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Changes in physical properties of natural pesticides formulated from liquid smoke during storage

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Abstract. Natural pesticides are commonly used active ingredients that are naturally non-chemical synthetic. The present work was to evaluate the changes in the quality of natural biopesticides formulated from rice husk liquid smoke during storage for 6 months. The formulation of biopesticide was carried out in the form of emulsified concentrate (EC) has 3 formulas (F1, F2, and F3). The physical properties were characterized by the observed pH, specific gravity, phase separation, particle size, and PDI (polydispersity index). The particle size tended to increase during the storage, however, PDI only F3 was increased and F1 and F2 fluctuated. Moreover, neither pH nor specific gravity changed significantly after six months of storage. pH and specific gravity ranged from 3.11 to 4.31 and 1.0179 to 1.0309, respectively. There was phase separation on all formulas, where F1 and F2 showed lower separation than F3 after 6 months of storage. The phase separation ranged from 71.42 to 92.86 %. This study provides information that natural pesticides can be stored for 6 months with slight changes in their physicochemical properties.

1 Introduction

Synthetic pesticides are widely used to prevent and control agricultural pests; however, they exhibit high toxicity and leave a residue [1]. It causes serious environmental pollution, particularly in water, air, and soil, which impacts human health and kills non-target organisms [2]. Several active compounds from a variety of plant species have been studied to discover and develop insecticides for their efficacy, biodegradability, low toxicity, and availability of base materials [3]. Most researchers are now looking for opportunities to develop environment-friendly pesticides. The need for natural pesticides has become urgently needed to protect plants from insecticides.

Biopesticides are often target-specific, harmless to beneficial insects, and do not cause air and water quality issues in the environment [4]. The advantages of biopesticide because of cheapness, environmental safety, specific target insect, and no evidence of resistance [5, 6, 7].

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The development of insecticides from active compounds of plants has been investigated, particularly in their effectiveness on specific insect targets with various modes of action [3]. The essential oils showed good potential against insecticides because of their chemically active compounds as aldehydes, phenols, alcohols, acids, ketones, terpenes, and others [8, 9]. Moreover, liquid smoke can be developed to provide an alternative to synthetic pesticides, because liquid smoke can promote plant health, thus reducing the use of synthetic insecticides and parasites [10]. Liquid smoke is produced from the thermal degradation process of lignin (up to 300 or 400˚C). It reported that it contains chemical compounds that act as antioxidants, and antimicrobials [11]. In this research, three formulas have been bioassay tested for laboratory against brown planthopper. The best effective mortality against brown planthopper was found at 10 % concentration with the formula of F1, F2, and F3 (92.67, 95.33, and 95.0%, respectively) were selected for extended storage until 6 months [12]. The present work was to evaluate the changes in the quality of natural biopesticides formulated from rice husk liquid smoke during storage for 6 months.

2 Material and Methods

2.1 Material

Liquid smoke is produced from rice husk by thermal pyrolysis at a temperature of around 300˚C. Tween 20 (as emulsifier), oleic acid, glycerol, teefol, essential oils (citronella and clove oil), and distillate water.

2.2 Preparation of natural biopesticide

The natural pesticide was formulated from the rice husk liquid smoke, essential oil, and distillate water (Table 1). The other ingredients were teefol, tween 20, oleic acid, and glycerol of 5%, respectively. All non-aqueous ingredients were placed into a beaker glass and mixed using an ultra-turax homogenizer (IKA T25 digital, Germany) for 10 minutes at a speed of 1000 rpm. Distillate water was then gradually dropped into the mixture while mixing and homogenized for 10 minutes at the same speed. Liquid smoke was subsequently added dropwise into the mixture while mixing. Homogenization was continued for 10 minutes at a speed of 300 to 500 rpm [12]. Then the product was stored for 6 months to investigate the changes in physical properties.

2.3 Particle Size and Polydispersity Index

The dynamic light scattering technique was used for the particle size analyzer and polydispersity index (PDI) (Nano-Zeta Sizer, Malvern Instruments, Malvern, UK). The sample was treated by dilution 50 times with deionized water to the required concentration [13].

2.4 Microscopic structure

The microscopic structure was analyzed by an optical microscope BX41, type RV 30 AT + mini vid linked to a digital camera with a magnification of 100×. A drop of the sample was placed on a glass slide and a cover slip was on top [14].

2.5 Phase separation

Phase separation was determined by [15]. Each formula was placed into a tube with a homogeneous shape and size. Every month the phase separation of the emulsion formula was observed. The result of phase separation was expressed by the percentage of stable emulsion as follows:

$$\textit{Stability of emulsion (\%)}= \frac{hu}{ho} x\ 100\% \quad (1)$$

whereas *hu*: high of stabile emulsion, and *ho*: initial emulsion

2.6 pH

pH was determined by pH meter Hanna.

3 Result and Discussion

The F1 formula had a milky appearance, which might be contributed by the colour of the citronella oil emulsion. However, F2 and F3 had brownish colour which was dominated by the colour of liquid smoke as well as clove oil (Figure 1).

F1 F2 F3

Fig 1. The appearance of biopesticide emulsion

3.1 Particle size

From the beginning, the particle size was around 162.0 nm to 316.7 nm, and the PDI of 0.312 to 0.419 (Figure 2). The formula F1 had the smallest particle size (162.0 nm). Formula F3 had the largest particle size, followed by F2. The particle sizes of all formulas tended to get bigger during the storage period (Figure 2). The stability of emulsion depends on the size of the particles. The smaller the dispersed particles, the emulsion will be more stable. Conversely, when particles increase in size, the system becomes unstable [16, 17].

The particle size must be sufficiently small to permit the formation of films around droplets in a dispersed phase in order to achieve optimal stability. The smaller particle size allows more particles to be distributed at the interface, which makes the emulsion more stable. However, particles that are too small can easily be removed from the interface which can cause instability in the formulation [18]. The particle size affected the pH value when the particle size increased the pH value decreased. This is because hydrogen ions in the emulsion also increased 13].

F2 had the lowest PDI and the formula and F3 had the highest. For the F1 and F2, the PDI tended to fluctuate during the storage, however, the F3 showed an increase during storage up to 6 months (Figure 2). It has been reported that colloidal systems that have a lower polydispersity index it means has higher stability because of the lower ripening rate [19].

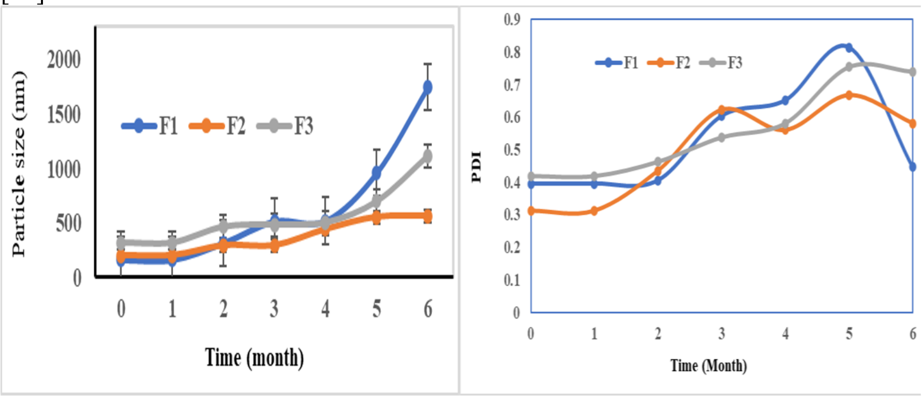
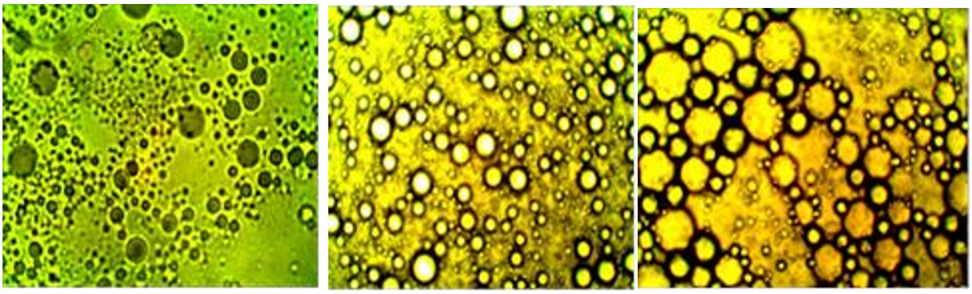


Fig 2. The particle size and PDI 0f emulsion on formulas

3.2 Microstructure

The microstructure of the emulsion was assessed using a light microscope. The majority of oil droplets were spherical in shape (Figure 3). Some droplets were larger than others and appeared irregular in shape. The microstructure showed in-line results with the largest particle size being F1 followed by F3 and F2.



F1 **F2** **F3**

Fig 3. Microstructure of emulsion under light microscope

3.3 pH and specific gravity

The pH of the formulation remained unchanged for six months, especially for the F3 (Figure 4a). At the beginning of storage, F1 has a pH of around 4.3 which is similar to the pH of F3. It suddenly decreased after one month slightly increased in two months and remained stable until six months. Interestingly, the pH of F2 was raised to 4.3 after one month of storage and declined slightly after continuous storage for up to 6 months.

The specific gravity of the formula during storage experienced a slight variation. The biopesticide's specific gravity was unchanged for the first month it was stored, but it rose after that time. F2 showed a sharp increase after 2 months of storage, then constant.

The biopesticide formula's F1 and F3 saw small increases, however, F2 remained stable for up to 6 months (Figure 4b). Based on the spraying velocity when applying pesticides, the specific gravity will have an impact on particle formation and droplet size [19].

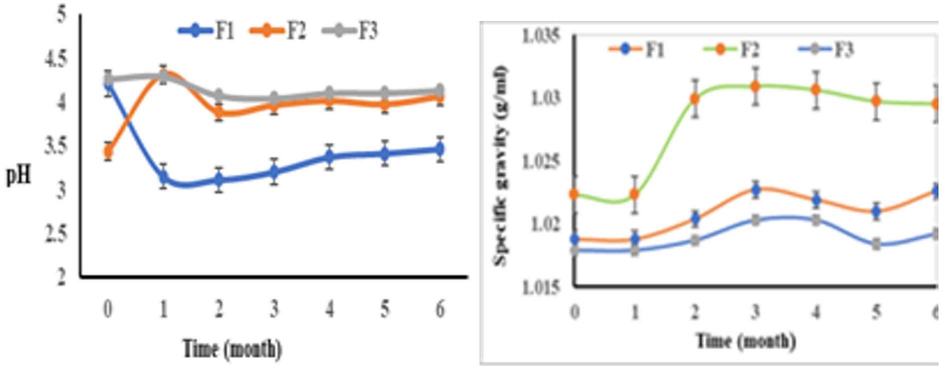


Fig 4. Changes in pH (a) and specific gravity (b) for every formula during storage

3.4 Stability of emulsion

The formula that had the greatest amount of phase separation, was followed by F1 and F2 (Figure 5). The highest separation, according to F3, occurred in the second month and persisted during the following six months. During storage, F1 showed an increased phase separation for one to two months before remaining unaltered for 6 months.

The phase separation marginally decreased with storage for two to six months in F2, which showed the opposite tendency. The destabilization that persisted may be due to the modest increase in phase separation shown in F1. During storage, the oil droplets may experience coalescence or Ostwald ripening, which will increase their size [20]. Emulsion destabilization can occur slowly or quickly depending on a variety of variables, including the initial droplet size and viscosity [21]. This investigation revealed an intriguing phenomenon: the formula with the biggest beginning oil droplet size (F1) demonstrated a shorter separation phase than the formula with the smallest initial oil droplets (F3). This might be associated with the absence of essential oil in the formulation, which might contribute to the emulsion viscosity. The increase in viscosity might be advantageous as it provides inhibition against phase separation. Most of the emulsion forms are unstable and inclined to change during storage, this is probably due to flocculation, and phase separation [20].

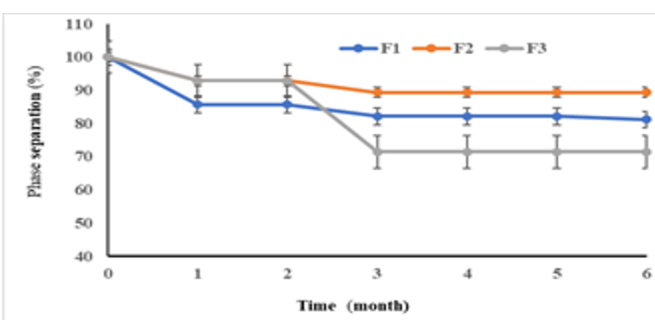


Fig 5. The percentage of phase separation of the emulsion during storage

Liquid smoke contains components derived from the thermal degradation of lignin, such as phenols that act as antioxidants, acids that act as antimicrobials, guaiacol, and its derivatives [22].

4 Conclusion

The storage of natural biopesticides in the form of emulsions was changed in particle size, pH, specific gravity, and emulsion stability. In addition, after 1 month of storage, the emulsion solution underwent a separation of phase and was stable from the second month to the sixth month.

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