Formulation of insecticide profenofos using Surfactant Diethanolamide (DEA) based on palm olein

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Abstract. Soybean is one of the major food commodities in Indonesia that the consumption is increasing each year, but this is not in line with the domestic soybean production capacity. One cause of the low production capacity is the armyworm attack. Generally, the armyworm attack controled by spread insecticide profenofos. Profenofos need to be dissolved, but profenofos couldn’t dissolved in water. So that, it need the right formulation between the solvent and other ingredients which can supprotprofenofos performance. One of that ingredient is surfactant. This research used surfactant diethanolamide (DEA) based on palm olein. DEAfunction in insecticide formulation are as homogenizer, dispersant, sticker and spreader agent. The aims of this research are to obtain the best emulsion insecticide product based on profenofos as the active ingredients and DEA as the surfactant, moreover it also to obtain information of the physico-chemical properties. The formulation test performed with completely randomized design (CRD) with two factors, first factor is DEA concentration and the second factor is profenofos concentration. Data of physico-chemical properties test was analyzed by analysis of variance (ANOVA) and significant result tested by Duncant Multiple Range Test (DMRT). The result showed that, surfactant DEA could make good emulsion between profenofos and sodium ethoxide as the solvent. The best treatment which obtain from formulation stage is concentrate with DEA 10% and profenofos 40%. Physico-chemical properties test result showed that droplet size is 1,76-2,07 μm, contact angle 11,575-24,218˚, density 0,996-0,998 g/cm³, surface tension 16,56-40,72 dyne/cm, viscosity 1,032-1,078 Cp and pH 6,87-8,22.

1. Introduction
Surfactant is a substance that has a surface active nature of molecules ampyphi lic which has two groups with different properties in a single molecule is hydrophilic group and lypophilic. Surfactants have the ability to decrease the surface tension between the two phases that have different polarity, e.g liquid-liquids, solids-liquids, gas-liquid phases. Term of surface refer to interface that one of the two phases is gas [1]. Surfactant in low concentration has ability to ensconce between two interfaces of two medias that can’t be dissolve, so that significantly change physical characteristics of the interface. Surface-active agents in surfactant has abilityto modifying the surface characteristics of a liquid or solid, to be coagulant, wetting agent, emulsifier, dispersant, adhesive, etc [2].

One of surfactant that potentially can be used to applied in insecticide to controlling the armyworm is dietanolamide (DEA). DEA will dispersing, homogenizing, leveling and sticking the active ingredient.
with other additives and the carrier media. Based on research conducted by a team of researchers SBRC in 2012, DEA has lowest surface tension value (20.97 dyne / cm) compared to other surfactants such as APG (21.22 dyne / cm), ethoxylate (23.25 dyne / cm), and lauryl betaine (31.17 dyne / cm) which is widely used in pesticides industry. Therefore, DEA potentially an increase insecticides effectiveness [3].

DEA can be produced from methyl esters and fatty acid reacted with diethanolamine. DEA on the market today produced from coconut oil and palm kernel oil (PKO), which are quite expensive. However SBRC LPPM has been able to produce surfactant DEA with palm olein that cheaper than PKO. So that it needs a research to utilize DEA application in insecticide product.

Soybean is one of the major food commodities in Indonesia that the consumption is increasing each year, but this is not in line with the domestic soybean production capacity, so the government had to import soybeans each year to full fill the domestic need. Data from the Ministry of Agriculture showed soybean production in 2015 based on Forecast Figures (FF) Central Statistics Agency (CSA) is 998,870 tons of soybean dry seeds, whereas public consumption up to 2.54 million tons of soybean dry seeds. This soy deficit caused Indonesia to import soybeans to full fill domestic soybean need. One of the factors that caused this low soybean production is armyworm attack. Armyworm (Spodopteralitura) causes loss of harvests up to 80% [4]. Armyworm pest controlling usually do by spraying profenofos insecticide. The objectives of this research are to obtain the best emulsion insecticide product based on profenofos as the active ingredients and DEA as the surfactant, moreover it also to obtain information of the physico-chemical properties.

2. Materials and Methods

2.1. Instruments and Materials

Vortex HeidolphReax top, and a rotor stator homogenizer Daihan HG-15D models, Tokebi homogenizer (22000 rpm) were tools that used in this research. Another tool used for sample analysis were pH meter (pH Meter Schott), density meter (Anton Paar DMA 4500M), potentiometer (Spinning Drop Tensiometer), viscometer (Rheometer Brookfield DV-III Ultra). The materials used in this research were surfactants diethanolamide (DEA) obtained from SBRC LPPM IPB, active ingredient was profenofos obtained from PT. Petrosida Gresik. Other materials were NaOH and 96% ethanol.

2.2. Solvent Preparation

The insecticide concentrate was prepared by mix NaOH in ethanol or natriumetoxcide to profenofos and followed by adding DEA, then they were homogenized for 5 minutes in 22000 rpm speed using Tokebi Homogenizer.

2.3. Insecticide Formulation

This stage aims to study the DEA and profenofos effect to the emulsion physical character. Physico-chemical character that tested were contact angle (°), droplet size (µm), dencity, surface tension viscosity and pH. Completely Randomized Design (CRD) were used to done the insecticide formulation which is the first factor was DEA concentration (0, 10, 15 dan 20%) and second factor was profenofos concentration (40, 50, dan 60%).

3. Results and Discussion

3.1. Solution Formulation Insecticides

Generally, pesticides formulations can be classified into two major categories, they are liquid formulations and solid formulations. Liquid formulations are usually composed by active ingredients, solvents, and additives such as emulsifiers, leveling, adhesives and others. EC (emulsifiable
Concentrate), is one type of liquid formulation that prepared by dissolving the active ingredient in a particular solvent and by adding one or more surfactants or emulsifiers [5]. Insecticide solution in this study were made in EC formulation.

Most of insecticide products in the market is produced by mixing active ingredient and additive (adjuvant). The active ingredients are toxic chemical compounds that kill the insects, but the presence of extra material is needed to increase the active ingredient action mechanism. Additional materials are often used in insecticide formulations include solvents (solvent), surfactant (emulsifier), adhesive (sticker), wetting agents (wetting agent), colorant (coloring agent) and others [5].

Surfactant is an active compound that can decrease the surface tension. Surfactants are amphipatic molecules which have a hydrophilic nature (polar), and hydrophobic nature (non-polar). This property caused surfactant can be dissolved in a solution that has two different polarity degrees and hydrogen bonds such as water and oil. Surfactants are divided into four main groups and are widely used in almost all sectors of modern industry. The types of surfactants are anionic, cationic, nonionic and amphoteric surfactants [6]. The surfactant DEA that used in this study is a non-ionic surfactant.

Profenofos has the chemical name O-(4-bromo-2-chlorophenyl) O-ethyl S-propyl phosphorothioate [6]. Profenofos belonged toxic organophosphate stomach and contact poison [5]. Refer to profenofos molecular structure, it has negative poles on Chlor and Brom, so generally profenofos has electronegativity. The big profenofos molecular structure caused it can’t be dissolved in water and can’t be evaporated in room temperature but it can receive positive group from other compound. So that, it needs a right formulation to form good insecticide emulsion. Figure 1. Shows the profenofos chemical structure.

![Profenofos Chemical Structure](image)

**Figure 1.** Profenofos Chemical Structure [6]

Refer to the negative charge, profenofos can be coated by kationic compound or a compound that have relatively positive charge, so in this research natriometixcide (NaOC2H5) have choosen. Natrium etoxide has positive pole in the natrium and also in –CH3 groups, so that natriometoxcide forming Van Der Waals bond with profenofos. –Na group that has positive charge will go to –Cl and –Br groups to forming Van Der Waals bond, it caused profenofos particle has a mantel with negative charge that formed by CH3 group of natriometoxcide. Thi mantel formed zeta potential that increase koloid stability. But this bond is not too strong, so if it diluted by water, it will detached and the emulsion come apart. So that, it need to add a surfactant that can form a new mantel, in this research palm oleindietanolamide (DEA) was used.

Ethanol group of DEA has a relatively positive charge so that DEA will be heading -CH3 of sodium ethoxide, forming a new Van der Waals bond and a new outwardmantel, it makes the distance to the outer layer of profenofos particles getting further and the colloid becomes more stable because zeta potential that formed getting larger. If the emulsion mixed into water, it will forming a water mantel from the bond of –CH3 group of DEA and water. Larger zeta potential makes more stable colloid. Figure 2. Showed DEA molecular structure.
Insecticide formulation in this research aims to study the DEA and profenofos effect to the emulsion and also its physico-chemical characters. The concentrate visual appearance that formed in this stage have transparent tawny color and specific profenofos scent that similar with very strong garlic scent. After 700 ppm of the concentrate diluted in water, the solution become white color, still has specific profenofos scent but not too strong. Figur 3 and 4. Showed the insecticide concentrate and its 700 ppm solution in water.

![Figure 3. Profenofos insecticide concentrate solution](image1)

![Figure 4. 700 ppm profenofos insecticide solution in water](image2)

The physico-chemical test results showed droplet sizes range was 1.41-2.07 μm, the contact angle range was 11.56-24.22°, the density range was 0.996-0.998 g/cm3, the surface tension range was 15.833-40.717 dyne/cm, viscosity range was 1.032-1.115 cP and pH range was 6.328-8.207. Table 1. Showed the physico-chemical insecticide characteristic test result.
Table 1. The physico-chemical insecticide characteristic test result (0.07% profenofos in water)

| Surfactant DEA (%) | Droplet size (µm) | Profenofos (%) |
|--------------------|-------------------|----------------|
|                    | 40                | 50            | 60            |
| 0                  | 1.843 ± 0.334b    | 1.770 ± 0.127b| 2.073 ± 0.046b|
| 10                 | 1.987 ± 0.287c    | 2.057 ± 0.165b| 1.950 ± 0.190b|
| 15                 | 1.777 ± 0.053b    | 1.850 ± 0.275b| 1.757 ± 0.061b|
| 20                 | 1.990 ± 0.045b    | 1.413 ± 0.074a| 1.777 ± 0.045b|

| Contact Angle      | 0                  | 10             | 15            |
|--------------------|-------------------|----------------|---------------|
| 0                  | 22.830 ± 0.238c   | 14.398 ± 0.367a| 24.218 ± 0.557c|
| 10                 | 18.477 ± 0.747b   | 13.631 ± 0.304a| 23.543 ± 0.376c|
| 15                 | 13.730 ± 0.519a   | 12.137 ± 0.292a| 11.575 ± 0.467a|
| 20                 | 11.556 ± 0.565b   | 16.413 ± 0.651b| 21.070 ± 0.356c|

| Density            | 0                  | 10             | 15            |
|--------------------|-------------------|----------------|---------------|
| 0                  | 0.996 ± 0.00004a  | 0.996 ± 0.00004a| 0.997 ± 0.00008d|
| 10                 | 0.997 ± 0.00004b  | 0.998 ± 0.00004e| 0.998 ± 0.00004f|
| 15                 | 0.997 ± 0.00009c  | 0.998 ± 0.00004g| 0.998 ± 0.00004f|
| 20                 | 0.997 ± 0.00020d  | 0.997 ± 0.00023d| 0.996 ± 0.00026a|

| Surface Tension    | 0                  | 10             | 15            |
|--------------------|-------------------|----------------|---------------|
| 0                  | 40.717 ± 1.276e   | 19.257 ± 1.138b| 32.627 ± 0.677d|
| 10                 | 27.22 ± 0.682c    | 19.390 ± 1.074b| 28.090 ± 3.275c|
| 15                 | 26.890 ± 1.553c   | 16.563 ± 0.959a| 19.570 ± 1.740b|
| 20                 | 26.793 ± 2.704c   | 15.833 ± 0.927a| 17.333 ± 0.684c|

| Viscosity          | 0                  | 10             | 15            |
|--------------------|-------------------|----------------|---------------|
| 0                  | 1.032 ± 0.003a    | 1.038 ± 0.003a | 1.040 ± 0.013a|
| 10                 | 1.078 ± 0.008b    | 1.075 ± 0.005b | 1.073 ± 0.012b|
| 15                 | 1.075 ± 0.013b    | 1.077 ± 0.008b | 1.078 ± 0.002b|
| 20                 | 1.097 ± 0.007c    | 1.115 ± 0.015c | 1.115 ± 0.015d|

| pH                 | 0                  | 10             | 15            |
|--------------------|-------------------|----------------|---------------|
| 0                  | 8.207 ± 0.038d    | 6.873 ± 0.847a | 7.787 ± 0.060b|
| 10                 | 8.153 ± 0.035d    | 7.887 ± 0.040c | 7.310 ± 0.036a|
| 15                 | 8.118 ± 0.022d    | 7.757 ± 0.045b | 7.210 ± 0.010a|
| 20                 | 7.863 ± 0.032c    | 7.860 ± 0.875c | 6.328 ± 0.080a|

Description: Figures in columns and rows followed by the same letter show no significant based DMRT at α = 0.05

3.2. Droplet size (µm)
Droplet size in this study was measured in micro unit. In emulsion system, the droplet size that have size 0.1 µm or 0.1-50 µm formed granules that dispersed well in other liquid. The smaller droplet size of emulsion means that it has good character [7]. Droplet size in this study was measured in micro unit. In emulsion system, the droplet size that have size 0.1 µm or 0.1-50 µm formed granules that dispersed well in other liquid. The smaller droplet size of emulsion means that it has good character. So that, droplet size observation was needed to ensure the insecticide emulsion is already well formed and homogen. Droplet size analysis in this stage done by diluted 700 ppm insecticide in aquadest.

Analysis of variance results showed that DEA has significant effect to the droplet size in confidence interval 95%. It means that DEA has function as mantel protector in the emulsion system with dilution
up to 700 ppm. The result also showed that profenofos has no significant effect and there were no interaction between DEA and profenofos in the insecticide solution although it has been diluted up to 700 ppm.

![Figure 5.](image)

**Figure 5.** relationship of 0.07% profenofos insecticide solution in water to the droplet size

**Information:**
A1: The addition of DEA 0%
A2: The addition of DEA 10%
A3: The addition of DEA 15%
A4: The addition of DEA 20%
B1: The addition of Profenofos 40%
B2: The addition of Profenofos 50%
B3: The addition of Profenofos 60%

![Figure 6.](image)

**Figure 6.** Droplet size distribution of 0.07% profenofos insecticide in water

Duncant Multiple Range Test (DMRT) result showed that DEA has significant effect at A2B1 treatment (20% DEA : 50% profenofos). It means that the bond of DEA-natriumetoxcide-profenofos formed stable emulsion.
3.3. Contact angle (˚)
Contact angle measured to see the insecticide solution ability to stick and spread on the soybean leaf. Insecticide products are expected to have as narrow as possible. It related to the insecticide ability to stick and spread on the object. 0˚ means there is no angle that formed so the droplet get well sticking on the object, 90˚ means that the droplet on the object can only stick but can’t spread, and 180˚ means the droplet can’t stick on the object and immediately slip [9]. Contact angle measured to see the insecticide solution ability to stick and spread on the soybean leaf. Insecticide products are expected to have as narrow as possible. It related to the insecticide ability to stick and spread on the object. 0˚ means there is no angle that formed so the droplet get well sticking on the object, 90˚ means that the droplet on the object can only stick but can’t spread, and 180˚ means the droplet can’t stick on the object and immediately slip. Contact angle of the insecticide in this research was 11.55˚-24.2˚, this range could be caused by the differences in natural absorption of the leaf. ANOVA results showed that DEA, profenofos and they interaction have significant effect to the 700 ppm insecticide in water. it could be caused by the differences of the leaf to absorbing DEA and profenofos. Figure 7. Showed realtionship between the insecticide with contact angle.

![Figure 7. relationship of 0.07% profenofos insecticide solution in water to the contact angle](image)

Information:
A1: The addition of DEA 0%
A2: The addition of DEA 10%
A3: The addition of DEA 15%
A4: The addition of DEA 20%
B1: The addition of Profenofos 40%
B2: The addition of Profenofos 50%
B3: The addition of Profenofos 60%

The significant effect that given by DEA to the insecticide in this research prove that DEA has function as dispersant, homogenizer, leveler and sticking agent in profenofos solution with natriumetoxide as the solvent. This was in accordance with [8] who said that visual observation about droplet of a solution that containing surfactant could be spreaded out and penetrater between fine hairson a leaf via it’s capilarity. Whereas, the solution droplet without any surfactant could be spreaded out and could only stucked on a leaf.
3.4 Density (g / cm³)

Emulsion system of the profenofos insecticide solution in this research was oil in water (o/w) that showed by the dispersion of oil phase into the dispersant that was water. Density is an important parameter to be analysed because density value related to surface tension value which is one of emulsion stability factor.

Density defined as the ratio of the weight of a material to the total volume of the constituent materials. Density analysis performed by using density meter. The analysis shows that the density of the insecticide solution ranges from 0.996 to 0.998 g / cm³. The results of the data analysis by analysis of variance showed that the addition of surfactant DEA and profenofos have significant effect to the density of the solution, and there was an interaction between the DEA and profenofos in the insecticide solution 700 ppm in the water. Figure 8. Shows the relationship of 0.07% profenofos insecticide in water to the density.

![Bar Chart](image)

**Figure 8.** Curve relationship of 0.07% profenofos insecticide solution in water to the density

Information:
- A1: The addition of DEA 0%
- A2: The addition of DEA 10%
- A3: The addition of DEA 15%
- A4: The addition of DEA 20%
- B1: The addition of Profenofos 40%
- B2: The addition of Profenofos 50%
- B3: The addition of Profenofos 60%

DMRT result showed that the DEA and profenofos have significant effect to density at 95% confidence interval, and there was an interaction in 700 ppm insecticide solution in the water. This results could be caused by DEA that has less density than the profenofos density, solvent and water used as a diluent so that the addition of surfactant DEA can reduce the density of 700 ppm insecticide solution in water.

3.5 Surface Tension (dyne / cm)

The surface tension of a liquid is caused by the internal pressure of affinity molecules on the surface to the bottom surface of the liquid [9]. The surface tension is formulated as $\gamma = \frac{F}{d}$, $\gamma$ is surface tension, $F$ is the force of surface tension in N and $d$ is the length or diameter with units of m so that the surface tension has units of N / m. Formula profenofos the DEA surfactant sodium ethoxide with an emulsion system so that the onset of surface tension caused by the particles in the emulsion and
solvent. Changes in surface tension is influenced by the content of particles and particle size of the emulsion surface. Curves formulations insecticide solution to the surface tension shown in Figure 9.

![Surface Tension Chart](chart.png)

**Figure 9.** Curve relationship of 0.07% profenofos insecticide solution in water to the surface tension.

**Information:**
A1: The addition of DEA 0%
A2: The addition of DEA 10%
A3: The addition of DEA 15%
A4: The addition of DEA 20%
B1: The addition of Profenofos 40%
B2: The addition of Profenofos 50%
B3: The addition of Profenofos 60%

Analysis results showed that the insecticide surface tension 700 ppm in water were 15.833-40.717 dyne/cm. ANOVA results showed that DEA and profenofos have significant effect to the surface tension and there were interaction between them.

DMRT results showed that DEA and profenofos have significant effect to the surface tension in 95% interval confidence and there were interaction between them. Profenofos in natriumetoxide with DEA is an emulsion system, so that surface tension formed by the particles in it’s solvent. Surface tension change was influenced by particles and particle size in the emulsion surface. The significant effect could be caused by DEA that could decrease the density of insecticide formula, the smaller density makes distance between particles, so that the attractive force between particles become weaker and the surface tension got decrease.

3.6 Viscosity (cP)
Viscosity is one of the fluid properties are affected by the size and intermolecular forces. Viscosity indicates the viscosity level of a fluid. The higher viscosity means the higher the viscosity level of a fluid, which indicates changes in the structure and bonding between the molecules. The increase in viscosity due to increased concentrations of particles, as well as the flow properties will depend on the viscosity and density of the liquid [7].

Armyworms controlling in the field done by spraying profenofos insecticide. One important factor that will affect the use of insecticide solution on spray tool is viscosity, the higher viscosity of the
insecticide solution will be more difficult to be sprayed, the lower viscosity of the insecticide, it will
be easier to be sprayed. Therefore, the viscosity of insecticides in this study need to be observed.
Data analysis results show that the viscosity of the insecticide were 1.032 to 1.115 cP. The results of
the data analysis by ANOVA showed that DEA has significant effect to the viscosity, but profenofos
has no significant effect and there is no interaction between them. Curves on viscosity formulations
insecticide solution is shown in Figure 10.

![Figure 10. relationship of 0.07% profenofos insecticide solution in water to the viskosity](image)

Information:
A1: The addition of DEA 0%
A2: The addition of DEA 10%
A3: The addition of DEA 15%
A4: The addition of DEA 20%
B1: The addition of Profenofos 40%
B2: The addition of Profenofos 50%
B3: The addition of Profenofos 60%

High viscosity due to high particle concentrations as well, so the flow properties of materials depend
on the liquid viscosity and density. Liquid that easy to flow is the liquid that has low viscosity and
materials that difficult to flow are the material with high viscosity. The test further results by DMRT
showed that the addition of surfactant DEA significantly different with the viscosity at 95% confidence interval, this can be caused by surfactants DEA that has a high viscosity compared with viscosity profenofos and solvent, so that the treatment the addition of surfactant DEA has a significant effect on the viscosity. This is also supported by previous data, the addition of surfactant DEA significantly affect the density, which is one factor of viscosity grades. In addition, the insecticide solution can be seen also in this study had a small droplet size, so it can be said emulsion insecticide solution has a small particle size, the smaller the particle size, the surface area for greater frictional forces so that the viscosity becomes.

3.7. The degree of acidity (pH)
Values of pH or acidity is used to express the degree of acidity or alkalinity of pH. pH also relates to
the concentration of hydrogen ions, as a component of acidity and concentration of hydroxyl ions as a
component of alkalinity (Rondinini et al.2001). At neutral pH conditions, the concentration of the two
ions balanced but if the hydrogen ion concentration is greater than hydroxyl ions then tend to acidic
pH (lower). In general, the pH value of a substance in the range of grades 0-14. The range of pH values of 0-6 indicates an acidic substance, while the range pH 8-14 shows the alkaline material [7]. The results of data analysis showed that the pH of the resulting insecticide solution ranges from 6.328 to 8.207, but the data can be seen that the insecticide solution has tended toward alkaline pH, ie a pH greater than 7.

pH is the degree of acidity, expressed as -log [H+] and therefore in this study, the pH value of the data is transformed into a logarithm form in order to obtain the value of [H+] prior to analysis of variance and a further test. Curves formulations insecticide solution against pH shown in Figure 11.

![Figure 11. Curve relationship of 0.07% profenofos insecticide solution in water to the pH](image)

Information:
A1: The addition of DEA 0%
A2: The addition of DEA 10%
A3: The addition of DEA 15%
A4: The addition of DEA 20%
B1: The addition of Profenofos 40%
B2: The addition of Profenofos 50%
B3: The addition of Profenofos 60%

The results of the data analysis by ANOVA showed that the addition of surfactant DEA and active ingredients profenofos gives significant effect on pH and there is interaction between the two. Further test results by DMRT showed that the addition of surfactant DEA and active ingredients profenofos significant effect on the pH of the 95% confidence interval, and there was an interaction between them. It can be caused by DEA surfactant that has alkaline properties that can raise konsektrasi [H+] in insecticide solution so that the pH becomes alkaline.

4. Conclusion
The best insecticide solution obtained from this study is the insecticide solution with an active ingredient profenofos 40% in solvent and surfactant sodium ethoxide DEA 10% (A2B1). Chemical physical properties possessed by this insecticide solution that droplet sizes ranging from 1.41 to 2.07 μm, the contact angle ranged between 11.56 - 24.22°, the density ranges from 0.996 to 0.998 g / cm3, the surface tension ranges 15.833-40, 717 dyne / cm, 1.032 to 1.115 cP viscosity and pH ranged from 6.328 to 8.207.
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