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Chemical composition and insecticidal activity of two eagle fern (*Pteridium aquilinum*) extracts on the poplar aphid *Chaitophorus populialbae* (*Hemiptera-Aphididae*)

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ABSTRACT

Within the framework of the development of the Algerian flora, the seriousness of the damage caused by aphids of which the species *Chaitophorus populialbae* Boyer de Fonscolombe, infested the white poplar and the search for alternative solutions more respectful for the environment relying on the role of biopesticides of plant origin, we chose a plant *Pteridium aquilinum* Linné, belonging to the family Dennstaedtiaceae which presents various biological activities, in particular insecticide. This study is intended to improve our knowledge of these chemical compounds. and evaluate the insecticide activity in vitro, in laboratory conditions, of this plant's methanolic and chloroformic extracts on viviparous apterous females of *Chaitophorus populialbae* Boyer de Fonscolombe. The best extraction yield of the plant was recorded with the polar solvent methanolic with a rate of 50.5% compared to chloroform, whose extraction rate was 43.5%. The chemical compounds identified by GC/MS are 29 from the ultrasound extraction. Among the 29 chemical compounds, 21 are terpenes, including 6 monoterpenes and 8 oxygenated monoterpenes, 7 fatty acids, and 1 amino acid. Toxicity tests of methanolic and chloroformic extracts, at a concentration of 50 µL/mL reveal corrected mortality of 100% after 24 h of exposure for all the viviparous apterous females of *Chaitophorus populialbae* Boyer de *Fonscolombe*.

Keywords: *Pteridium aquilinum* Linné, Poplar leaf aphids, GC/MS, chemical compounds, plant extract

INTRODUCTION

The natural range of the genus *Populus* extends throughout the northern hemisphere (United States, Canada, Europe, Russia, China, Japan, North Africa). In the southern hemisphere, poplars are cultivated in parts of Argentina, Chile, Australia, Uruguay, southern Africa, and New Zealand (Palancean, 2018). White poplars in the *Populus* and *Leuce* section are distributed between the 30th and 50th parallel in Euro-Asia and North Africa (Roiron et al., 2004).

In Algeria, the main species are composed of 15 coniferous and 46 deciduous trees, including 4 poplars (DGF, 2007). Four species of the genus *Populus* are present in Algeria (Zabielski, 1986). *Populus alba* Linné is found throughout northern Algeria. *Populus nigra* Linné is located along the wadis, along the roads, and in the massifs of Tlemcen. *Populus tremula* Linné is located in the forests of the high mountains of Babors, and finally, *Populus euphratica* Oliver, which is found in the west and south of the country, in the beds of Saharan wadis, Ghardaia, El Oued, and El Goléa (Harfouche et al., 2005).

They are species adapted to different soils, with high added value and productivity (Valadon and Villar,

1998). These main advantages and benefits of vegetative propagation are that it provides diverse sources (wood, pulp, paper, handicrafts, etc.), goods and services such as phytoremediation of degraded areas, combating desertification, water and soil conservation, carbon storage (Rennenberg and Peuke, 2005; Mikaili et al., 2012), and spaces for biodiversity (Tuskan et al., 2006).

Because of their great versatility, Algeria aims to extend its forest heritage to 1,245,000 hectares, of which 25,000 are poplars, in the long term, through its National Reforestation Plan (Ball et al., 2005). Meanwhile, poplars are very sensitive to fungi, virus diseases (Mekki, 2011), and to insect damage caused by Coleoptera, Lepidoptera and Hemiptera orders (Labiod et al., 2007), especially the genus *Chaitophorus* (Asiry, 2015). This genus causes significant damage due to the injected toxic saliva and honeydew excretion, providing a favorable conditions for the development of microorganisms and cryptogamic diseases responsible for yield and wood quality losses (Dedryver et al., 2010).

In the forestry sector, chemical control has had variable and ephemeral results, to say the least, mainly due to the very different spectra and modes of action of the major molecular families of products used (Brévault et al., 2008). Therefore, there is an urgent need to design and implement diverse methodologies adapted to the range of forest situations and existing pests. Plant chemodiversity represents new potential sources of biopesticides (Aouinty et al., 2006). In this context, we chose the plant *Pteridium aquilinum* Linné, belonging to the Dennstaedtiaceae family, which presents diverse biological activities, notably insecticide, to carry out laboratory tests on viviparous females of *Chaitophorus populialbae* Boyer de Fonscolombe on white poplar in order to provide a contribution to alternative pest control that respects the environment.

MATERIAL AND METHODS

Plant material

The leaves of *Pteridium aquilinum* Linné were collected in the spring of 2017 on plots of a farm of private status, located in the northeast of the country, of geographical coordinates 36°47'33'' N 6°47'50'' E. Botanical identification was made by Abdelkrim Hassen, Professor at the laboratory of the Botany Department of the Ecole Nationale Supérieure d'Agronomie (ENSA). The leaves were washed to eliminate impurities. They were then dried for two weeks at room temperature in the absence of light and humidity to preserve the molecular structure. To obtain fine powders, they were then subjected to milled with an electric grinder. The powders were then stored after sieving in amber glass bottles (Zaid et al., 2020).

Insects

The natural population of viviparous apterous females of the species *Chaitophorus populialbae* Boyer de Fonscolombe was collected on white poplar (*Populus alba* Linné), at the experimental station of the Institut National de la Recherche Forestière, in Baraki, located 14 km south-east of Algiers, Algeria. The species *Chaitophorus populialbae* Boyer de Fonscolombe was identified by Raphaëlle MOUTTET of the Entomology and Invasive Plants Unit of INRA, Montpellier (France). Aphids were deposited on the host plant (*Populus alba*) at the 4-leaf stage and covered with a muslin net to avoid any escape of the aphids. *Chaitophorus populialbae* Boyer de Fonscolombe was kept at 26±1 °C, 60±10% relative humidity and 16-8 h light/dark cycle.

Plant extraction process

20 g of fine powder of *Pteridium aquilinum* Linné leaves were put into 500 mL glass vials containing either 200 mL methanol (MeOH) or 200 mL chloroform ($CHCl_3$). Both mixtures were put in a Labnet shaker (model 211DS) at 150 rpm, with continuous stirring, for 48 h and at a temperature of 25 °C. After filtration using Watman paper, the filtrates obtained were concentrated under reduced pressure at a temperature of 40 °C using a rotary evaporator type (Büchi@ R-215). The obtained solutions were suspended in acetone to prepare stock solutions (Zaid et al., 2021). The obtained solutions were put in amber glass vials and then stored at 4 °C until use.

Extraction of chemical molecules

1g of fine powder of *Pteridium aquilinum* Linné leaves was mixed with 10 mL of methanol. The resulting solution was introduced into glass tubes. Then, the glass tubes were placed in a water bath sonicator at 60 °C and irradiated at 40KHz for six hours. After extraction, each glass tube had two phases, one liquid located on the top, and the other solid, located below. Using a 3 mL pipette, the liquid phase was removed from each glass tube. The collected liquid phase was introduced into new 15 mL glass tubes. These were centrifuged in a centrifuge at 3000 rpm for 10 min. The supernatant from each glass tube was collected and maintained at 4 °C.

GC/MS analysis

The chemical analysis of the compounds was performed by coupling (GC-MS) on an Agilent Technologies 6890N coupled to an Agilent Technology 5973 Mass Selective Detector equipped with an autosampler, equipped with a fused silica capillary column of (30 m x 0.250 mm x 0.25 µm), type HP-5MS. The temperature of the column was initially set at 60 °C for 1 min, then gradually increased to 150 °C until reaching 250 °C, at a rate of 5 °C / min. The carrier gas used, helium, had a purity of 99.9999%. The mass spectra were recorded at 70 eV. The control and analysis of the data were performed by the Agilent MSD Productivity ChemStation m/z software for MS. Molecule identification was performed on retention times in co-injection with available standards from Carinsa and SIGMA-Aldrich, Spain, and their spectra compared to those contained in the NIST 2011 computerized spectra library. The relative percentage of individual molecules was calculated based on the GC peak area corrected with an internal standard (Tridecane).

Bioassays of plant extracts

Bioassays on *Chaitophorus populialbae* Boyer de Fonscolombe were conducted in vitro at the laboratory of the Zoology Department of ENSA following the slightly modified protocol of Khoshraftar et al. (2020). Third-generation aphids from the laboratory rearing were selected for these trials. Treatments were performed separately in glass-bottomed Petri dishes with a diameter of 90 mm. A cotton soaked with one milliliter of distilled water was placed on each Petri to prevent the desiccation of *Populus* leaves during the treatments. 25 adult female apterous *Chaitophorus populialbae* Boyer de Fonscolombe were placed in each dish. Petri dishes were wrapped with a very thin mesh to avoid aphids escaping and to permit aeration of the dishes. After preliminary tests the concentrations 12,5µL/mL, 25µL/mL, 50µL/mL were selected. 500 µL of each concentration of each extract was uniformly distributed over all the Petri dishes. The solutions were prepared just before the treatments. For each plant extract, the treatment was repeated five times. All experiments were performed under the same laboratory conditions of 25 ± 1 °C, 60 ± 10% relative humidity, and 16: 8 (L : D) photoperiod. Mortalities were counted using a binocular magnifying glass at different exposure times to treatments, namely, 24, 48, 72, and 96 h. The control was treated with solvent only. The mortality rate was determined according to Abbott's (1925) formula. The CL_{50} and CL_{90} were calculated according to the method of Finney (1952).

Data analysis

Data was carried out using R@ v 3.6.1 Copyright 2019 The R Foundation for Statistical Computing for statistical analyses. Treatment differences were detected by ANOVA (analysis of variance). *P* values < 0.05 were considered significant. The Tukey test (HSD) was used for multiple (pairwise) comparisons. Lethal concentrations, which corresponds to the 50% and 90% mortality rates, were estimated using the regression line equation obtained by transforming the corrected percentage mortality into probits.

RESULTS

Yield of plant extracts

The highest extraction yield of *Pteridium aquilinum* Linné was recorded with the methanolic solvent with a rate of 50.5% compared to the chloroform solvent, whose extraction rate was 43.5%. Consequently, the yield of *Pteridium* Linné *aquilinum* extract was higher with the polar solvent methanol than with the less polar solvent chloroform (Table 1).

Table 1. Yield of *Pteridium aquilinum* extracts

Extract	Weight (g)	Yield (%)
MET	10.1	50.5 ± 1.11
CHL	8.7	43.5 ± 2.54

MET: Methanol, CHL: Chloroform. Results are expressed as mean ± standard error

Chemical composition of Pteridium aquilinum

Pteridium aquilinum Linné consists of 29 chemical compounds, including 21 terpenes, mainly monoterpenes (06) and oxygenated monoterpenes (08), 7 fatty acids (Myristic acid, Palmitic acid, Linolenic acid, Stearic acid, Oleic acid, Hexanoic acid, and Enanthic acid), and 1 amino acid (L-Glutamic acid) (Table 2).

Temporal effectiveness of Pteridium aquilinum treatments

Under the conditions of our experiment, the results show toxicity in viviparous apterous females of *Chaitophorus populialbae* Boyer de Fonscolombe (Figures 1 and 2). The toxicity is acute for methanolic and chloroform extracts, at a 50 µL/mL concentration, for viviparous apterous females of *Chaitophorus populialbae* Boyer de Fonscolombe. The effect of MET and CHL extracts, at the concentration of 50 µL/mL, reveals corrected mortality of 100% after 24 h of exposure for all the viviparous apterous females. At a 12.5 µL/mL concentration, the MET extract shows corrected mortality of 15.13% after 48 h of exposure for apterous viviparous females of *Chaitoophorus populialbae* Boyer de Fonscolombe and corrected mortality of more than 20% after 72 h of exposure. At the same concentration, the CHL extract shows corrected mortality of 10.7%, after 48h of exposure, for viviparous apterous females and 21.3%, after 72 h of exposure. The L-MET extract, at a concentration of 25 µL/mL, shows corrected mortality of 64%, after 48 h of exposure, for apterous viviparous females of *Chaitophorus populialbae* Boyer de Fonscolombe and more than 86%, after 72 h of exposure. At the same concentration, the CHL extract shows corrected mortality of 59.9%, after 48 h of exposure, for viviparous apterous females and 80.1% after 72 h of exposure.

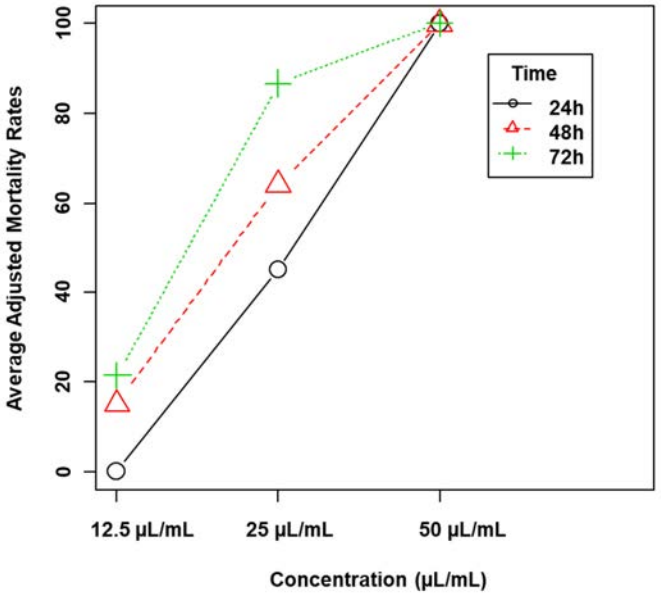

Figure 1. Temporal effect of the methanolic extract on viviparous apterous females of *Chaitophorus populialbae* Boyer de Fonscolombe

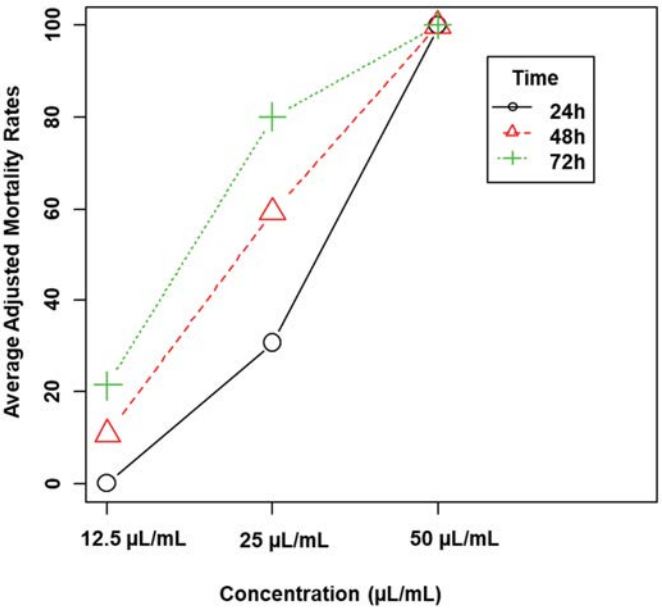

Figure 2. Temporal effect of chloroform extract on viviparous apterous females of *Chaitophorus populialbae* Boyer de Fonscolombe

LC_{50} and LC_{90}

The LC_{50} and LC_{90} lethal concentration values (Table 3 and Figures 3, 4), the MET and CHL extracts have an LC_{50} of 26.91 µL/mL, and 28.18 µL/mL, respectively. The LC_{90} of MET and CHL extracts are 40.73 µL/mL) and 42.65 µL/mL, respectively, after 24 h of exposure.

Table 2. Chemical composition of *Pteridium aquilinum*

	Compounds	Ultrasound %	Rt (min)	RI $^{K (exp)}$	RI $^{t (Kovats)}$	Chemical formula
1	beta-Pinene	0.49	6.713	934	964	$C_{10} H_{16}$
2	1-Octyn-3-ol. 4-ethyl-	0.34	6.967	951	-	$C_{10} H_{18} O$
3	D-Limonene	0.64	8.117	1027	1030	$C_{10} H_{16}$
4	o-Cymene	0.50	8.826	1078	1039	$C_{10} H_{14}$
5	Benzene. 1.2.3.4-tetramethyl-	1.89	9.403	1122	1151	$C_{10} H_{14}$
6	Citronellol	7.97	13.510	10716	1225	$C_{10} H_{20} O$
7	8-Quinolinol. 2-methyl-	4.67	13.714	1488	-	$C_{10} H_{9} NO$
8	Nerol oxide	3.78	13.738	1409	-	$C_{10} H_{16} O$
9	(-)-trans-Pinan	2.35	16.995	1832	972	$C_{10} H_{18}$
10	Phytol	14.84	19.247	2105	2106	$C_{20} H_{40} O$
11	gamma-Sitosterol	6.25	21.212	2374	3203	$C_{29} H_{50} O$
12	Butyrolactone	0.23	6.38	910	915	$C_{4} H_{6} O_{2}$
13	Catechol	0.15	6.491	918		$C_{6} H_{6} O_{2}$
14	3-Octanol	1.45	7.598	992	994	$C_{8} H_{18} O$
15	Benzyl alcohol	0.47	8.169	1033	-	$C_{7} H_{8} O$
16	p-Cresol	0.25	8.715	1070	1070	$C_{7} H_{8} O$
17	Phytol. acetate	5.39	17.002	1833	-	$C_{22} H_{42} O_{2}$
18	Glyceryl acetate	0.31	7.063	958	1095	$C_{5} H_{10} O_{4}$
19	Phenol. 2.4-bis(1.1-dimethylethyl)-	2.01	13.92	1508	-	$C_{14} H_{22} O$
20	Hexanoic acid	0.80	7.3786	978	-	$C_{6} H_{12} O_{2}$
21	Enanthic acid	1.69	8.694	1071	1062	$C_{7} H_{14} O_{2}$
22	Myristic acid	3.26	15.442	1662	1766	$C_{14} H_{28} O_{2}$
23	Oleic Acid	1.48	18.014	1951	-	$C_{18} H_{34} O_{2}$
24	linolenic acid	15.40	19.486	2136	2099	$C_{18} H_{30} O_{2}$
25	palmitic acid	12.00	20.396	2259	2498	$C_{16} H_{32} O_{2}$
26	stearic acid	9.92	21.763	2454	-	$C_{18} H_{36} O_{2}$
27	L-Glutamic acid	0.91	6.171	895	-	$C_{5} H_{9} NO_{4}$
28	p-Xylene	0.11	5.702	865	865	$C_{8} H_{10}$
29	Coumaran	0.44	6.751	937	1219	$C_{8} H_{8} O$
Total Terpenes and Terpenoids		**54.53**				
Total Fatty acids		**44.55**				
Total Amino Acid		**0.91**				

Table 3. Calculation of the LC_{50} and LC_{90} of *Pteridium aquilinum* Linné extracts after 24 h of exposure on the mortality rate of adult viviparous females of *Chaitophorus populialbae* Boyer de Fonscolombe

Extracts	LC_{50} (mL/mL)	LC_{90} (mL/mL)	Regression equation	R^2
MET	26.91[a]	40.73[a]	y = 13.437x - 14.344	0.9722
CHL	28.18[a]	42.65[a]	y = 13.437x - 14.594	0.9962

MET: Methanol, CHL: Chloroform, LC_{50} is a lethal concentration that kills 50% of the exposed population; LC_{90} is a lethal concentration that kills 90% of the exposed population. Means followed by the same letter in the same column are not significantly different

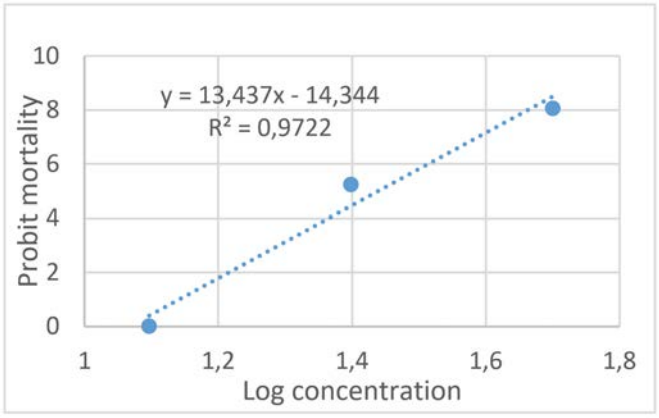

Figure 3. Regression line of the log dose-probit analysis relationship of the methanolic extract

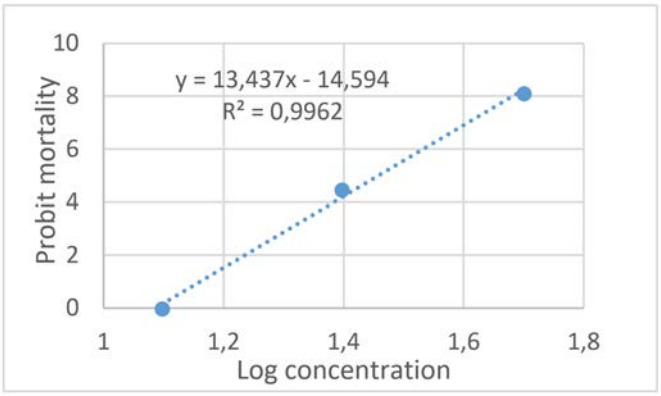

Figure 4. Regression line of the log dose-probit analysis relationship of the chloroform extract

Comparative effects of Pteridium aquilinum Linné extract concentrations

Table 4 shows a significant difference between the values of the corrected mean mortality of apterous viviparous females of *Chaitophorus populialbae* Boyer de Fonscolombe for the different applied concentrations of MET and CHL extracts. Tukey's post hoc test (HSD) resulted in three distinct concentration groups. Group (A) corresponds to the 12.5 µL/mL concentration with the low corrected mortality rates, 12.17% for MET extract and 10.66% for CHL extract (Figure 5). Group (B) refers to the 25 µL/mL concentration, and group (C) matches the 50 µL/mL concentration with the highest corrected mortality rate averages.

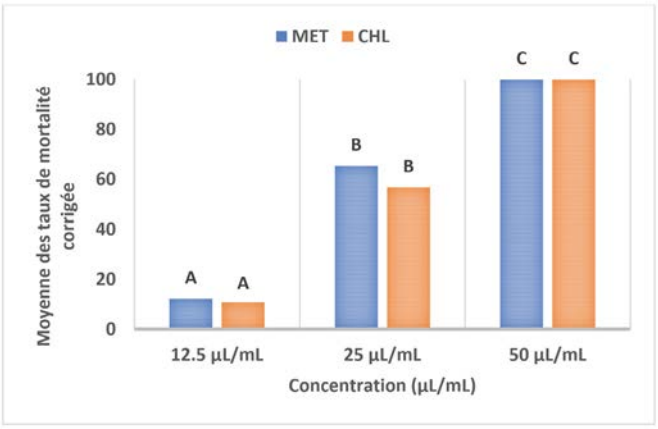

Figure 5. Comparative effects of methanol and chloroform extract concentrations on the mean corrected mortality rates of *Chaitophorus populialbae*

Methanol, CHL: Chloroform, Means followed by the same letter are not significantly different

Table 4. Analysis of variance of the corrected mortality of *Chaitophorus poplialbae*

Extracts	Factors	Sum of squares	DDL	Means of squares	F-ratio	*P*
MET	Concentration	11.37	2	5.868	31.1	*P*<0.001
	Residus	1.114	6	186		
CHL	Concentration	11.974	2	5.987	24.66	*P*<0.001
	Residus	1.457	6	243		

MET: Methanol, CHL: Chloroform

DISCUSSION

The yield obtained from the plant extract by the polar solvent methanol is higher than with the less polar solvent chloroform. By penetrating deeply into the plant substrate, the polar solvent destabilizes the cell walls upon contact with a greater amount of solute, thus promoting extraction (Penchev et al., 2010). Therefore, our results are supported by those obtained by Brown et al. (2013) and Nakamura et al. (2017), with extracts from the leaves of *Gleditsia triacanthos* Linné and *Sasa quelpaertensis* Nakai, which indicate that the yields are higher with methanol than with chloroform. The chemical composition of *Pteridium aquilinum* Linné is based on the fatty acid/phytol chemotype. The results of previous studies on *Pteridium aquilinum* Linné under different operating conditions and analytical methods indicate the presence of chemical groups such as sterols, polyterpenes, polyphenols, flavonoids, catechic tannins, and quinone substances (Nwiloh et al., 2014; Adou et al., 2016).

Only Halarewicz and Szumny (2010) in Poland showed that the main constituent of *Pteridium aquilinum* Linné extract is benzaldehyde, followed by chemical compounds in very small amounts such as limonene, linalool, terpineol, and citral. The scientific literature reveals that the quality and quantity of chemical compounds of the plant depend on many factors such as geographical and climatic conditions, nature and components of the soil, genetic heritage of the plant, its organ, vegetative cycle, harvesting schedule and techniques, and extraction method and its course (Djeddi, 2012). Plants are, therefore, regularly influenced by environmental variations, and in response to these sub- and super-optimal conditions, plants exhibit variations in their growth and development (Parent et al., 2008; Antoun, 2013). As for the biocidal potential, the results show that the 50µL/mL concentration is much more effective than the other concentrations applied to the corrected mortalities of adult viviparous females of *Chaitophorus populialbae* Boyer de Fonscolombe.

Huang et al. (2010) indicated that methanolic extracts of 22 ferns have clear bioactivity against *Aedes albopictus* Skuse and *Musca domestica* Linné. Gerhardt et al. (2012) revealed that aqueous leaf extract of *Pteridium aquilinum* Linné results in over 70% contact mortality on *Myzus persicae* Sulzer aphid. Selvaraj et al. (2005) stated that chloroform extract and ethanolic extract of the fern showed differences in their toxic and growth disrupting responses (mortality and susceptibility) on *Helicoverpa armigera* Hübner.

In addition, our results are consistent with those obtained by Rahuman et al. (2009) and those of Kamaraj et al. (2009), who reported that the insecticidal effect is related to dose, exposure time, and extract type. These positive results reveal that some phytochemical constituents are effective against various organisms, including worms and insects (Chaiyasit et al., 2006; Liu et al., 2006). They act directly on the cuticle of soft-bodied insects and mites (Bostanian et al., 2005). Their efficacy varies depending on the phytochemical profile of the plant extracts, the entomological target, and the exposure time (Regnault-Roger et al., 2012).

CONCLUSION

The purpose of this research was to extend the knowledge of the chemical constituents of *Pteridium aquilinum* Linné, and to evaluate the activity of the methanolic and chloroform extracts of the plant *Pteridium aquilinum* Linné, on the natural population, resulting from the breeding in the laboratory, of viviparous apterous females of *Chaitophorus populialbae* Boyer de Fonscolombe.

The results of the extractions led to a yield of 50.5% with the polar solvent methanolic than with the less polar solvent chloroform. The research led to the identification by GC/MS of 54.53% of terpenes and terpenoids, including 48.27% of monoterpenes and oxygenated monoterpenes, mainly phytol with 14.84%, 44.55% of fatty acids, including linolenic acid (15.40%), palmitic acid (12.00%) and stearic acid (9.92%), and less than one percent of the amino acid (0.91%). For toxicity tests, under laboratory conditions, methanolic (MET) and chloroformic

(CHL) extracts, at the concentration of 50 µL/mL, were shown to be toxic to the natural population of viviparous apterous females of *Chaitophorus populialbae* Boyer de Fonscolombe, with a corrected mortality rate of 100%, after 24 h of exposure.

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