Pesticidal Potency of a Newly Chlorinated Paraffin Mineral Oil CLPMO as Emulsifiable Concentrate and Mayonnaise with Reference to KZ 95% EC and Alboleum 80% Mayonnaise

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Abstract: The physical properties of chlorinated paraffin mineral oil (CLPMO) were measured according to The American Chemical Society of Testing and Materials (ASTM). CLPMO was formulated as Emulsifiable Concentrate and Mayonnaise to be CLPMO 85% EC and CLPMO 80% mayonnaise. Their physic-chemical properties were measured with reference to KZ 95% EC and Alboleum 80% mayonnaise. The data appeared that, the physical properties of CLPMO 80% EC was more effective than KZ 95% EC. Also, the formulated mayonnaise was more effective than Alboleum 80% mayonnaise. The physical properties of spray solutions, foaming, emulsion stability, viscosity, surface tension, pH, conductivity, salinity and TDS for EC and mayonnaise were studied. The phytotoxicity of newly formulated mineral oils at 1.5% for both EC and mayonnaise were recorded. The pesticidal activity of CLPMO as EC and mayonnaise were evaluated against *Aphis fabea* with reference to KZ 95% EC and Alboleum 80% mayonnaise which appeared that, the efficiency of every oil were approximately equal its reference oil. The outdoor experiment against *Parlatoria ziziphi* appeared that, the efficiency of CLPMO 85% EC was more effective than KZ 95% EC where it was vise versa in case of mayonnaise mineral oil.

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1.Introduction:

CPs have been produced since the 1930s and have been used as additives in lubricants and cutting fluids for metal-working, plasticizers/flame retardants in plastics. CPs starting to decompose at temperatures above 300°C.

PCAs are chlorinated linear chain alkanes with the general formula $C_xH_{(2x+2)-y}Cl_y$. They are produced by the chlorination of different n-alkane fractions derived from petroleum distillation. The industrial formulations of PCAs are also known as chlorinated paraffins (CPs). They are divided into three groups: short-chain CPs comprising 10 to 13 carbon atoms (SCCPs or sPCAs), medium-chain CPs comprising 14 to 17 carbon atoms (MCCPs or mPCAs) and long-chain CPs with more than 18 or more carbon atoms (LCCPs). The many possible positions for the chlorine atoms and presence of chiral carbon atoms lead to a large number of potential positional isomers, and enantiomers and diastereoisomers. For example, there are theoretically 122 161 positional isomers for MCCPs, based on the assumption that no more than one chlorine atom binds to any carbon atom (Government of Canada, 2004).

Historically, world production of all CPs was estimated at 300,000 tonnes in 1985 (WHO, 1996) and this increased by up to 1% per year between 1989 and 1998 (Lahaniatis *et al.,* 2000).

CPs are viscous colourless or yellowish dense oils. Physico- chemical properties of CPs vary within large ranges, and are governed by two factors: the carbon chain length and the degree of chlorination (Drouillard *et al.,* 1998a,b; Government of Canada, 2004). CPs are lipophilic in nature and have the potential to biomagnify in food chains.

POPs are defined as organic compounds that are lipophilic, persistent, toxic to both fauna and humans, and that can undergo long-range transport. CPs have attracted increasing attention in the last decade as they represent a potential 'new' category of POPs (Alcock and Jones, 1999; Poremski *et al.*, 2001). Other reviews and scientific studies detail data for CPs on lipophilicity/bioaccumulation (Fisk *et al.*, 1998, 2000; Muir *et al.*, 2003, Government of Canada, 2004), persistence (Allpress and Gowland, 1999; Friesen *et al.*, 2004; Government of Canada, 2004), long-range transport (Tomy *et al.,* 1999; UNEP, 2002, 2003) and toxicity (WHO, 1996; Tomy *et al.*, 1998; Government of Canada, 2004).

In this study we aimed to formulate parafine mineral oil as emuslifable concentrate and mayonnaise and evaluate their physic-chemical properties with reference to KZ oil 95% EC and Alboleum 80% mayonnaise oil under different storage conditions, initial time, accelerated hot storage at 54ºC for 3 and 14 days and cold storage at 0ºC for 7 days, also determined the phytotoxicity of the new formulations and evaluate their pesticidal efficiency on black bean aphid (*Aphis fabae)* with comparison to reference oils. Finally, outdoor experiment of EC and mayonnaise CP mineral oil were carried out against black parlatoria scale (*Parlatoria ziziphi)*.

2.Material and Methods:

I. Materials:

I.1. Experimental mineral Oil:

Unformulated mineral oil and two formulated mineral oils were used throughout the course of the present study. The used oils were as follows:

*Chlorinated Paraffin Mineral Oil (CLPMO), processed by Petroleum Research Institute, Cairo, Egypt

* KZ Oil 95% EC, provided by Kafr El-Zayat Co. For Pesticides and Chemicals

*Alboleum Oil 80% Mayonnaise, provided by Kafr El-Zayat Co. for Pesticides and Chemicals

I.2. Emulsifiers:

Different ionic and non-ionic surfactants used in preparation of the mineral oil formulations were brought from the Egyptian local market.

I.3. Solvents:

Different non-polar solvents in ratio up to 5% were used for preparation of the used mineral oil crudes emulsifable concentrate.

1.4. Insect Strain:

1.4.1. Black bean aphid (*Aphis fabae)*

A laboratory strain of *Aphis fabae,* provided by Pest Rearing Department, Central Agricultural Pesticides Laboratory, Agricultural Research Center, Dokki, Egypt, was reared in laboratory on faba bean (*Vicia faba*) seedlings at 18°C and 90 % RH.

1.4.2.Black parlatoria scale (*Parlatoria ziziphi)*

Small highly infested trees of citrus reticulate at age of 2-3 years were selected according to the uniformity of trees in size, shape and high density of black parlatoria scale insect (*Parlatoria ziziphi*) on the leaves to evaluate the efficiency of the oils outdoor.

II. Methods:

II.1. Preparation of formulated mineral oil:

II.1.1 Emulsifable concentrates (EC) mineral oil:

The crude mineral oil CLPMO in different volumes were mixed with a proper solvent (non polar). Blind of (ionic and non ionic) surfactants were mixed together in different ratios, and then added to previously stirred mineral oil with solvent at 60 °C. The mixture was stirred for 1 h at the proper temperature. Finally, the formulation of the prepared mineral oil emulsifable concentrates (EC) were kept cool and then its properties were estimated. The chloroniated parafine mineral oil (CLPMO) was formulated as 85% emulsifiable concentrate (EC).

II.1.2 Mayonnaise mineral oil:

Formulation of the crude mineral oil CLPMO as mayonnaise formulation is little bit different than (EC) formulation.

In this type of formulation we used 80% of the mineral oils with a mixture of the solvents, emulsifiers and water. The mixture was homogenized for 1h and left at room temperature, then its physico-chemical properties was determined.

II.2.Physical Studies:

II.2.1. Physical properties of the crude oils:

The physical properties of crude mineral oil sample was carried out according to American Society of Testing and Materials (ASTM) to detect density & specific gravity (D 1298–99), distillation point (D 1160–99), unsulfonated residue (D 483–97), surface tension (D 1331-89:2001), refractive index (D 1218–02), viscosity (D 2196-2010), flash point (D 93–2008) and pour point (D 2386–97) (ASTM 93, 97, 99, 2001, 2005, 2008 and 2010).

II.2.2. Physical properties of the EC and mayonnaise mineral oils:

The emulsifiable concentrate of CLPMO 85% and KZ 95% were stored at 54 ± 1°C for 3&14 days (MT 46.1.3) and other at 0 °C for 7 days (MT 39.3) (Dobrat and Martijn, 2005). The physical properties of the EC formulated mineral oil samples (initially, and after cold and hot storage) were carried out according to CIPAC handbook for detecting free acidity/ or alkalinity (MT 191) (Dobrat and Martijn, 2005), density and specific gravity (MT 3.1) (Dobrat and Martijn, 1995). Also, surface tension (D 1331-89:2001), refractive index (D 1218–2002), viscosity (D 2196-10) and flash point (D 93–2008) were carried out according to American Society of Testing and Materials (ASTM 2001, 2002, 2005, 2008 and 2010). The physical properties of mayonnaise oils CLPMO and Alboleum 80% were carried out at initial time and after cold storage at 0ºC for 7 days in according to the previous test methods.

II.2.3. Physical properties of the spray solution of EC and mayonnaise mineral oils:

The physical properties of the spray solutions of both EC formulations (CLPMO 85% and KZ 95%) and mayonnaise (CLPMO and Alboleum 80%) mineral oils under dilution rate 5% for both soft and hard water were carried out according to CIPAC handbook to detect persistent foam (MT 47.2), emulsion stability (MT 36.3), pH (MT 75.3), and conductivity (MT 32) (Dobrat and Martijn, 1995).

Also, surface tension (D 1331-89:2001), refractive index (D 1218–2002), viscosity (D 2196-10) and flash point (D 93–2008) were carried out according to American Society of Testing and Materials (ASTM 2001, 2002, 2005 and 2008).

III. Biological studies:

III.1 Phytotoxicity studies

III.1.1. Determination of chlorophyll and carotenoids contents.

Chlorophyll and carotenoids contents of faba bean and maize leaves were colorimetrically determined after1, 2 and 3 weeks after sprayed with the tested mineral oils formulations each at 1.5% according to Hiscox and Israelstan (1979).

A sample of leaf tissue discs of 10 mg from treated bean and maize plants were placed in test tube containing 5 ml dimethyl sulfoxide (DMSO), and 10 mg of untreated leaf tissue were used as control. Chlorophyll and carotenoids were extracted into the fluid without grinding at 55°C overnight. The extracted liquid was filtered and transferred to a graduated tube and completed to total volume of 10 ml with DMSO. Absorbance was measured by Spectrophotometer (The Milton Roy Spectronic 21D UV-Visible Spectrophotometer) at 644 and 662 nm for chlorophyll (A and B) determination and 470 nm for carotenoids.

Total chlorophyll, chlorophyll a and chlorophyll b were calculated using Arnon equation (1949), while Canal *et al*., (1985) was used for carotenoids

Arnon equation:

Chl.a = 12.7 x O.D. 662 - 2.69 x O.D. 644 mg/l

Chl.b = 22.9 x O.D. 644 – 4.68 x O.D. 622 mg/l

Chl. a + b =20.2 x O.D. 644 + 8.02 x O.D. 622 mg/l

Reading at specific optical density (O.D.)

Villanueva equation:

$$\text{Carotenoids} = \frac{A\ 470 - 1.28(\text{Chl.a mg/l}) + 56.7(\text{Chl.b})}{256 \times 0.906}$$

Fresh weight and dry mater content per one plant was recorded.

Statistical analysis of all data was carried out using the “SAS” program.

III.2. Testing and evaluation the aphicidal efficiency

Three mineral oil CLPMO with 2 type of formulations EC and Mayonnaise at seven concentration in addition to (KZ Oil 95% EC) and (Alboleum Oil 80% Mayonnaise) as reference oil at concentration of 28500, 14250, 7125, 3562, 1781, 980 and 445 ppm for EC and 625, 1250, 2500, 5000, 10000, 20000 and 40000 ppm for Mayonnaise were tested against the adults of *Aphid fabae* according to the following method:

A stock solution of each tested oil was prepared and subsequent serial concentration were made by diluting with water (v/v) to give the necessary concentration to give about 20% - 80% mortality of each material. *Vicia fabae* plants were grown for at least 1 week in 7 cm diameter plastic pots before testing for treating with mineral oils

The corrected mortality percentage was used to calculate the LC_{50} values according to Finney (1971). The toxicity lines were drawn for evaluating LC_{10}, LC_{25}, LC_{50} and LC_{90} and the slope for every treatment was estimated. The toxicity indexes and relative potencies were calculated according to Sun (1950).

$$\text{Toxicity Index} = \frac{LC_{50}\text{ of the most effective pesticide}}{LC_{50}\text{ of the other pesticide}} \times 100 = ...\%$$

$$\text{Relative Potency} = \frac{LC_{50}\text{ of the least effective pesticide}}{LC_{50}\text{ of the other pesticide}} = ...\text{Fold}$$

III.3. Effectiveness of EC and mayonnaise formulations of CLPMO on black parlatoria scale (*Parlatoria ziziphi*)

Experiment was carried out outdoors on about 20 trees of citrus reticulate grown in Giza Governorate, Dokki, Egypt (2010) heavily infested with black parlatoria scale (*Parlatoria ziziphi)*.

Trees were about five years old 2-2.5 meter high and were not exposed to insecticide for more than three years prior to this experiment. Four treatments were applied on three replicates, one tree each (3 tree per treatment). Spraying was carried out on 20 July 2010 when the temperature and relative humidity were 30-33 and 59-92 R.H., respectively.

The treatments used were as follows.

Treatment 1: CLPMO oil 85% EC at the rate of 1.5%

Treatment 2: CLPMO oil 80% Mayonnaise at the rate of 2%

Treatment 3: KZ oil 95% EC at the rate of 1.5%

Treatment 4: Alboleum Oil 80% Mayonnaise at the rate of 2%

To study the scalicidal activity of the tested compounds, infested leaves sample of ten leaves were picked up from all directions, replicated three times i.e. 30 leave for each treatment. These samples were taken to the laboratory for counting alive adult female of the insect.

A pre-treatment count was made before spraying and 3 post treatment counts were taken 1, 2 and 3 weeks after application. The reduction percentages in the population density of *Parlatoria ziziphi* in relation to the pre-treatment count, was calculated according to Henderson and Tilton formula (1955):

Reduction percentage= 1- Ta Cb/Tb Ca X 100

Where= Tb and Ta are respectively pre-and post-treatment counts and Cb, Ca are respectively untreated checks before and after treatment.

Data of the percentage reduction were subjected to a simple analysis of variance by using computing and modal designs in an IBM compatible computer.

The three mineral oils at six concentrations and in addition of Mineral Kz oil, at concentrations of 9500, 4750, 2375, 1187.5, 593.75 and 148.44 ppm were tested against the adults of *Aphid fabae* according to the following method:

A stock solution of each tested compound was prepared and subsequent serial concentrations were made by diluting with water (v/v) to give the necessary concentration to give about 20% - 80% mortality of each material. The faba bean leaves were dipped in the insecticidal prepared solution for a period of 20 seconds then left for complete dryness on paper towel, then placed upside down on agar bed in small Petri dish (60 mm diameter). Ten healthy apterous adults were placed on the treated leaves surface of each Petri dish while leaves dipped in tap water were used as control. Five replicate batches of aphides (i.e. 50 insects) were used per each concentration; serial concentrations were used for each compound. Petri dishes containing aphids were carefully closed and kept in conditioned room for 18 °C and 90% RH for a period of 24 hours. The mortality percentages were corrected according to (Abbott, 1925).

3. Results and Discussion:

1. Physical properties of crude chlorinated paraffin mineral oil (CLPMO):

The physical properties of the chlorinated paraffin mineral oil were evaluated under different temperatures 20, 25, 30, 35, 40, 45 and 50ºC. The viscosity, surface tension, density, specific gravity, refractive index, pour point, unsulfonated residues and distillation point were measured according to their test methods. Data in table (1) appeared that the viscosity of CLPMO was varied during the changing in temperature where it was 154.77 cp. at 20ºC followed by 116.58 cp. at 25ºC and decreased to 29.99 cp. at 50ºC. Also, the surface tension of CLPMO was varied from 36 dyne/cm at 50ºC to 38 dyne/cm at 20ºC. The viscosity and surface tension were inversely proportional to temperature where they were decreased when temperature increased **(Seiz, 1953)**. Density and specific gravity of CLPMO were not changed considerably during the changing in temperature where they were 1.116 gm/cm^3 and 1.090 at 20ºC and 1.095 gm/cm^3 and 1.060 at 50ºC which reflect the stability of CLPMO. Also, the CLPMO was clear and transparent during the changing in temperature which appeared from the negligible change in the refractive index values were it was 1.4930 at 20ºC and 1.4819 at 50ºC. CLPMO was flowable initially -28ºC and unsulfonated residues were more than 98% which reflects the absence of naphthalinic compounds which causes phytotoxicity of mineral oils. Also, it was narrow range oil where its distillation point at 16% of its value was 300ºC followed by 50% at 340ºC and finally 90% at 355ºC.

Table (1): Physical properties of chlorinated paraffin crude mineral oil(CLPMO) at different temperatures

Physical properties	CLPMO						
	20°C	**25°C**	**30°C**	**35°C**	**40°C**	**45°C**	**50°C**
Viscosity (cp.)	154.77	116.58	91.98	67.39	49.59	38.39	29.99
Surface tension (dyne/cm)	38	37.8	37.2	36.8	36.5	36.3	36
Density (gm/cm^3)	1.116	1.112	1.109	1.105	1.102	1.099	1.095
Refractive index	1.4930	1.4913	1.4895	1.4878	1.4851	1.4838	1.4819
Specific gravity	1.590	1.085	1.080	1.075	1.070	1.065	1.060
Pour point				**-28 ºC**			
Distillation point	10% =300			50% =340		90% =355	
UR. value				**98%**			

2. Physical properties of emulsifiable concentrate CLPMO 85% and KZ 95% at different storage conditions:

Data in table (2) illustrated the physical properties of emulsifiable concentrates mineral oils CLPMO 85% and KZ 95% EC under different storage conditions; initial time, accelerated hot storage at 54ºC for 3 and 14 days and cold storage at 0ºC for 7 days. All the tested samples showed flash point over 75ºC, the density and specific gravity of KZ 95% EC were less than the density of water where it was 0.85 gm/cm^3 at initial time with a negligible change during storage conditions. While the density and specific gravity of CLPMO were more than that of water which make the oil flow through the water solution (spontaneous emulsion) which enhanced the miscibility and physical properties of CLPMO 85%EC.

The acidity of formulated mineral oil CLPMO at the initial time was 6.7 times more than the acidity of KZ oil. Also, the free acidities of CLPMO were 8.3, 8.2 and 8.2 times than the acidity of KZ oil for the following storage conditions; cold storage, accelerated hot storage at 54ºC for 3 and 14 days, respectively. As the acidity of mineral oil increases as its efficiency increases this acts to neutralize field dilution water which is alkaline due to the later aspects we expected that CLPMO 85% EC will be more effective than KZ 95% EC (Tawfik and El-Sisi, 1987).

The viscosity of formulated mineral oil CLPMO 85% EC at initial conditions was 7 times more than the viscosity of KZ 95% EC. Also, the viscosities of CLPMO 85% were 7.7, 7.7 and 6.1 times more than their values of KZ 95% EC oil for the following conditions; cold storage at 0ºC for 7 days, accelerated hot storage at 54ºC for 3 and 14 days, respectively. As the viscosity increase the adhering of oil drops on the leaf surface increase, also, as the viscosity of mineral oil increase, increasing the worming the leaf surface which protect the plant from cold climates during the long night of winter (Richardson, 1974)

The surface tension of CLPMO 85% EC was greater than that of KZ 95% EC at initial time by about 3.41 dyne/cm where it was 1.1 and 0.87 dyne/cm for accelerated hot storage at 54ºC for 3 and 14 days, respectively while the surface tension of KZ 95% EC was greater than the CLPMO 85% EC by 3.6 dyne/cm in case of cold storage at 0ºC for 7 days.

As the surface tension increase the spreading of oil droplet on the leaf surface increase where prevent the drafting of oil spray solution from the leaf surface and increase the time of exposure of the plant for the formulation and we expected that the CLPMO 85% mineral oil will be more effective than KZ 95% EC(Osipew 1966 and Furmidge, 1962).

Table (2) Physical Properties of Prepared Mineral Oil EC Formulations after different storage conditions

Pesticide used	Initial		Storage at 54°C For 3 days		Storage at 54°C For 14 days		Storage at 0°C For 7 days	
	CLPMO 85%	KZ 95% (Ref.)	CLPMO 85%	KZ 95% (Ref.)	CLPMO 85%	KZ 95% (Ref.)	CLPMO 85%	KZ 95% (Ref.)
Flash point	Over 75 ºC	Over 75 ºC	Over 75 ºC	Over 75 ºC	Over 75 ºC	Over 75 ºC	Over 75 ºC	Over 75 ºC
Free Acidity (% as H_2SO_4)	0.037	$5.5 X 10^{-3}$	$4.3 X 10^{-2}$	$5.2 X 10^{-3}$	$4.3 X 10^{-2}$	$5.2 X 10^{-3}$	0.036	$4.3 X 10^{-3}$
Density (gm/cm^3)	1.11	0.85	1.11	0.85	1.110	0.851	1.110	0.852
Specific Gravity	1.10	0.84	1.10	0.85	1.110	0.850	1.120	0.850
Viscosity (cp.)	167.56	24.02	167.00	23.67	146.25	23.84	188.72	24.29
Surface Tension (dyne/cm)	35	37	34.43	33	37.71	36.84	36	39.60
Refractive Index	1.4930	1.4655	1.4928	1.4659	1.4928	1.4660	1.4927	1.4660

3. Physical properties of mayonnaise CLPMO 80% and Alboleum 80% at different storage conditions:

Data in table (3) illustrated the physical properties of mayonnaise formulation CLPMO and Alboleum 80% under different storage conditions, at initial time and after cold storage at 0ºC for 7 days. They appeared that, CLPMO 80% was acidic in both case of treatment, initial and cold storage where Alboleum mayonnaise was alkaline during the treatment. The acidity of mayonnaise oil make the spray solution acidic which neutralize the alkalinity of water dilution field so we expect that, the CLPMO will be more effective than Alboleum mayonnaise mineral oil (Tawfik and El-Sisi, 1987).

Density of the two mayonnaise mineral oils didn't affected by cold storage condition where there were no change before and after storage for the both mayonnaise oils. The density of Alboleum 80% mayonnaise was 0.87 gm/cm^3 which make this formulation floating on the surface of dilution field water, while the density of CLPMO 80% mayonnaise was greater than density of water which help the oil to floating through the dilution water field and causes spontaneous emulsion which enhance the miscibility of CLPMO mayonnaise oil with the spray solution so we expected that the changing in density may enhance the effect of CLPMO than the Alboleum mayonnaise oil (Furmidge, 1962).

The condition of storage for both oils didn't affect on the homogeneity of the formulation where the viscosity of the mayonnaise oil were almostly constant. The viscosity of CLPMO oil was 32 times more than of the Alboleum oil at initial time where it was 31.6 times in case of cold storage. As the viscosity increase the adhering of oil drops on the leaf surface increase, also increases the viscosity make worming of the plant leaves which protect it from cold climates during the long night of winter (Moustafa *et al.* 1990).

Table (3): Physical properties of mayonnaise mineral oil formulation at initially and after storage at 0°c for 7 days.

Pesticide used	**Initial**		**After Storage at 0°C For 7 days**	
	CLPMO 80%	**Alboleum 80% (Ref.)**	**CLPMO 80%**	**Alboleum 80% (Ref.)**
Free Acidity/or alkalinity	0.0213 % as H_2SO_4	0.179 % as NaOH	0.0366 % as H_2SO_4	0.3189 % as NaOH
Density (gm/cm^3)	1.069	0.8737	1.070	0.8766
Viscosity (cp.)	73404	2283	73412	2321

4. Physical properties of spray solutions for emulsifiable concentrates mineral oils at different storage conditions:

Physical properties foaming, emulsion stability, viscosity, surface tension, pH, conductivity, salinity and total dissolved salts (TDS) of the spray solutions soft and hard water for CLPMO 85% and KZ 95% were carried out and illustrated in table (4). All the spray solutions in soft and hard water showed foaming value under permitted limit 10 ml and they were passed in the emulsion stability, where there were not any sedimentation or oily separation. The viscosity of spray solutions was varied in range 2.18 to 2.24 cp. Also, the surface tension of soft and hard water of CLPMO 85% and KZ 95% EC were changed from 31.1 dyne/cm of KZ 95% EC hard water after 14 days of storage at 54ºC to 39.6 dyne/cm of CLPMO 85% EC hard water at initial time. The pH values for spray solutions soft and hard water of CLPMO 85% EC were more acidic than KZ 95% EC for the corresponding spray solutions of soft and hard water. The conductivity for spray solutions soft and hard water of CLPMO 85% EC were greater than that of KZ 95% EC. Salinity values were changed in range 0.1 to 0.4 ‰, also, TDS were varied from 69 to 376 mg/l.

Table (4): Physical Properties of Spray solutions of Emulsifiable Concentrates CLPMO 85% and KZ 95% at different storage conditions.

Storage conditions	Initial				Storage at 54ºC for 3 days				Storage at 54ºC for 14 days				Storage at 0ºC for 7 days			
Formulations used	CLPMO 85% EC		KZ 95% EC (Ref.)		CLPMO 85% EC		KZ 95% EC (Ref.)		CLPMO 85% EC		KZ 95% EC (Ref.)		CLPMO 85% EC		KZ 95% EC (Ref.)	
	Soft water	Hard Water	Soft water	Hard Water	Soft water	Hard Water	Soft water	Hard Water	Soft water	Hard Water	Soft water	Hard Water	Soft water	Hard Water	Soft water	Hard Water
Foaming (mm)	10	10	6	2	-	-	4	2	1	-	5	5	5	2	3	2
Emulsion Stability	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass
Viscosity (cp.)	2.24	2.22	2.18	2.21	2.21	2.20	2.25	2.24	2.24	2.24	2.20	2.22	2.23	2.24	2.20	2.23
Surface Tension (dyne/cm)	38.6	39.6	39.2	35.2	36	36	33.5	33.5	31.1	33.7	32.33	31.1	35.2	36	32.3	34.4
pH	4.33	4.81	6.02	5.76	4.05	4.04	6.03	5.77	3.43	3.52	5.77	6.10	4.04	4.07	6.26	6.40
Conductivity (µs)	216	758	154	712	219	764	159.6	704	231	781	147.4	684	216	768	146	695
Salinity (‰)	0.1	0.4	0.1	0.3	0.1	0.4	0.1	0.3	0.1	0.4	0.1	0.3	0.1	0.4	0.1	0.3
T.D.S (mg/l)	103	363	73	340	104	368	75	338	110	376	70	335	102	368	69	334

5. Physical properties of spray solutions for mayonnaise mineral oils at initial time and cold storage at 0ºC for 7 days:

Data in table (5) illustrated the physical properties of spray solutions soft and hard water at initial time and after cold storage for 7 days at 0ºC presented that; no foams appeared for soft water and hard water for both CLPMO and Alboleum 80% mayonnaise at initial time and after cold storage. All these spray solutions passed the emulsion stability test where the viscosities were varied between 2.16 and 2.23 cp. Also, surface tensions were changed between 36.2 to 41.7 dyne/cm. The spray solutions of CLPMO were acidic and ranged between 3.79 to 4.89 where the spray solutions of Alboleum were alkaline and they were ranged between 8.67 to 8.68. The salinity were varied between 0.1 to 0.5‰ and TDS between 85 to 469 mg/l. Hard water spray solutions were greater in conductivity than soft water spray solutions which were varied between 179.6 to 555 µs.

Table (5): Physical Properties of Spray solutions of Prepared Mayonnaise Formulations at different storage conditions.

Storage conditions	Initial				Storage at 0°C for 7 days			
Formulations used	CLPMO 80% Mayonnaise		Alboleum 80% Mayonnaise (Ref.)		CLPMO 80% Mayonnaise		Alboleum 80% Mayonnaise (Ref.)	
	Soft water	Hard Water	Soft water	Hard Water	Soft water	Hard Water	Soft water	Hard Water
Foaming (mm)	-	2	-	-	-	2	-	-
Emulsion Stability	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass
Viscosity (cp.)	2.19	2.16	2.23	2.20	2.20	2.14	2.22	2.18
Surface Tension (dyne/cm)	39.6	41.7	36.4	36.8	39.6	41.7	36.2	36.8
pH	4.09	4.89	8.67	8.68	3.79	4.49	8.67	8.68
Conductivity (µs)	179.6	728	558	973	179.6	728	555	969
Salinity (‰)	0.1	0.4	0.3	0.5	0.1	0.4	0.3	0.5
T.D.S (mg/l)	85	350	265	469	88	362	269	462

6. Phytotoxicity:

6.1. Effect of emulsifiable concentrates mineral oils chlorophylls and cartenoids contents of treated beans and maize leaves:

Effect of tested EC formulated mineral oils on chlorophyll content in tested green bean leaves, comparing to KZ were shown in table (6). After 7days of treatment, CLPMO increased the chlorophyll a (Chl.a) to 531.0 mg/g fresh weight of bean leaves comparing to 316.0 and 330.4 mg/g in treatment of KZ and untreated control, respectively. After 14 days of treatment, both CLPMO and KZ reduced the chl. a to 304.1 and 309.2 mg/g, while the same treatment increased the chl. a to 339.0 and 341.1 mg/g, after 21 days, compared to 326.0 and 320.9 mg/g in untreated control respectively. The content of chlorophyll B (chl. b) in treated bean leaves were 549.9 mg/g with CLPMO treatment and 609.8 mg/g with KZ, compared to 548.6 mg/g in untreated control. Both CLPMO and KZ reduced the chl. b to 541.5 and 522.5 mg/g after 14 days to 562.2 mg/g in untreated control. After 21 days the CLPMO reduced the chl. b to 543.3mg/g, while KZ increased the Chl.b to 659.9 mg/g in untreated control. The total chlorophyll content of bean leaves was increased to 900.8 and 925.8 mg/g with CLPMO and KZ treatments after 7 days, compared to 879.1 mg/g in untreated control, while after 21 days it was increased to 882.3 and 1037.0 mg/g with same treatment, respectively, Compared to 878.5 mg/g in untreated control. After 14 days, the treatments reduced the total chlorophylls content to 818.6 and 831.7 mg/g, compared to 888.6 mg/g in untraded control. The carotene content in treated green bean leaves was 132.6 and 147.5 mg/g with CLPMO and KZ, compared to 132.4 mg/g in untreated control. The CLPMO and KZ reduced the carotene content to 124.3 and 126.2 mg/g compared to 135.8 mg /g in untreated control, after 14 days. Results revealed that while CLPMO reduced the carotene to 131.1, the KZ increased to 168.4 mg/g, compared to 134.7 mg/g in untraded control at 21days.

These resulted revealed that EC formulated mineral oils (CLPMO and KZ) highly reduced the content of chlorophyll a and b and total chlorophylls and carotene after 14 days of treatments.

The tested EC oils increased the above parameters after 7 days of treatments except chlorophyll a with KZ, on the other hand the KZ increased all tested parameters, the CLPMO increased the content of chlorophyll a and total chlorophylls, while reduced chlorophyll b and carotene.

Effect of EC formulation mineral oils on (CLPMO and KZ) on chlorophyll and carotene content in treated maize leaves were shown in table (6). The tested EC oils reduced the chl. a content, except KZ and CLPMO after 14 days and 21 days, respectively, where the amount of chl.a were 333.6 and 402.2 mg/g, compared to 332.4 and 347.4 mg/g in untreated control. The chl. b content in treated maize leaves was 548.8, 562 and 682.1 mg/g with CLPMO, while it was 583.9, 553.0 and 730.0 mg/g with KZ, compared to 540.7, 578.6 and 776.9 mg/g in untreated control after 7, 14 and 21 days respectively. It is obvious that the treatments reduced the chl. b content in maize leaves. Both CLPMO and KZ reduced the total chlorophylls content in maize leave to 887.7 and 916.8 mg/g, compared to 931.9 mg/g in untreated control respectively. The same notice was recorded after 14 and 21 days of treatment. Were the amount of total chlorophyll was 879.9 & 1024.4 mg/g and 886.5 & 1127.2 mg/g, compared to 911.0 and 1174.7 mg/g in untreated control, respectively. The CLPMO and KZ treatments reduced the carotene content in maize leaves to 132.4 & 141.1, 135.8 & 133.5 and 164.7 & 176.4 after 7, 14 and 21 days of treatments , compared to 142.7, 139.8 and 187.9 mg/g in untraded control, respectively.

These result indicated that the CLPMO and KZ reduced the tested parameters i.e. Chl.a, Chl.b, total chlorophylls and carotene contents in treated maize leaves except Chl.a after 14 and 21 days with KZ and CLPMO, respectively.

Table (6): Effect of tested emulsifiable concentrates formulated mineral oils on chlorophylls and cartenoids contents of treated bean and maize leaves.

	Period	7 Days						14 days						21 Days					
	Treatment	Bean			Maize			Bean			Maize			Bean			Maize		
	Type of chlorophyll	Chl. A	Chl. b	Total Chl.	Chl. a	Chl. b	Total Chl.	Chl. a	Chl. B	Total Chl	Chl. a	Chl. b	Total Chl.	Chl. a	Chl. b	Total Chl	Chl. a	Chl. b	Total Chl.
Chlorophylls content*	CLPMO 85%	350.95	549.84	900.79	338.92	548.80	887.72	304.07	514.54	818.61	317.93	562.01	879.94	338.96	543.31	882.27	402.24	682.11	1084.35
	KZ 95%	315.96	609.84	925.80	332.98	583.85	916.83	309.21	522.51	831.71	333.57	552.96	886.53	341.09	695.89	1036.97	397.23	729.97	1127.20
	Cont.	330.44	548.64	879.08	341.30	590.65	931.94	326.42	562.17	888.59	332.39	578.59	910.98	320.87	557.64	878.51	397.79	776.87	1174.7
Carotenoids content**	CLPMO 85%	132.64			132.45			124.26			135.78			131.12			164.73		
	KZ 95%	147.49			141.05			126.16			133.49			168.4			176.42		
	Cont.	132.45			142.67			135.77			139.75			134.71			187.90		

* Amount of chlorophyll mg/g fresh wt. ** Amount of cartenoids mg/g fresh wt

6.2. Effect of mayonnaise mineral oils chlorophylls and cartenoids contents of treated beans and maize leaves:

The mayonnaise formulated mineral oils affected on the content of green bean leaves from chlorophyll a, b and total chlorophylls and carotene (Table7)

The CLPMO mayonnaise reduced the Chl.a Content to 398.7 mg/g, while the Alboleum increased it to 417.6 mg/g after 7 days, compared to 399.3 mg/g in untreated control. The CLPMO mayonnaise increased the Chl.a to 385.4 and 420.3 mg/g, while the Alboleum increased it to 379.8 and 425.3 mg/g, compared to 367.9 and 408.0 mg/g after 14 days and 21 days of treatments. Where the Chl.b amount was 681.2 and 623.7 mg./g and 1108.3 and 630.1 mg/g , compared to 665.7 and 612.6 mg/g in untreated control, respectively. The treatments of CLPMO and Alboleum reduced the content of Chl.b to 714.9 and 724.0 mg/g after 21 days of treatment, compared to 752.3 mg/g. the above observations were recorded with total chlorophylls content. Where the CLPMO and Alboleum increased the total chlorophyll after 7 and/or 14 days to 1079.9 and 1009.1 mg/g, and 1108.3 and 1009.9 mg/g respectively, compared to 1065.0 and 980.5 in untreated control. The same treatments also reduced it to 1135.2 and 1144.3 mg/g, compared to 11160.3 mg/g in untreated control. The CLPMO mayonnaise and Alboleum treatments increased the carotene content to 164.8 and 1007 mg/g, compared to 170.7 mg .g in untreated control, while its increased to 150.5 and 152.1 mg/g compared to `47.9 mg/g in untreated control. The same treatments reduced the carotene content to 172.6 and 174.8 mg/g, compared to 181.9 mg/g in untreated control (Table7).

The results revealed that the tested mayonnaise formulated mineral oils (CLPMO and Alboleum) didn’t affect on the Chl.a in treated green bean leaves until 21 days of treatments. Results also revealed that the tested mayonnaise clearly reduced the amount of Chl. b, total chlorophylls and carotene in treated green bean leaves, compared to the untreated control.

Effect of tested mayonnaise formulation mineral oils (CLPMO and Alboleum) are shown in table (7). After 7 days of treatment, the CLPMO mayonnaise and Alboleum reduced the Chl.a in treated maize leaves to 476.2 and 482.5 mg/g, compared to 482.5 mg/g in untreated control, while the same treatments increased the Chl.a content to 469.4 and 456.6 mg/g compared to 454.3 mg/g in untreated control after 14 days, respectively.

Results showed that while the CLPMO mayonnaise increased the Chl.a content to 465.1 mg/g, the Alboleum reduced the Chl.a to 449.3 mg/g, compared to 461.4 mg/g in untreated control respectively. The Chl.b content in treated maize leaves was in the range of 682.5 to 781.9 mg/g with CLPMO mayonnaise, while it was in the range of 752.4 to 788.1 mg/g with Alboleum, compared to the range of 776.9 to 851.9 mg/g in untreated control It is clear that the tested mayonnaise oils reduced the Chl.b content, except Alboleum after 14 days of treatment. The same observations were recorded with total chlorophylls content in treated maize laves when treated with CLPMO mayonnaise and Alboleum. The amount of total chlorophylls content was 1258.1, 1152.1 and 1232.5 mg/g with CLPMO mayonnaise and 1254.7, 1244.7 and 1201.7 mg/g with Alboleum, compared to 1334.4, 1231.2 and 1255.4 mg/g in untraded control after 7, 14 and 21 days, respectively. It is obvious that all tested mayonnaise oils reduced the total chlorophylls except Alboleum after 14 days. The CLPMO and Alboleum reduced the carotene content to 188/7 and 188.8 mg/g after 7 days, compared to 205.8 mg/g in untreated control, respectively (table,2)

Results revealed that the CLPMO mayonnaise reduced the carotene content to 164.4 mg/g, the Alboleum increased it to 190.3 mg/g, compared to 187.6 mg/g in untreated control.

The two mayonnaise oils reduced the carotene to 187.5 and 181.7 mg/g, respectively compared the carotene amount of 191.7 mg/g in untreated control.

These result revealed that the CLPMO mayonnaise reduced all tested parameters except Chl.a content in maize leaves after 7, 14 and 21 days. The Alboleum increased Chl.a, Chl.b, total chlorophylls and carotene especially after 14 days of treatment. Results indicated that the clear differences were recorded between faba bean and maize leaves in there content from Chl.a, Chl.b, total chlorophylls and carotene content as results to spray with CLPMO EC, KZ, CLOMP mayonnaise and Alboleum.

Table (7): Effect of tested mayonnaise formulated mineral oils on chlorophylls and cartenoids contents of treated bean and maize leaves.

	Period	7 Days						14 days						21 Days					
	Treatment	Bean			Maize			Bean			Maize			Bean			Maize		
Chlorophylls content*	Type of chlorophyll	Chl. A	Chl. b	Total Chl.	Chl. a	Chl. B	Total Chl.	Chl. a	Chl. B	Total Chl	Chl. a	Chl. b	Total Chl.	Chl. a	Chl. b	Total Chl	Chl. a	Chl. b	Total Chl.
	CLPMO 80%	398.70	681.23	1079.93	476.17	781.94	1258.11	385.41	623.69	1009.09	469.63	682.48	1152.11	420.26	714.91	1135.17	465.12	767.39	1232.51
	Alboleum 80%	417.64	1108.32	1108.32	472.50	782.20	1254.70	379.79	630.14	1009.93	456.62	788.05	1244.67	425.26	724.02	1149.28	449.26	752.42	1201.68
	Cont.	399.31	665.67	1064.98	482.54	851.89	1334.43	367.88	612.62	980.50	454.29	776.86	1231.15	407.97	752.30	1160.27	461.40	793.96	1255.35
Carotenoids content**	CLPMO 80%	164.51			188.73			143.25			164.44			172.63			185.22		
	Alboleum 80%	166.72			188.81			146.98			190.33			174.84			181.65		
	Cont.	160.70			205.80			147.90			187.61			181.85			191.74		

* Amount of chlorophyll mg/g fresh wt. ** Amount of cartenoids mg/g fresh wt

7. Pesticidal effect of emulsifiable concentrates (CLPMO 85% and KZ 95%) and mayonnaise (CLPMO and Alboleum 80%) against *Aphis fabae:*

Data in table (8) presented the pesticidal effect of emulsifiable concentrates (CLPMO 85% and KZ 95%) and mayonnaise (CLPMO and Alboleum 80%) against *Aphis fabae* after 24 hrs of treatments. The emulsifiable concentrate mineral oil CLPMO and KZ showed a comparative effect on *Aphis fabae* where their LC_{50}'s were 1922.05 and 1029.95 ppm while LC_{90}'s were 107925 and 1423097 ppm. The toxicity index of CLPMO was 54% of KZ oil. CLPMO 85% EC mineral oil was more effective due to its slope 0.73, where it was 0.41 for KZ 95% EC.

The mayonnaise mineral oil CLPMO and Alboleum 80% appeared pesticidal effect on *Aphis fabae* where their LC_{50}'s were 4561.39 and 4403.92 ppm and their LC_{90}'s were 84556.55 and 86426.36 ppm. The toxicity index of CLPMO 80% mayonnaise was 97% of Alboleum 80% mayonnaise, where its slope was 1.01 and it was 0.99 for Alboleum 80% mayonnaise mineral oil.

Table (8): Effect of emulsifiable concentrates (CLPMO 85% and KZ 95%) and mayonnaise (CLPMO 80% and Alboleum 80%) mineral oils against Aphis fabae after 24 hrs of treatment.

Treatment	Concentrations in ppm (µg/ml)							LC_{50}	LC_{90}	Toxicity Index	Relative Potency	Slope
	A	B	C	D	E	F	G					
CLPMO 85% EC *	32.08	40.32	49.03	57.78	66.16	73.81	80.45	1922.05	107925.48	53.59	12.7	0.73
KZ 95% EC *	44.09	48.97	53.87	58.7	63.41	67.93	72.19	**1029.95**	1423097.2	**100**	23.7	0.41
CLPMO 80% mayonnaise**	19.15	28.50	39.60	51.61	63.48	74.18	82.97	4561.39	84556.55	96.55	6.13	1.01
Albolium 80% Mayonnaise**	20.03	29.39	40.37	52.18	63.80	73.26	82.89	**4403.92**	86426.36	**100**	6.34	0.99

A, B, C, D, E, F and G concentrations are:

* 445, 890, 1781, 3562, 7125, 14250 and 28500 ppm (µg/ml), respectively.

** 625, 1250, 2500, 5000, 10000, 20000 and 40000 ppm (µg/ml), respectively.

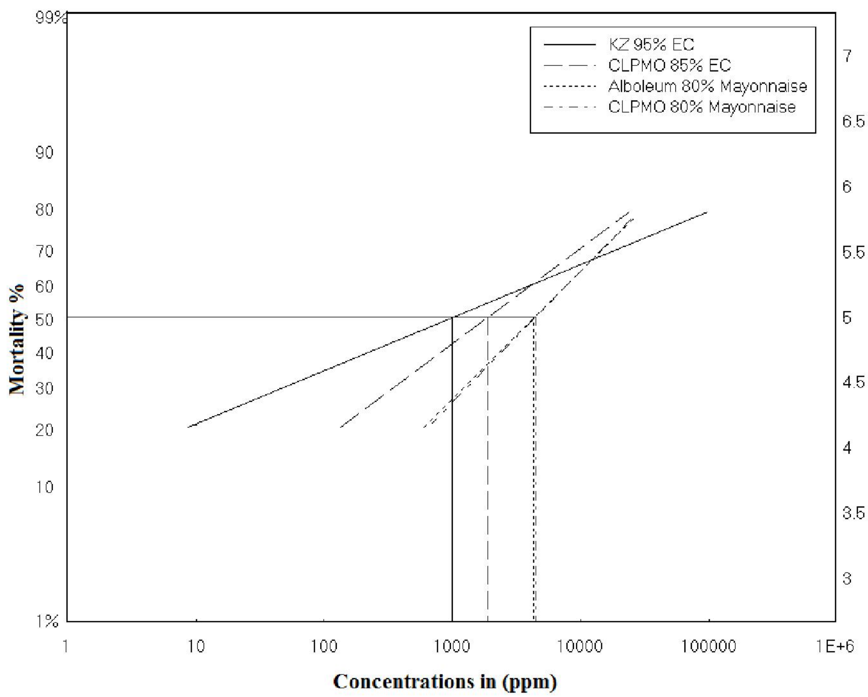

Fig.(1): Ldp lines of the emulsifiable concentrates (CLPMO 85% and KZ 95%) and mayonnaise (CLPMO 80% and Alboleum 80%) mineral oils against *Aphis fabae* after 24 hrs of treatment.

8. Outdoor application of emulsifiable concentrates (CLPMO 85% and KZ 95%) and mayonnaise (CLPMO 80% and Alboleum 80%) mineral oils against black parlatoria scale (*Parlatoria ziziphi*):

Data in table (9) represented the outdoor application of emulsifiable concentrates (CLPMO 85% and KZ 95%) and mayonnaise (CLPMO 80% and Alboleum 80%) mineral oils against *Parlatoria ziziphi*. The emulsifiable concentrate CLPMO 85%

showed mortality percent through 7, 14 and 21 days were 49.5, 66.1 and 79.5% against *Parlatoria ziziphi* when treated by 1.5% concentration, while the KZ 95% EC mineral oil showed 42.7, 60.8 and 76.6% morality percent. The latter data appeared that CLPMO 85% EC was more effective than KZ 95% EC mineral oils. The CLPMO 80% mayonnaise showed mortality percent against *Parlatoria ziziphi* 48.7, 70.1 and 74.9% while Alboleum 80% mayonnaise showed 48.7, 64.3 and 76.6% mortality % against *Parlatoria ziziphi*. This data appeared that Alboleum 80% mayonnaise mineral oil was more effective than CLPMO 80% mayonnaise during 21 days of treatment

Table (9): Outdoor application of emulsifiable concentrates (CLPMO 85% and KZ 95%) and mayonnaise (CLPMO 80% and Alboleum 80%) mineral oils against *Parlatoria ziziphi*

Treatment	7 Days				14 Days				21 Days			
	Rep. (1)	Rep. (2)	Rep. (3)	Mean	Rep. (1)	Rep. (2)	Rep. (3)	Mean	Rep. (1)	Rep. (2)	Rep. (3)	Mean
CLPMO 85% EC	52.4%	52.8%	43.4%	**49.5%**	68.7%	64%	65.7%	**66.1%**	76.8%	80.3%	81.2%	**79.5%**
KZ 95% EC	38.1%	46%	43.9%	**42.7%**	58.9%	61.9%	61.5%	**60.8%**	76.8%	74.3%	78.6%	**76.6%**
CLPMO 80% Mayonnaise	43.4%	49.4%	53.2%	**48.7%**	70.8%	70%	69.6%	**70.1%**	72.3%	77.3%	75.2%	**74.9%**
Alboleum 80% Mayonnaise	47.6%	51.5%	46.9%	**48.7%**	64.4%	65.7%	62.7%	**64.3%**	77.3%	77.3%	75.2%	**76.6%**

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