

## **Investigation of Polyvinyl Alcohol/Clove Essential Oil Property as a Repellent Against Corn Leaf Aphids (*Rhopalosiphum Maidis F.*)**

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DOI: <https://doi.org/10.30880/rpmme.2021.02.02.052>

Received 02 Aug. 2021; Accepted 27 Nov. 2021; Available online 25 December 2021

**Abstract:** Clove essential oil have become immensely popular over the past decade as it is high in antioxidants, antiviral and antifungal properties. This research is to study the ability of a polymer based product which were made from polyvinyl alcohol (PVA) and clove essential oil (CEO) whether it can repel the presence of insects towards certain crop plants or not. The objective of conducting this research is to study the effect of clove essential oil on mechanical properties of PVA as well as to evaluate the aphid behaviour of repellency towards the prepared materials which the samples were prepared through casting method. The casting process were done by dissolving CEO in 100 ml PVA solution at 80°C and then were left dry at ambient temperature. The analysis was conducted through tensile test, solubility, biodegradability as well as repellency test. From the results obtained, it stated that for tensile test, the lower the oil concentration in the prepared sample, the higher the average length different produce thus indicates that it has a higher tensile strength. As for solubility test, it is shown that, the sample that has the right ratio of PVA composition and oil concentration, gives a better structural advantages as it has both tensile strength and hydrophobic properties and thus will help the sample to maintain their shape better during being immersed in water. The same concept applied for biodegradability test, the result stated that, the sample that has the right ratio of PVA composition and oil concentration, help the sample to maintain their shape and prevent them from degrades better. Lastly, for repellency test it stated that, the higher the oil concentration in the prepared sample, the higher the percentage of repellency and thus the longer the repellency process can withstand.

**Keywords:** Clove Essential Oil, Polyvinyl Alcohol, Insect, Repellency.

## 1. Introduction

Polyvinyl alcohol (PVA) is a non-toxic, hydrophilic, and highly polar synthetic biodegradable polymer. PVA is an environmentally friendly material which can be easily degraded to carbon dioxide and water [1]. One of its specialities is that it is effective in film forming, emulsifying and has an adhesive quality. PVA is used in a variety of applications including in the medical application due to its biocompatibility, low tendency for protein adhesion and also because of its low toxicity characteristic. Nowadays, the PVA-based polymers are being widely used in additive manufacturing and in certain countries such as in Japan, its major use is to produce a Vinyon fiber. Vinyon is a manmade heat and chemical resistant fiber (also called Juche fiber) fabricated from PVA and is widely used in textiles, quilts wadding, ropes, and shoes [2].

### 1.1 Polyvinyl Alcohol and Clove Essential Oil Application

During the last century, many applications were developed with PVA in different sectors, such as food and medicine, yielding products such as lacquers, resins, surgical threads and food packaging materials [3]. PVA is also used in paper coating and textile sizing as it is a biodegradable imitation of a natural polymer. This polymer is widely used by blending with other polymer compounds such as biopolymers and other polymers with hydrophilic properties [4]. PVA is commonly utilized for various industrial applications to enhance the mechanical properties of films because of its hydrophilic properties and compatible structure. Some man-made polymers, which are made from non-renewable and non-biodegradable sources, such as PVA, are available [5]. PVA is a widely used thermoplastic polymer that is benign to living tissues, harmless, and nontoxic. This polymer is widely investigated because of its use in cross-linked products and nanofillers [6]. As for the application of clove oil, clove bud oil (CBO) is an important essential oil used as traditional medicines owing to its enormous medicinal properties such as, antiviral, antimicrobial, antiseptic and antifungal properties [7]. Clove is also an important incense material in Chinese and Japanese culture. Clove essential oil is used in aromatherapy and clove oil is widely used to treat toothache in dental emergencies. Essential oils (EOs) are very interesting natural plant products and among other qualities, they possess various biological properties. Essential oils are the important raw materials of the fragrance and aroma industry. They are, also used in the food and pharmaceutical industries due to their therapeutic, antimicrobial and antioxidant activities. Nevertheless, they have biological activities that make them suitable for use, as herbicides, pesticides and anticancer compounds [8].

### 1.2 Efficiency of Herbal Insecticides against Aphids

Several studies show that essential oils are one of the effective ways that are environmentally safe to be used as insect repellent against the aphid species. Among the well-described examples of essential oils with insecticidal including repellency activities, promising efficacies have been demonstrated for essential oils extracted from clove, *Syzygium aromaticum* [9]. From one of the previous studies the result shows that they were able to use the essential oil against the aphids. Despite the promising findings for the clove essential oil at computational and laboratory levels, it is worth to take into consideration that clove essential oil or its major components (e.g., eugenol) can exhibit intense and persistent organoleptic qualities that could taint any food crop to which they are applied [9]. Recent study has also indicated that the essential oil has been tested on the aphid species. The present study reports on the chemical composition and biological activity of essential oils from selected Asteraceae plants, such as *A. millefolium*, *A. absinthium*, *S. chamaecyparissus*, *Tagetes patula*, and *T. vulgare*, towards the adult females of the green peach aphid [10].




## 2. Materials and Methods

Methodology is the steps used to conduct this study. This chapter discusses all the step used from the early stages of research carried out to the earnings results. To produce the samples that is needed in this research, some experiments have successfully been done. The experiments included was the process of preparing material called as casting method. After the samples was done, step to determine all the physical and mechanical properties of the produced have been tested through structured analysis and testing. For this study, polyethylene glycol have been used as plasticizer and the clove essential oil were obtained from shop

### 2.1 Materials Preparation

The main material used for this research are the Polyvinyl Alcohol (PVA), Clove Essential Oil (CEO) and Polyethylene glycol (PEG) which act as the plasticizer. The CEO used were consist of five different concentration and they have been diluted in the PVA solutions. The material mentioned are shown in Table 2.1 below.

**Table 2.1: Materials used**

| Material                 | Form   |
|--------------------------|--|
| Polyvinyl Alcohol, PVA   |   |
| Clove Essential Oil, CEO |  |
| Polyethylene Glycol, PEG |  |

### 2.2 Preparation of PVA/CEO Films

Before preparing the required sample from the main materials, the percentage by weight (wt%) of PVA and the desired concentration of the clove essential oil were decided referring to the Table 2.2

**Table 2.2: Show the ratio of PVA and Oil**

| Sample | Ratio of desired<br>PVA and Oil<br>PVA:Oil (wt%) | Polyvinyl<br>Alcohol<br>PVA (g) | Clove<br>Essential Oil<br>(ml) | Polyethylene<br>glycol (ml) |
|--------|--|---------------------------------|--------------------------------|-----------------------------|
| M1     | 100:0  | 4.0                             | 0.00                           | 1.5                         |
| M2     | 80:20  | 3.2                             | 1.00                           | 1.5                         |
| M3     | 75:25  | 3.0                             | 1.25                           | 1.5                         |
| M4     | 70:30  | 2.8                             | 1.50                           | 1.5                         |
| M5     | 60:40  | 2.4                             | 2.00                           | 1.5                         |

For this research, the solutions were prepared by using 4% (wt%) Polyvinyl Alcohol and 5% (wt%) of Clove Essential Oil. The same amount of Polyethylene Glycol 1500 (37.5wt% of polymers) were added as a plasticizer. Firstly, the required amount PVA powder as well as Clove oil were weighted using the digital balance. Figure 2.1 below show the digital balance used for this process.

**Figure 2.1: Digital balance used for weighing process**

After that, the solution were prepared by dissolving the weighted amount of PVA powder into 100 mL of distilled water. The solution were then heated in microwave to help the PVA powder dissolved in the distilled water. Figure 2.2 below show the heating process.

**Figure 2.2: Heating process using Microwave**

Then, the heating process were continued using the Hotplate Magnetic Stirrer at controlled temperature of 80°C. During the process, the required amount of Clove essential oil were added as well as the Polyethylene glycol. The stirring process were done using moderate speed to prevent any bubbles present in the solution and the process were done about half an hour to make sure the oil binds with the PVA correctly. Figure 2.3 below show the process of stirring.



**Figure 2.3: Stirring process using Hotplate Magnetic Stirrer**

After the stirring process finished, 20 ml of the solutions were then poured into petri dish. After that, the samples were left to dry at ambient temperature. After a few days, the samples then were ready to be used for testing. Figure 2.4 below shows one of the sample solution in petri dish.



**Figure 2.4: Drying process one of sample**

### 2.3 Tensile Test

The material prepared were tested respect to its tensile strength. The purpose was to find the tensile strength for each of the samples which were represented by the elongation at break. For the testing, the films were cut into strips of 10 mm wide and 60 mm long. The thickness of the sample was 1 mm. Both end of each samples were punctured to create a hole for grip purpose. Figure 2.5 below show the sample after being cut into required dimensions.



**Figure 2.5: The sample after being cut**

During the testing, certain weight were applied by hung them at the bottom end of the sample. The amount of weight were added until the sample ruptured. The initial length and length at rupture point as well as the total weight applied was recorded for analysis purposed. Figure 2.6 below show the setup of the testing and Figure 2.7 show the process during the testing.



**Figure 2.6: Preparation for tensile test**



**Figure 2.7: Process for tensile test**

### 2.3 Solubility Test

The sample sets then were tested respect to its solubility towards water. For Solubility Test (ST), the samples were cut into 50 mm wide and 50 mm long. Each samples were placed in a small bottle containing plain water. The samples were left immersed in the water for about 20 days and after that, any changes on its physical appearance as well as its physical properties were analyzed and recorded. Figure 2.8 below show the samples preparation for the testing.



**Figure 2.8: Preparation for solubility test**

### 2.4 Biodegradability Test

Another test for the samples are the biodegradability test. Each of the sample set were tested whether it degrade or not when exposed to the soil medium. For Biodegradability Test (BT), no specific dimension were set as only the leftover of the samples were used. The leftover were obtained from the cutting process of the samples used in the tensile test. Each samples were placed in a half cut bottle containing soil as the testing medium. The depth of the samples placed in the soil from the surface were set at 5 cm. The samples were left embedded in the soil for about 15 days. After that, any changes towards its physical appearance were analyzed and recorded. Figure 2.9 below show the samples preparation for the testing.



**Figure 2.9: Preparation for Biodegradability test**

## 2.5 Repellency Test

Firstly, before the repellency test (RT) can be done, the plants were planted for the experiment setup location. The plants were kept grow from seedling until it grow mature enough for about two month. Figure 2.10 below show the plants being planted at early stage.



**Figure 2.10: The plants used for testing**

After few weeks, a few selected plants were transplanted into a long pot for growth purpose. During the start of experiment day, the repellency test were evaluated by placing two samples at the plants having aphid population. Each of the sample were placed separated which one of them was the control sample and the other one was the observed sample. The samples were then left for three days for observation purposed. Figure 2.11 below shows one of the set up experiment for the repellency test.



**Figure 2.11: Preparation for repellency test**

After three days, the conditioned of the aphids were recorded. Then, the next sample were placed for the next data collection and respectively for other samples. The number of aphids landing will be counted. The repellency was indirectly calculated using the formula:

$$\text{Repellency, } R (\%) = 100 - \left( \frac{C-T}{C} \right) \times 100 \quad (\text{Equation 2.1})$$

Where C is the number of aphids landing for controlled number of aphids and T is the number aphids landing for testing result.

## 3. Results and Discussion

This chapter discusses the results of the experiment that have been conducted. From these gathered data, the characterization of samples could be determined. Some testing have been conducted to analyze the characteristic of the prepared sample. The mechanical and physical properties of the prepared samples were analyzed through tensile testing, solubility, biodegradable as well as insect repellency testing. All the sample were prepared through the procedure stated in Chapter 2.

### 3.1 Tensile Test Results

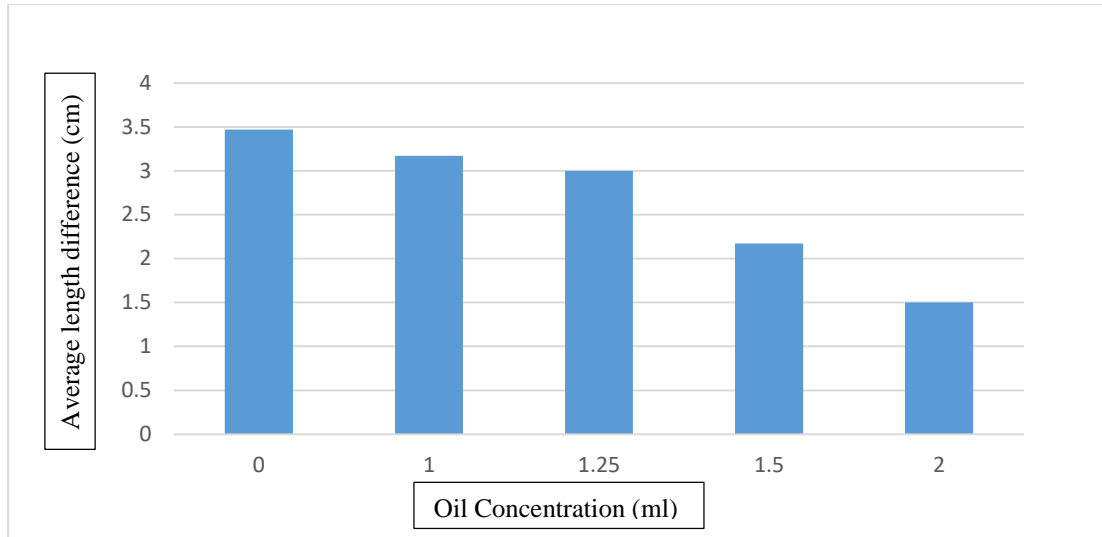
The results for the tensile test for this experiment are obtained by finding the elongation at break for each samples. The data recorded are the total weight applied during the sample ruptured as well as the final length during elongation. The test was performed three times for each concentration of oil samples. The data then were calculated to find the difference in length for each set of samples as well as it average values for length difference and mass applied. The data for all the samples for each oil concentration have been recorded. The value obtained are listed in Table 3.1 shown below.

**Table 3.1 Tensile test results for samples prepared**

| Oil concentration(ml) | Sample | Length (cm) |       | Mass during point break (g) | Average length different (cm) | Average mass applied (g) |
|-----------------------|--------|-------------|-------|-----------------------------|-------------------------------|--------------------------|
|                       |        | initial     | final |                             |                               |                          |
| 0.00                  | A      | 4.0         | 7.5   | 540                         | 3.47                          | 536.67                   |
|                       | B      | 4.0         | 7.4   | 530                         |                               |                          |
|                       | C      | 4.0         | 7.5   | 540                         |                               |                          |
| 1.00                  | A      | 4.0         | 7.2   | 360                         | 3.17                          | 356.67                   |
|                       | B      | 4.0         | 7.2   | 360                         |                               |                          |
|                       | C      | 4.0         | 7.1   | 350                         |                               |                          |
| 1.25                  | A      | 4.0         | 7.0   | 280                         | 3.00                          | 280.00                   |
|                       | B      | 4.0         | 6.9   | 270                         |                               |                          |
|                       | C      | 4.0         | 6.8   | 270                         |                               |                          |
| 1.50                  | A      | 4.0         | 6.2   | 220                         | 2.17                          | 216.67                   |
|                       | B      | 4.0         | 6.2   | 210                         |                               |                          |
|                       | C      | 4.0         | 6.1   | 220                         |                               |                          |
| 2.00                  | A      | 4.0         | 5.6   | 140                         | 1.50                          | 136.67                   |
|                       | B      | 4.0         | 5.5   | 130                         |                               |                          |
|                       | C      | 4.0         | 5.4   | 140                         |                               |                          |

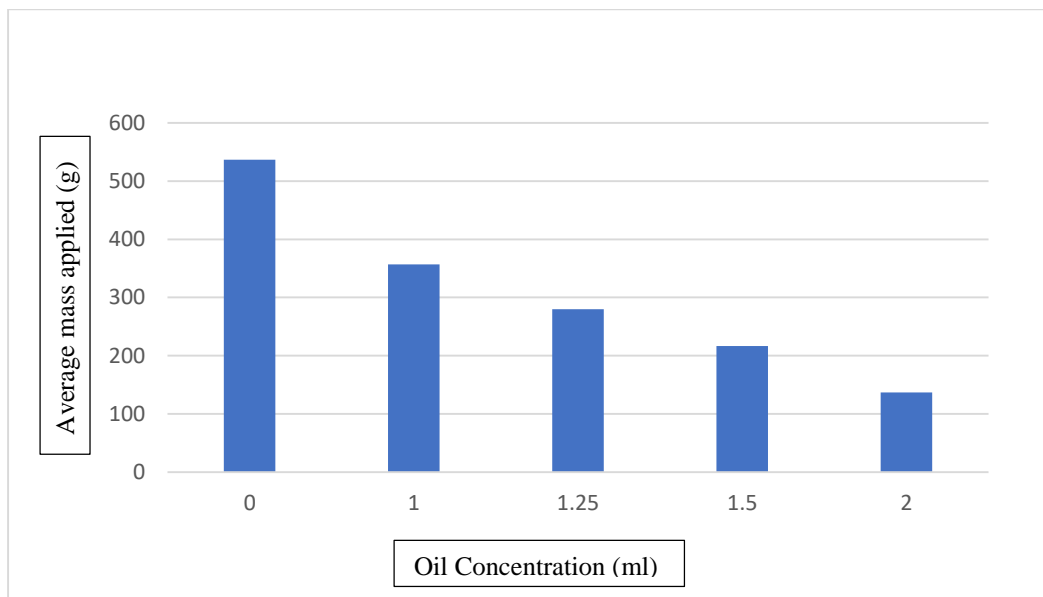
The results of the tensile test as recorded in the table above was analyzed through two bar graphs as shown in Figure 3.1 and Figure 3.2 below. The graph of average length different versus oil concentration of prepared samples is plotted and the differentiation of the five oil concentration can be made. The same concept applied for the second graph. The graph of average mass applied versus oil concentration is plotted and the differentiation of the five concentration can be made.





**Figure 3.1 Bar chart for average length different vs oil concentration**

The graph plotted in Figure 3.1 shows the relationship between the average length different with the oil concentration in the samples produced. The sample with 2.0 ml of oil concentration has the lowest length different with the value of 1.5cm on average. The highest value from the graph is the sample that has 0.0 ml of oil concentration. The value of length different is affected by the ratio of PVA and CEO in the samples. If the samples has a higher value of oil concentration, then it has a lower value of PVA. Having higher value of PVA will give a structural strength to the sample and thus making it stronger. The stronger the sample, the higher the value of length different produce. Thus, the graph can be observed that when the sample has a higher value of oil concentration compared to PVA by ratio, the lower value of length different on average produce. In other words, the lower the oil concentration in the prepared sample, the higher the average length different produce thus indicates that it has a higher tensile strength.




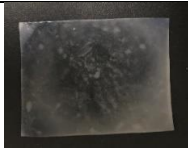



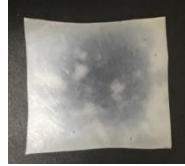
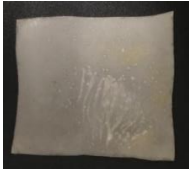

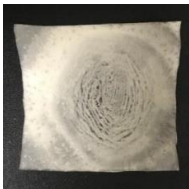

**Figure 3.2 Bar chart for average mass applied vs oil concentration**

As for the graph plotted in Figure 3.2, it shows the relationship between the average mass applied with the oil concentration in the samples produced. The sample with 0.0 ml of oil concentration has the highest mass applied with the value of 536.67 g on average followed by having the second highest value for 1.0 ml oil concentration with the value of 356.67 g. The same concept applied for this graph as the value of mass applied is affected by the ratio of PVA and CEO in the samples. If the samples has a lower value of oil concentration, then it has a higher value of PVA. Having higher value of PVA will give a structural strength to the sample and thus making it stronger. The stronger the sample, the higher the value of mass can be applied. The mass applied represent the tensile strength the sample can withstand and also indicates the point of elongation at break. If the sample required a greater load to reach it elongation at break, thus it shows that it has a higher tensile strength. Thus, the graph can be observed that when the sample has a lower value of oil concentration compared to PVA by ratio, the higher value of mass applied on average produce.

### 3.2 Solubility Test Results

To measure whether the sample dissolve in water or not, the solubility test has been done. This test was conducted for a duration of 20 days for each samples of oil concentration. The result for observation for each sample are shown in Table 3.2

**Table 3.2 Physical changes after solubility test of prepared samples**

| Oil concentration (ml) | Before  | After 20 days   |
|------------------------|---|---|
| 0.00                   |  |  |
| 1.00                   |  |  |
| 1.25                   |  |  |
| 1.50                   |  |  |
| 2.00                   |  |  |











From Table 3.2, it can be observed that after being immersed in water for 20 days, all the sample did not completely dissolve and disappear. But, the data shows that, the sample that has no concentration

of oil with a value of 0.0 ml, has the obvious changes toward its physical changes. The sample become more clearly than before but still manage to maintain its regular shape. It is known that, PVA has the properties of showing high tensile strength and flexibility as well as it is soluble in water. Thus, this shows that, the sample that has completely composition of PVA react well towards the water thus showing the characteristic of the PVA. Apart from that, the ability of the sample to maintain it shape may be due to the fact that the sample were plasticized by the PEG. The presence of PEG in the composition of the sample effectively increase the chain mobility between the PVA and CEO. As for the samples that has oil concentration, the lowest oil concentration with a value of 1.0 ml shows less physical changes compared to other. On the other hand, the sample with the value of 2.0 ml oil concentration is the most clearer sample compared to other sample that has oil concentration after being immersed in water after 20 days. The sample having more oil concentration composition means that it has the lowest PVA concentration composition by ratio. Thus, the sample will carry less characteristic of the PVA and vice versa. It can be concluded that, the sample that has the right ratio of PVA composition and oil concentration, gives a better structural advantages as it has both tensile strength and hydrophobic properties as it will help the sample maintain their shape better.

### 3.3 Biodegradability Test Results

The purpose of this test is to measure whether the sample degrade in soil or not. Thus, the biodegradability test has been done. This test was conducted for a duration of 15 days for each samples of oil concentration. The result for observation for each sample are shown in Table 3.3

**Table 3.3 Physical changes after biodegradability test of prepared samples**

| Oil concentration (ml) | Before  | After 15 days   |
|------------------------|---|---|
| 0.00                   |  |  |
| 1.00                   |  |  |
| 1.25                   |  |  |
| 1.50                   |  |  |
| 2.00                   |  |  |

From Table 3.3, it can be observed that after being exposed and planted in the soil medium for 15 days, all the samples are able to maintain their shape and did not completely degrade and disappear.

From the table, we can see that, sample that has the highest value of oil concentration shows the obvious colour changes. The colour of the sample changed from clear white into light brownish after being exposed to the soil. This is due to the fact that during the experiment being conducted, the samples were watered every day and thus some of the sample absorbed the colour of the soil. The sample that has the highest oil concentration ratio has the lowest composition of PVA. Thus, the sample eventually carried lesser PVA characteristic which is, it is insoluble towards organic solvent. This is the reason why the sample that has 0.0 ml oil concentration composition did not shows any changes towards the colour but the sample that has the highest oil concentration composition shows the obvious colour changes. Apart from that, the same concept applied for this test is that, the ability of the sample to maintain it shape may be due to the fact that the sample were plasticized by the PEG. The present of PEG in the composition of the sample effectively increase the chain mobility between the PVA and CEO and thus, all the samples did not degrades towards the soil even being exposed for a long duration. Thus, it can be concluded that, the sample that has the right ratio of PVA composition and oil concentration, gives a better structural advantages and the properties of insolubility towards organic solvent and eventually help the sample to maintain their shape more.

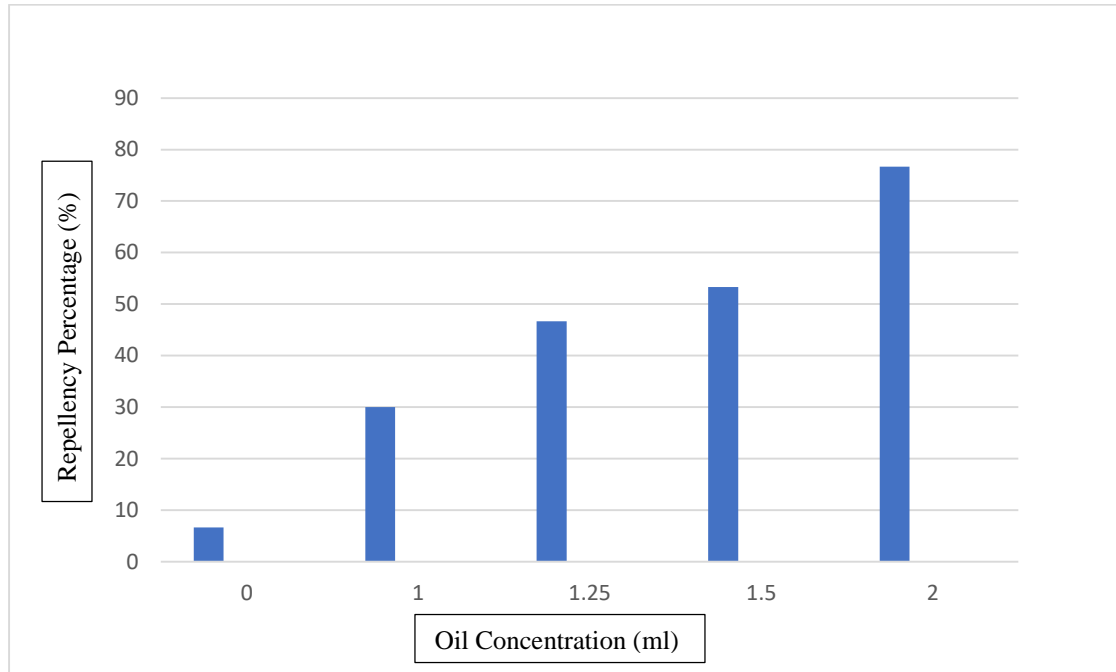
### 3.4 Repellency Test Results

The purpose of this test is to measure to measure the ability of prepared sample to repel the presence of insect towards selected plant, the repellency test have been done. This test was conducted for a duration of three days and after that, the data was collected. The data recorded are the total number of aphids landing for each sets of oil concentration samples. Then, equation 2.1 in chapter 2 is used to calculate the repellency percentage of the samples. The control value for this experiment is 30 numbers of aphids. The value obtained are listed in Table 3.4 shown below.

**Table 3.4 Repellency test results for samples prepared**

| No. | Oil concentration (ml) | Days | Number of aphids landing | Total number of aphids landing | Repellency, R (%)<br>$R = 100 - \left(\frac{C-T}{C}\right) \times 100$ |
|-----|------------------------|------|--------------------------|--------------------------------|--|
| 1.  | 0.00                   | 1    | 0                        | 2                              | 6.67   |
|     |                        | 2    | 1                        |                                |  |
|     |                        | 3    | 1                        |                                |  |
| 2.  | 1.00                   | 1    | 4                        | 9                              | 30.0   |
|     |                        | 2    | 4                        |                                |  |
|     |                        | 3    | 1                        |                                |  |
| 3.  | 1.25                   | 1    | 6                        | 14                             | 46.67  |
|     |                        | 2    | 5                        |                                |  |
|     |                        | 3    | 2                        |                                |  |
| 4.  | 1.50                   | 1    | 7                        | 16                             | 53.33  |
|     |                        | 2    | 5                        |                                |  |
|     |                        | 3    | 4                        |                                |  |
| 5.  | 2.00                   | 1    | 10                       | 23                             | 76.67  |
|     |                        | 2    | 8                        |                                |  |
|     |                        | 3    | 5                        |                                |  |

The results of the repellency test as recorded in the table above was analyzed through a bar graph as shown in Figure 3.3 below. The graph of repellency versus concentration of oil is plotted and the differentiation on the five different concentration can be made.



**Figure 3.3 Bar chart for repellency percentage vs oil concentration**

For this testing, the graph plotted in Figure 3.3 shows the relationship between the repellency percentage towards aphids against the oil concentration in the samples produced. From the graph we can see that the sample with 2.0 ml of oil concentration has the highest percentage of repellency towards the insects with a value of 76.67%. The gap difference between the repellency percentage for oil concentration of 1.25 ml and 1.5 ml are not too far compared to 1.5ml and 2.0 ml oil concentration with a value of 6.66% to 23.34%. It is clear that, the higher the value of oil concentration, the higher the percentage for repellency. This is due to the fact that, the sample that has higher oil concentration composition carries better characteristic of the clove oil. Referring to the Table 4.4, we can see that the number of aphids landing decreasing for each day. This is because the samples have the odour characteristic which came from the oil and the odour will not last long. This mean that, the longer the sample were exposed, the lesser the number of aphids falling by day but having a higher clove oil concentration will give the sample the ability to last longer and thus more aphids can be repel. Thus we can conclude that, the higher the oil concentration, the higher the percentage of repellency and the longer the repellency process can withstand.

#### 4. Conclusion

The findings and analysis from previous chapter can be concluded that the objectives of this study are achieved. The mechanical properties of the sample are determined by conducting tensile test. From the results that has been discusses in chapter 3, it is stated that if the sample have a higher ratio of PVA composition, the value of average length different as well as the average mass applied also increases. This indicated that the sample have a higher tensile strength and flexibility. The analysis for physical properties of the prepared samples also have been done through solubility test as well as biodegradability test. Through both test, all the samples are not soluble and degrade and the right ratio

of oil concentration with PVA is the best choice as it gives a better structural strength as well as carries more hydrophobic properties in the sample. Lastly, for repellency test, the comparison of five oil concentration ratio with PVA shows that, the sample that has higher oil concentration gives a better result as the number of aphid landing is higher. The number of aphid landing decreasing by day as the sample slowly loses the odours of the oil. Each sample shows the same pattern but the highest oil concentration gives a longer duration and stronger odours. It can be said, the higher the oil concentration used, the higher the repellency percentage and thus able to repel longer. Since the results produced for the tensile test are not fully success, some solution needs to be considered as an improvement for this research field. The improvements is needed to gets a better result. Thus, the first recommendation is that the parameter of this study can be improved by using the real weight instead of self-made weight. Then, the testing of the sample can be done using the actual mechanical testing machine to identify more accurate tensile strength.

### Acknowledgement

The authors wish to thank to the Faculty of Mechanical and Manufacturing Engineering, Universiti Tun Hussein Onn Malaysia that has supported on the accomplishment of research activity.

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