaves and 96% 5L ethanol.

2.1 Procedure to Produce Noni Leave Powder

A kilogram of Noni leaves was cut into small pieces to separate the bones from the leaves. Then, Noni leaves were dried using a solar dryer and oven with 500 grams of each method. The use of a solar dryer for 15 hours during the day with a span of 3 days. Meanwhile, the other 500 grams were dried using an oven for 10 hours with a maximum drying time of 5 hours a day. The oven temperature used is 50°C and while in solar dryer average temperature is 41,23ºC . During the drying period, the water content in the noni leaves was analyzed every hour by measuring the weight of the noni leaves per hour. Meanwhile, for drying using the solar dryer, the temperature in the solar dryer room and around the environment was recorded per hour. A solar dryer works on the density differential. The inlet air hole is at the lower side for entrance cold air and outlet air is at upper side. Meanwhile, in the oven air flow work horizontally to dry noni leaves. Noni leaves that have been dried were put into a blender to grind to get a smooth powder. Increasing the productivity of biopesticides can be done by applying a cultivation model to reduce the number of experimental trials (Prakash and Srivastava, 2008).

2.2 Maceration Stage

Noni leaves powder was weighed 50 grams each. Noni leaf powder was then dissolved using 96% ethanol with a ratio of 1:2, 1:4, and 1:8 w/v. This maceration process was carried out in a beaker glass which is tightly closed using aluminum foil. It was stored for 3 days at room temperature.

2.3 Biopesticide Purification Stage

After the maceration process was carried out for 3 days, the beaker glass was opened and it was continued with a filtration process by separating the extract from the remaining noni leaf powder used in the maceration process. Filtration was carried out using a separatory funnel covered with filter paper with Erlenmeyer underneath to accommodate the results. A vacuum pump was used which is connected to the Erlenmeyer using a hose. The results of the filtration were evaporated using a rotary evaporator with an operating temperature of 70°C. The researcher set this temperature expecting that the ethanol contained evaporates without damaging the existing metabolic compounds. Evaporation was carried out until the ethanol was no longer dripping from the condenser.

2.4 Biopesticide Analysis Stage

The biopesticide analysis stage used GC-MS which was carried out at the Laboratory of the Faculty of Mathematics and Natural Sciences, Sriwijaya University. The sample was selected with a variation of 1:4 w/v for the GC-MS test. The GC-MS test aims to identify the content of secondary metabolites in the biopesticide in the form of functional groups.

3. Result and Discussion

3.1 Influences of Solvent Amount on the Total Volume of Produced Extract

The extraction of dried noni leaves powder with the help of a solar dryer and an oven used the maceration method. The powder used was 50 grams per maceration sample. Maceration was carried out for 48 hours by leaving it at room temperature (Ngibad et al, 2021). The solvent used was ethanol with varying ratios of solvent and raw material powder. Raw materials that have been dried using a solar dryer were macerated with a solvent ratio of 1:2, 1:4, and 1:8. While the raw materials dried using an oven were macerated using a solvent ratio of 1:4. The first solvent ratio for raw materials dried using a solar dryer was 1:2. A total of 100 ml of ethanol was used to extract 50 grams of noni leaf powder. The extract yield was 3.6 ml and it was not continued to the evaporation stage as the extract yield obtained was very small for further evaporation. The extract obtained for 1:4 ratios was 68 ml. The ratio of the third solvent was 1:8 and obtained 294 ml.

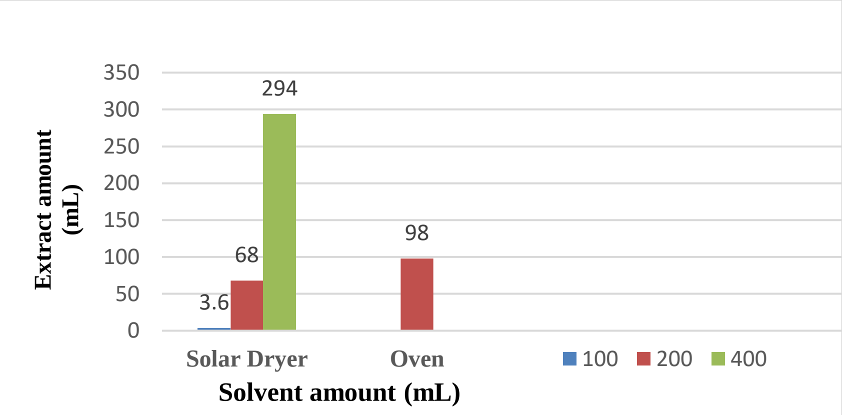

Figure 1 The Influence of Solvent Amount on the Extract Produced.

For drying raw materials using an oven, maceration was carried out using a solvent ratio of 1:4. The extract yield was 98 ml. The extract produced from a ratio of 1:4 from oven extraction is higher than the 1:4 solar dryer extraction. Drying using an oven is an ideal condition that can be adjusted for stability but requires electricity to operate (Song et al, 2019). Meanwhile, drying using a solar dryer highly depends on air conditions including temperature, speed, and humidity as a medium for heat transfer during the drying process so the drying process is relatively not constant in terms of time (Lakshmi et al, 2019).The filtered extract was evaporated for 10 hours using a rotary evaporator. The results of pure biopesticides with the 1:4 variations for solar dryers and ovens are not much different.

The raw material powder used during maceration was then dried using an oven with a temperature of 50°C. The results obtained were a reduction in the weight of the raw material powder before and after the maceration process. This indicates that the secondary metabolic compounds contained in the raw material powder have diffused and bound with the solvent used, namely ethanol. For biopesticides with a solvent ratio of 1:2 and the raw materials dried using a solar dryer obtained 47 grams of dried raw material waste. For biopesticides with a solvent ratio of 1:4 and 1:8 with raw materials dried using a solar dryer, obtained the same results, namely 41 grams. For biopesticides with a solvent ratio of 1:4 with raw materials dried using an oven obtained 41 grams. It can be concluded that for 50 grams of raw material powder, the secondary metabolic compounds that maximally diffuse were 9 grams in each solvent ratio used. Because of the researchers have found that the 1/4 w/v variation had reached the maximum extraction, so the 1/4 w/v variation sample was chosen for the GC-MS analysis.

3.2 Comparison of GCMS Analysis Results on the Quality of Biopesticide Between Using Solar Dryer and Oven

The biopesticides compared were in 1:4 of the first macerated solar dryer and oven variations. The results of the GCMS analysis show that the two biopesticides with different methods of drying have different dominating compounds that can act as pesticides.

Table 1: Data Comparison of Biopesticide GCMS Analysis Results between solar dryers and ovens

Solar Dryer		Oven	
%Area	Compound	%Area	Compound
1,28%	Ethanol	4,79%	4H-Pyran-4-one, 2,3-dihydro-3,5-dihydroxy-6-methyl
59,39%	Hydrazine, ethyl-	16,38%	Hexadecanoic acid, methyl ester
6,97%	Acetic Acid	4,22%	n-Hexadecanoic acid
6,97%	Propanedioic acid	24,73%	9-Octadecenoic acid (Z)-, methyl ester
4,84%	4H-Pyran-4-one, 2,3-dihydro-3,5-dihydroxy-6-methyl	25,06%	Phytol
2,52%	n-Hexadecanoic acid	6,00%	9,12,15-Octadecatrienoic acid, 2,3-dihydroxypropyl ester, (Z,Z,Z)-
1,76%	Phytol		
2,44%	9,12,15-Octadecatrienoic acid, (Z,Z,Z)-		

The dominating compound for drying raw materials with a solar dryer is Ethylhydrazine with an area of 59.39%, this compound has a function as an insecticide specifically targeting and has almost no impact on most non-target organisms. Other compounds that appear are acetic acid which functions as an herbicide and Propanedioic acid as a fungicide with an area of 6.97%. Meanwhile, the compound that dominates in biopesticides from oven drying is Phytol with an area of 25.06%, Phytol compounds have antifeedant abilities and belong to the terpene group which has repellent activity (Ninkuu et al., 2021). Other compounds that dominate are 9-Octadecenoic acid (Z)-, methyl ester of 24.73% and 9,12,15-Octadecatrienoic acid, 2,3-dihydroxy propyl ester, (Z,Z,Z)- of 6.00 %. This compound is a fatty acid that can inhibit microbial growth (Yang et al, 2019). In the biopesticides with solar dryer, there is a small content of Phytol of 1.76% and 9,12,15-Octadecatrienoic acid, (Z,Z,Z)- of 2.44%.

The compounds extracted were not much different between solar dryer and oven dried based on the results of the GCMS analysis. The compound 4H-Pyran-4-one, 2,3-dihydro-3,5-dihydroxy-6-methyl was 4.84% in solar dryer biopesticide and 4.79% in the oven. This compound belongs to the flavonoids which act as antioxidants and antimicrobials. The n-Hexadecanoic acid compound was 2.52% in the solar dryer and 4.22% in the oven, while the Hexadecanoic acid methyl ester was 16.38%. (Dey et al, 2020) states that the n-Hexadecanoic acid and Hexadecanoic acid methyl esters have a strong larvicidal effect.

The difference dominating compound in the content of active substances in biopesticides is caused by the different working principles of the two drying devices. The working principle of the solar dryer is natural convection with a non-vacuum drying chamber and ventilation so that free air can pass without additional tools (Tagne et al., 2020). Meanwhile, the working principle of the oven is forced convection with a closed or vacuum drying room. The results of noni leaf extract using a solar dryer obtained 30 ml less than that using an oven. This is because the drying process with a solar dryer has the disadvantage of not being able to control the amount of solar radiation that enters the drying chamber, where sunlight can degrade the phytochemical compounds contained in Simplicia (Bernard et al., 2014).

Chaves et al. (2020) explained that flavonoids and antioxidant compounds will decrease due to the influence of temperature variations during the drying process as these compounds are sensitive to light and heat. Flavonoid degradation occurs due to the breaking of molecular chains and the occurrence of oxidation reactions which cause oxidation of the hydroxyl groups and will form other volatile compounds quickly.

The formation of hydrazine compounds in biopesticide products produced from solar dryers was due to the reaction between nitrogen and hydrogen. Nitrogen was obtained from free air that passes through the drying chamber. The solar dryer has an open drying chamber so that the air entering the drying chamber can be excessive or in large quantities. Hydrogen atoms were obtained from water (H_2O) which evaporates from noni leaves in the drying chamber. The reaction between hydrogen and nitrogen forms hydrazine compounds. The formation of Ethylhydrazine occurs when noni leaf powder was macerated using ethanol for a 3-day soaking process which allows a reaction with ethanol and forms Ethylhydrazine compounds. Ethylhydrazine cannot be vaporized during the evaporation process as its boiling point is 114°C, while the operating temperature is only 70°C.

The formation of phytol compounds in biopesticide products resulting from oven drying was because phytol compounds are a group of ester compounds formed from the esterification reaction between carboxylic acids and alcohols. Carboxylic acids are formed from alcohol compounds that undergo two-stage oxidation (binding a lot of oxygen). In this case, the phenolic compounds contained in the noni leaf undergo excessive oxidation, namely the phenolic compounds bind with too much oxygen, resulting in carboxylic acids. This happens because oven drying used the principle of forced convection, namely the convection process produced by the exhaust oven which can force heat transfer quickly so a lot of oxygen is bound to phenolic compounds to produce carboxylic acids. Furthermore, an esterification reaction occurs between the carboxylic acid and alcohol, namely ethanol, and forms an ester compound (phytol). This phytol cannot be vaporized during the evaporation process as its boiling point is 204°C while the operating conditions at the time of evaporation were 70°C.

4. Conclusion

The difference in the method of drying biopesticide raw materials between using a solar dryer and an oven affects the content of the extracted metabolic compounds. Where noni leaves dried with a solar dryer produce biopesticides which contain the dominant compound of the amine group, namely ethylhydrazine. While drying with an oven produces biopesticides with dominant compounds in the ester and carboxylic acid groups, namely phytol and 9-Octadecenoic acid (Z)-, methyl ester.

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