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Assessing the insecticidal effect of *Citrus aurantium* and *Nerium oleander* extracts and basalt "Farina di Basalto®" as biological alternatives to control *Aphis punicae* and *Planococcus citri* in an organic pomegranate orchard

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Abstract: This study aims to evaluate the efficiency of basalt with two botanical aqueous extracts of *Citrus aurantium* and *Nerium oleander* (50g/L) against *Aphis punicae* and *Planococcus citri* in an organic pomegranate orchard. Basalt was applied as a foliar spray with two doses (1.5% and 3%), in the ground as a fertiliser (1.5%), and as a combined application (1.5%). Phenolic compounds of both plants were identified by HPLC-PDA/ESI-MS analysis. Seven and six flavonoids were detected in *C. aurantium* and *N. oleander* extracts, respectively. Hesperidin was more abundant in *C. aurantium* extracts; however, *N. oleander* extracts contained more quercetin rutinoside. *A. punicae* populations were significantly higher in control trees compared to the treated ones. *A. punicae* mortality rates reached high values above 90% for basalt and plants extracts and efficacy rates exceeded 80%. *P. citri* mortality rates reached 88% and 77%, and efficacy rates exceeded 75% and 66% for basalt and plant extracts, respectively. These findings may help to plan Integrated pest management strategies in organic orchards to avoid toxic chemical pesticides.

Keywords: basalt; biological control; extracts; efficacy rate; HPLC; mortality rate

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The aphid *Aphis punicae* Passerini (1863) (Hemiptera: Aphididae) is among the most essential aphids attacking pomegranates. *A. punicae*'s adults and nymphs attack leaves, inflorescences, and fruits (Ben Halima & Ben Hamouda 2005; Fakhour 2012). Ben Halima and Ben Hamouda (2004, 2005) affirmed the high reproduction capacity of this pest from April to June. It reduces plants' vigour and induces the development of sooty mould on leaves (Rouhani et al. 2013). Concerning control measures for this pest in Tunisia, chemical products are the most important method to control aphids in pomegranate orchards (Mdellel et al. 2015). Systemic insecticides have the advantage of killing. However, using these molecules leads to their persistence in parts of the plant and, thus, a higher risk of accumulation in the food chain (Bhatia et al. 2011). Moreover, insecticide resistance has been demonstrated in several aphid species (Barbagallo et al. 2007; Foster et al. 2007). The development of resistance is caused by the extensive use of insecticides against aphids (Nauen & Elbert 2003; Cao et al. 2003). For these reasons, alternative control methods should be developed. Many researchers have been studying the effect of plant extracts on aphids (Endersby & Morgan 1991; Moawad & Al-Barty 2011). Mealybug *Planococcus citri* Risso (1813) (Hemiptera; Pseudococcidae) is a major pest of *Citrus* fruits and many other orchards and ornamental crops in subtropical and tropical regions of the world (Blumberg et al. 1975). Bartual et al. (2012) affirmed that *P. citri* is a severe pest of pomegranate in Spain. *P. citri* can affect the marketing quality of pomegranate (Kahramanoglu & Usanmaz 2013). Only larvae and adult females cause damage. Males are incapable of feeding, fulfilling only the role of reproduction. Aerial parts of plants are the most affected; however, the roots are also attacked on some plants, such as coffee. *P. citri* preferentially attacks the fruit's peduncle and its point of insertion. Severe damage causes fruit growth disorders or abortion, as seen on *Citrus* fruits (orange trees) and cucurbits such as melon and cucumber. Moreover, mealybugs can develop on harvested fruits (Babin 2018). *P. citri* causes yellowing of leaves and growth disability, resulting in deformation, growth retardation, and loss in photosynthesis and plant productivity (Gill et al. 2016). On the Cocoa tree, *P. citri* attacks flower stalks, buds, and young pods (Entwistle 1972). The mealybug produces honeydew on which sooty mould grows, mainly the fungi of the genus *Cladosporium*. Sooty mould reduces the plant's photosynthetic activity when it covers the leaves, affecting growth and productivity. Moreover, the sooty mould renders fruits, vegetables, and ornamentals unsuitable for sale (Babin 2018). In Tunisia, the biological control of *P. citri* is mainly based on natural enemies such as the predator *Cryptolaemus montrouzieri* (Coleoptera: Coccinellidae) (Rahmouni & Chermiti 2012, 2013) and parasitoids like *Leptomastix dactylopii* (Hymenoptera: Encyrtidae) (Abbes et al. 2018). The use of pesticides in *Citrus* orchards causes parasitoid toxicity (Abbes et al. 2018). Besides, they are affecting the coccinellid predator *C. montrouzieri* (Rahmouni & Chermiti 2012). Panis (1977) added that *P. citri* populations in Morocco became resistant to several chemicals such as parathion, dimethoate, and diethion. Franco et al. (2004) affirmed that chemical insecticides are the primary method used to control *Citrus* mealybug due to the weak adaptation of natural enemies to the climatic conditions of the Mediterranean. In addition, they suggested that mass trapping and mating disruption should be considered alternatives in IPM strategies.

This study evaluates and compares three biological alternatives: aqueous plant extracts of two plants, bitter orange and common oleander, and a mineral product, basalt powder named "Farina di Basalto®" against *A. punicae* and *P. citri* on pomegranate orchards.

MATERIAL AND METHODS

Experimental site and study period. The present study was conducted in an organically certified pomegranate orchard situated in the region of Slouguia (Governorate of Beja) in Tunisia (36°36'12.7"N 9°29'51.3"E) (Figure 1). The orchard area is about 4.1 ha. No chemical pesticide treatments or fertilisers were applied at the experimental site. The last organically labelled fertiliser used in the orchard was the seaweed extract six months before the trial. The study was carried out from July 15 to August 21, 2020. The distance between orchard trees is about 4 m.

Treatments and experimental design. Fruits of bitter orange *Citrus aurantium* L. (1753) (Sapindales; Rutaceae) were collected from the Higher

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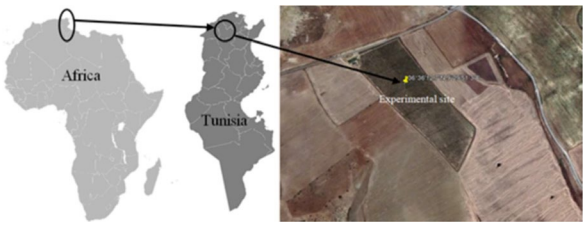

Figure 1. Geographical localisation of the experimental site

School of Agriculture of Mograne (ESAM) (Zaghouan, Tunisia). They were taken to the Laboratory of Entomology of ESAM and peeled. Leaves of Nerium *Nerium oleander* L. (1753) (Gentianales; Apocynacae) were collected from the Higher School of Engineering of Medjez El Beb (ESIM) (Béjà, Tunisia). Fruit peels of *C. aurantium* and leaves of *N. oleander* were dried at room temperature and then ground until they obtained a fine powder. Both extracts were tested at 50 g per litre of water. The powder of each extract was mixed with water and stood for 48 hours for maceration. Then, the mixture was filtered with muslin to remove large particles.

Basalt powder, or "Farina di Basalto®", was obtained from Basalti Orvieto from Italy. It is a micronised basalt powder with particles less than 30 µm obtained by mechanical grinding basalt, an effusive volcanic rock. Basalt powder is not harmful and does not damage the environment. It contains natural elements such as Silicon, Alumina, Potassium, and Calcium (Elimem et al. 2020; Rouz et al. 2020; Elimem et al. 2021) (Table 1). Basalt powder was mixed with water and applied as a foliar spray (FS) and a ground fertiliser (FG). Two doses of 1.5% and 3% were tested for both application modes.

Experimental design and treatment application. A Completely Randomised Block Design (CRBD) was adopted. Seven treatments were applied: 50 g/L of bitter orange extract (BE), 50 g/L of *Nerium* extract (NE), a 1.5% basalt aqueous foliar spray (FS 1.5%) and ground fertiliser (BC 1.5%), a 3% basalt aqueous foliar spray (FS 3%) and ground fertiliser (BC 3%), combined application of a 1.5% basalt aqueous foliar spray and ground fertiliser (BC 1.5%), and an untreated check (C) (Table 2). Treatments were pulverised twice on July 23, 2020 (T1) and August 7, 2020 (T2) using an agricultural atomiser. Each treatment was replicated three times. For each, three trees were randomly chosen, marked, and served for monitoring different parameters. Each tree is considered a plot with an area of about 10 m². The pomegranate tree's height in the different plots was 2 to 2.5 m.

Table 1. Chemical characteristics of basalt powder (Anonymous 2019)

Component	Percentage
SiO_2	49.0%
Al_2O_3	20.5%
K_2O	8.0%
Fe_2O_3	7.5%
CaO	7.2%
MgO	2.8%
Na_2O	2.5%

Table 2. Applied treatments with doses and abbreviations

Treatment	Abbreviation	Dose
Control	–	–
Bitter orange extract	BE	50 g/L
Nerium extract	NE	50 g/L
Basalt as a foliar spray at 1.5%	BFS 1.5%	1.5%
Basalt as a foliar spray at 3%	BFS 3%	3.0%
Basalt as a fertiliser at 1.5%	BFR 1.5%	1.5%
Combined basalt application as a foliar spray and as a fertiliser	BC 1.5%	1.5%

Sampling – four twigs 30 cm long were sampled from each side of the randomly chosen trees. Twigs were placed in plastic bags marked with the treatment name and taken to the laboratory. Sampling took one week before applying the different treatments (July 15, 2020) and then every two, three, seven, and fourteen days after each treatment.

Efficacy evaluation: Mortality rates of both pest species were corrected using the Abbott formula (Abbott 1925):

$$M_r = \left(\frac{\frac{M_t}{M_c}}{100 - M_c} \right) \times 100 \quad (1)$$

where: M_r – the mortality rate, M_t – the number of dead individuals in treated plots, M_c – the number of dead individuals in untreated plots (control).

Treatment efficacies were also calculated according to the Abbott formula (Abbott 1925):

$$E_r = \left(\frac{T_0 - T_t}{T_0} \right) \times 100 \quad (2)$$

where: E_r – the percentage of efficacy, T_0 – the number of living individuals on untreated plots, T_t – the number of living individuals on treated plots.

Identification of phenolic compounds by HPLC–PDA/ESI–MS analysis. The extracts were prepared following the general procedure de-

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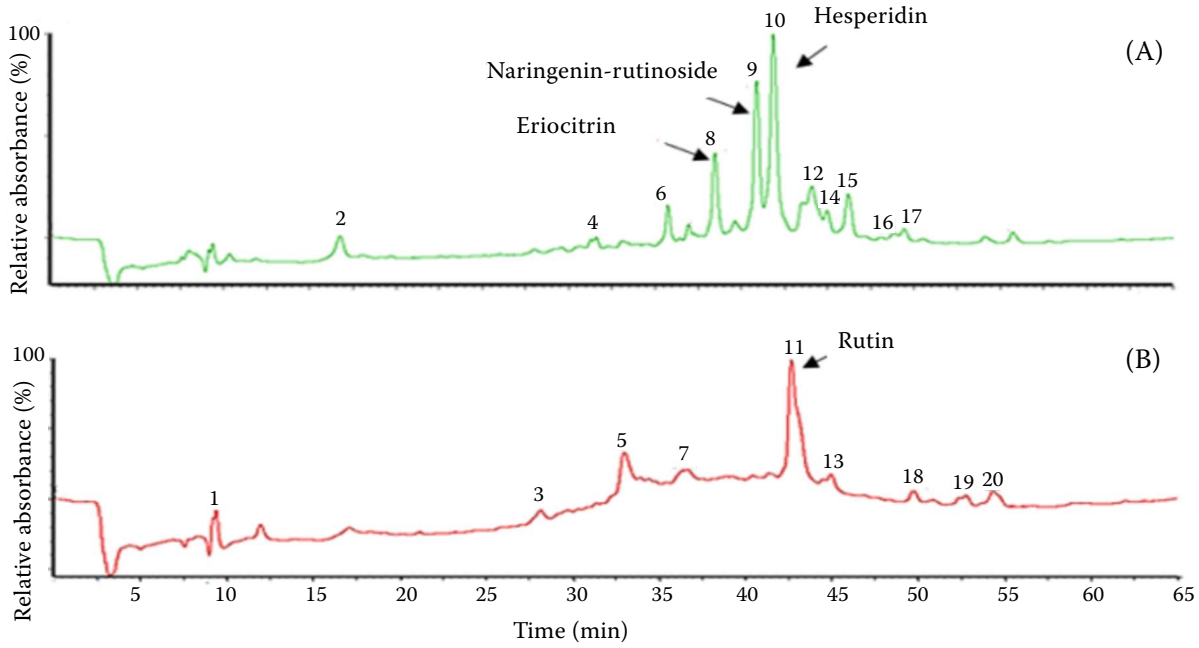

Figure 2. LC-PDA-TIC profile of phenolic components from *Citrus aurantium* (A) and *Nerium oleander* (B) aqueous extracts (assignment of peaks are given in Table 3)

scribed by Rhimi et al. (2019). In detail, at room temperature, 20 g of dried leaves for Nerium and 20 g of dried peels for bitter orange were macerated in 100 mL of water for 48 h. After filtration, samples were lyophilised, transferred to vials, and stored in the dark at four °C until further analysis. Phenolic compounds were identified using high-performance liquid chromatography coupled with a photodiode array and mass spectrometry detection (HPLC–PDA–ESI/MS) (Boulila et al. 2015).

LC–ESI–MS analysis was conducted in negative electrospray ionisation mode on an Agilent 1100 series HPLC system (Agilent Technologies, Palo Alto, CA, USA) equipped with a photodiode array detector (PDA) and a triple quadrupole mass spectrometer type Micromass Autospec Ultima Pt (Kelso, UK). Analysis was run on a reversed-phase Uptisphere C18 (Interchim) (2 mm × 100 mm, 5 µm particle size) at 40 °C.

The mobile phase for separating extracts' compounds was composed of water, 0.1% formic acid in water (A), and 0.1% formic acid in methanol (B). The following multi-step linear solvent gradient was employed: 0–5 min, 2% B; 5–60 min, 98% B, 60–65 min, 2% B.

Statistical analysis. Analysis of Variance (ANOVA) and Least Significant Difference (LSD) test for means comparison at $P \leq 0.05$ were performed on generated data from measured parameters using the statistical software program Statistical Package for the Social Sciences (version 23) (SPSS 23).

RESULTS

HPLC–PDA/ESI–MS analysis. The HPLC–PDA/ESI–MS allowed identifying seven flavonoid derivatives in *C. aurantium* extract (Table 3, Figure 2), including five flavanones (eriodictyol, hesperidin, naringenin-rutinoside, eriocitrin, brutieridin), one flavonol (quercetin), and one limonoid (limonin) (Figure 3). Hesperidin (peak 10, UV_{max} = 284 and 329 nm, $[M–H]^-$ ion at *m/z* 609) was the main compound identified in the *Citrus* extract. Besides, the aqueous extract was also rich in naringenin rutinoside (peak 9, $[M–H]^-$ at *m/z* 579 with a base peak at *m/z* 271 corresponding to the loss of a hexose unit) and eriocitrin [peak 8, $[M–H]^-$ at *m/z* 595 –› $[M–162–146–H]^-$ at *m/z* 287 corresponding to the aglycon eriodyctiol by the neutral losses of a hexose (162) and rhamnose (146), moiety]. Peak 14 exhibited a pseudo-molecular ion $[M–H]^-$ at *m/z* 753 and three fragments at 301, 609, and 651, assigned to

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Table 3. Retention time (RT), UV and mass spectral data and tentative identification of phenolic compounds in the aqueous extracts of *Citrus aurantium* and *Nerium oleander*

Peak No.	RT (min)	UV λmax (nm)	$[M-H]^-$ (m/z)	Other ions (*m/z*)	Probable compound	*Citrus auratium*	*Nerium oleander*
1	9.14	251	191	111, 173	quinic acid	−	+
2	16.74	268	287	575 $[2M-H]^-$, 323, 191, 125	eriodictyol	+	−
3	28.34	318	353	191, 179	caffeoylquinic acid isomer I acid	−	+
4	31.60	320	469	367, 191, 161, 425	limonin	+	−
5	33.01	249, 326	353	191, 179, 161	caffeoylquinic acid isomer II acid	−	+
6	35.76	271, 332	593	311, 649,497	unknown	+	−
7	36.64	259, 318	371	249, 555	unknown	−	+
8	38.47	284, 339	595	161, 191, 287	eriocitrin	+	−
9	40.89	226, 283	579	271	naringenin-rutinoside	+	−
10	41.85	284, 329	609	301	hesperidin	+	−
11	42.66	255, 353	609	301	quercetin rutinoside (rutin)	−	+
12	44.10	257, 327	441	407, 279, 485, 607	unknown	+	−
13	44.90	265, 335	593	285	kaempferol-rutinoside	−	+
14	45.12	284, 330	753	301, 609, 651	brutieridin	+	−
15	46.19	271, 332	593	–	unknown	+	−
16	49.14	257, 323	301	–	quercetin	+	−
17	49.40	257, 323	565	275, 279, 535, 601	unknown	+	−
18	49.74	270, 330	531	513, 305	unknown	−	+
19	52.75	270, 330	579	327, 309	unknown	−	+
20	54.32	274	515	191, 179	dicaffeoylquinic acid	−	+

(+) present; (−) absent

brutieridin. Limonoids were also detected in the *Citrus* sample. Peak 4 showed a pseudo-molecular ion $[M-H]^-$ at *m/z* 469 and was attributed to limonin. Peak 2 presented a pseudo-molecular ion $[M-H]^-$ at *m/z* 287. It was tentatively attributed to eriodictyol. Compound 16 showed a pseudo-molecular ion $[M-H]^-$ at *m/z* 301, corresponding to quercetin.

Six compounds were identified in *N. oleander* extract [quercetin-rutinoside (rutin), quinic acid, caffeoylquinic acid Isomer I, caffeoylquinic acid Isomer II, kaempferol-rutinoside, and dicaffeoylquinic acid] (Figure 4). Quercetin rutinoside (rutin) was the dominant compound (Peak 11, UV_{max} = 255 and 353 nm, $[M-H]^-$ at *m/z* 609 −› 301). Peak 1 exhibited a pseudo-molecular ion $[M-H]^-$ at *m/z* 191 attributed to quinic acid. Two quinic acid esters represented by caffeoylquinic acid Isomer I and II were detected in peaks 3 and 5, respectively. Peak 13 exhibited a pseudo-molecular ion $[M-H]^-$ at *m/z* 593 and one fragment ion $[M-162-146-H]^-$ at m/z 285, identified as kaempferol-rutinoside. Peak 20 ($[M-H]^-$ at *m/z* 515 −› 191, 179) was identified as dicaffeoylquinic acid.

Impact of the different treatments on *A. punicae* populations. Monitoring *A. punicae* (Figure 5) individuals in untreated trees (C) revealed that the average number of aphids did not have important variations, where the population decreased slightly from a maximum of 251.58 individuals on July 23 to a minimum of 144.08 individuals on August 6. In all treated trees, Aphid's average number decreased considerably just one day after the different treatments' first application (T1). Values were about 1.42, 2, 9.17, 9.33, and 16 individuals, respectively, in BC 1.5%, BFS 3%, NE, BE, and BFS 1.5%, while in control, it was about 213.58 aphids. That proves all treatments impacted aphids' populations one day after A1. During the third and seventh days after T1, *A. punicae* populations did not show any variations, and the number of individuals remained at low values. Fourteen days later, aphids' populations increased until reaching high values on August 7.

The same results were observed one day after the second application (T2) of the different treatments. The number of *A. punicae* individuals decreased considerably till the end of the trial, with some scattered single individuals ranging between a maximum of 1.33 and a minimum of 0.42 individuals for BFS 1.5% and BE, respectively (Figure 5).

Mortality rates caused by the different treatments showed that one day after T1, the highest percent-

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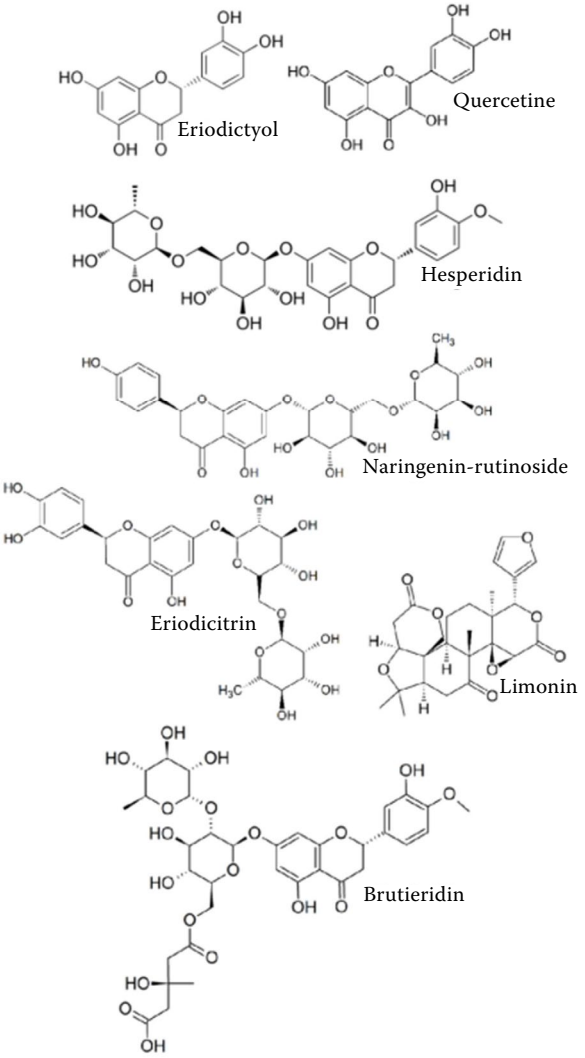

Figure 3. Chemical structures of the derivatives in *Citrus aurantium* extracts identified by HPLC–PDA/ESI–MS

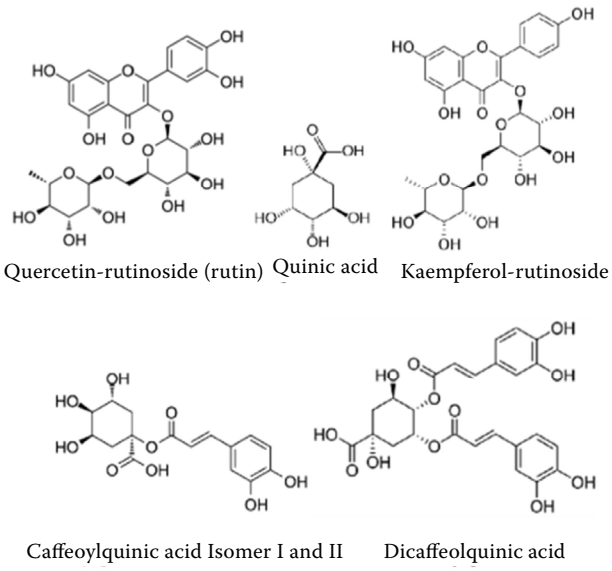

Figure 4. Chemical structures of the derivatives in *Nerium oleander* extracts identified by HPLC–PDA/ESI–MS

age of dead aphids was observed for BC 1.5% with 94.19%, followed by BFS 3% with 93.93%. No significant difference was observed for mortality rates caused by plant extracts, with average values of 77.38% and 77.74% for *Nerium* and bitter orange extracts, respectively (Figure 6). No significant differences were observed later between different treatments during a week after T1. 14 days after T1, mortality rates began to decrease, especially for plant extracts with average values of 44.42% and 49.94% for *Nerium* and bitter orange, respectively. Except for BFR 1.5%, treatments associated with basalt remained efficient, with average values over 60%. On August 8, mortality rates decreased to 29.18 and 41.55% for BE and BC 1.5%, respectively. That is related to the increase in aphids' populations observed during the same date. After T2, almost the same results were observed, with insignificant differences between treatments. Regarding BFR1.5%, it did not cause significant mortality of aphids. Mortality rates for this treatment ranged between a minimum of 2.53%, observed one day after T1, and a maximum of 26.15% registered 14 days after T2.

Efficacy rate evaluation showed no significant differences between applied treatments except for basalt used as fertiliser. For all foliar treatments, efficacy rates were above 80% (Figure 7).

Impact of the different treatments on *P. citri* populations. Regarding *P. citri* (Figure 9) in control plots (C), populations continuously increased to 43 individuals on July 26, 2020. Some variations were observed during the rest of the study period. Regarding all treated plots, the mealybug means a number of individuals decreased one day after the first treatment (T1) from 12.63 to 2.67, 19 to 7.92, 19.33 to 3.67, 18 to 3.25, and 19 to 5.83 for BSF 3%, BFS 1.5%, BC 1.5%, NE, BE, and BFR 1.5%, respectively. It must be noted that, unlike the results obtained concerning aphids, *P. citri* populations did not increase fourteen days after the first treatment and remained low and constant even after T2 till the end of the study period (Figure 8).

Monitoring *P. citri* mortality rates showed a significant difference between treated plots and control. One day after the first application of treatments, both basalt doses showed the highest

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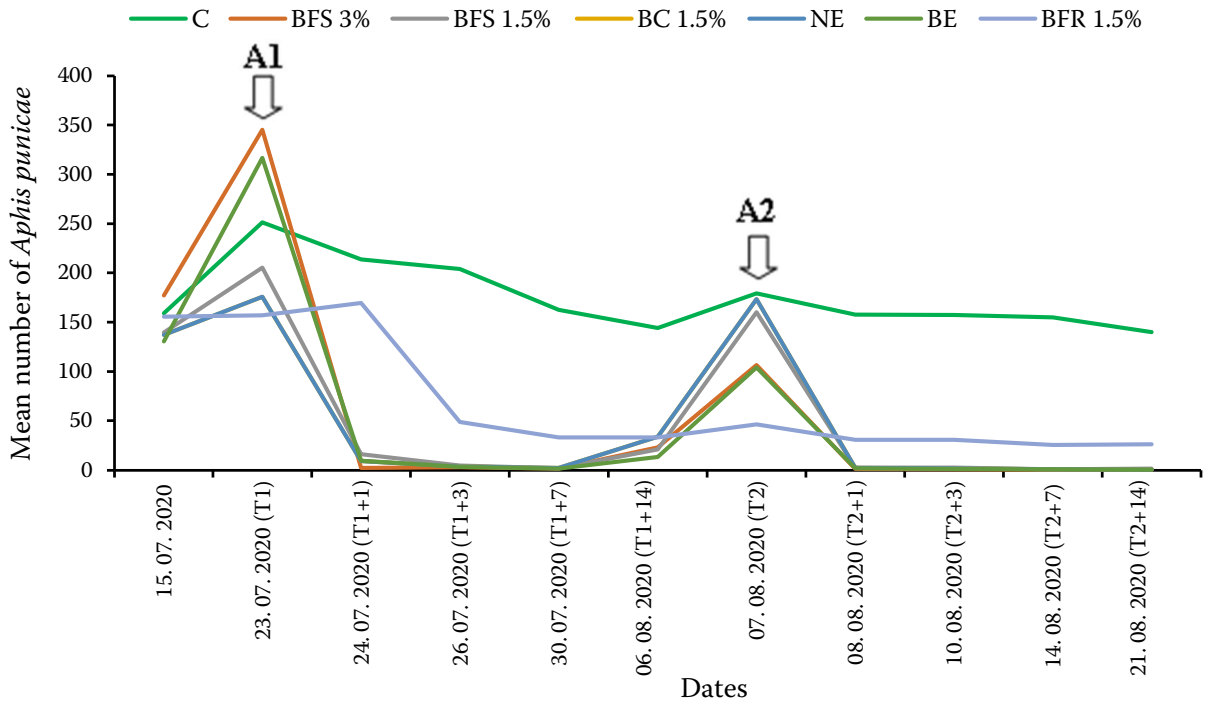

Figure 5. Monitoring alive *Aphis punicae* depending on treatments

A1 – 1st application; A2 – 2nd application; C – control; BFS 3% – basalt as a foliar spray at 3%; BFS 1.5% – basalt as a foliar spray at 1.5%; BE – bitter orange extract with 50 g/L; NE – nerium extract with 50 g/L; BFR – basalt as a fertiliser at 1.5%; BC 1.5% – combined basalt application as a foliar spray and as a fertiliser in the ground at 1.5%

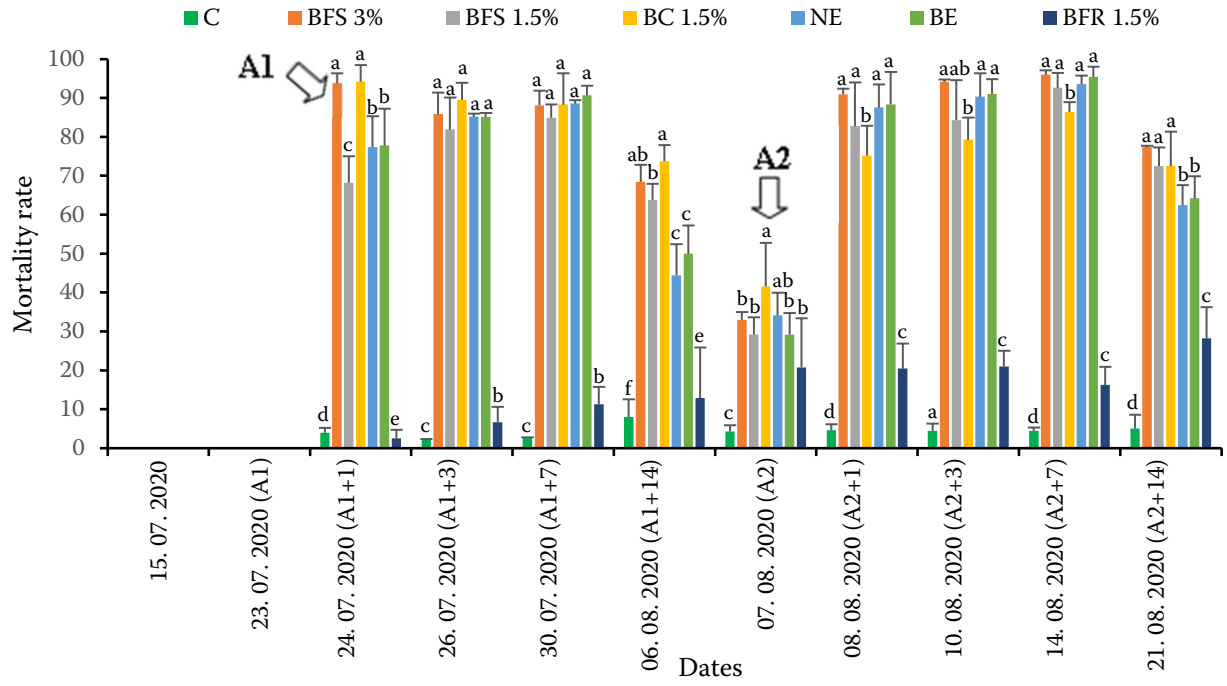

Figure 6. Mortality rates of *Aphis punicae* depending on treatments

A1 – 1st application; A2 – 2nd application; C – control; BFS 3% – basalt as a foliar spray at 3%; BFS 1.5% – basalt as a foliar spray at 1.5%; BE – bitter orange extract with 50 g/L; NE – nerium extract with 50 g/L; BFR – basalt as a fertiliser at 1.5%; BC 1.5% – combined basalt application as a foliar spray and as a fertiliser in the ground at 1.5% means followed by the same letter are not significantly different at $P \leq 0.05$

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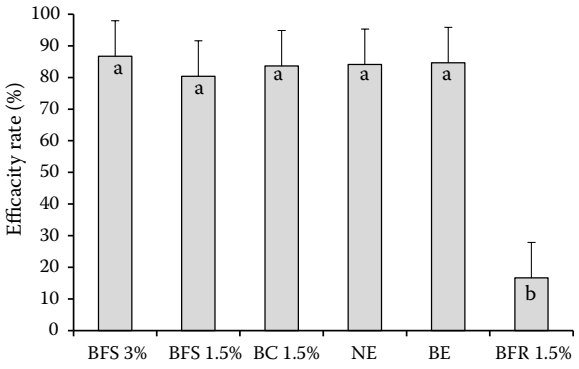

Figure 7. Efficacy rates of different applied treatments against *Aphis punicae*

BFS 3% – Basalt as a foliar spray at 3%; BFS 1.5% – Basalt as a foliar spray at 1.5%; BE – bitter orange extract with 50 g/L; NE – Nerium extract with 50 g/L; BFR – Basalt as a fertiliser at 1.5%; BC 1.5% – combined basalt application as a foliar spray and as a fertiliser in the ground at 1.5%
Means followed by the same letter are not significantly different at $P \leq 0.05$

mortality rates, with 58.95 and 57.00% for BSF 3% and BSF 1.5%, respectively. Regarding plant extracts, registered mortality rates one day after the first treatment were about 41.65% for bitter orange extract and 50.9% for *Nerium* extract, with no significant statistical differences. Two weeks after the first application of treatments, mortality rates increased considerably, reaching high average values of about 83.7% for BSF 3% and 82.98% for BE. The same results were almost noted after the second application. The lowest mortality rate values were observed in plots treated with a dose of 1.5% of basalt powder as a fertiliser in the ground, with the highest average value of about 32.19% fourteen days after the second application.

Regarding combined treatment (BC 1.5%), moderate mortality rates were observed after T1. However, after T2, average mortality rate values increased and showed no significant statistical difference from the other plots (Figure 9). Compared with aphids, mealybug mortality rates were lower and increased just after T2.

Efficacy rates evaluation showed that all treatments produced statistically significant values (above 50%) in comparison to the control (C) (20%). Nevertheless, BFS 3%, BE, and NE were more efficient, with 75%, 67%, and 65%, respectively. BFS 1.5% and BC 1.5% had statistically different efficacy rates compared to the previously mentioned treatments, with 60% and 52%, respectively (Figure 10).

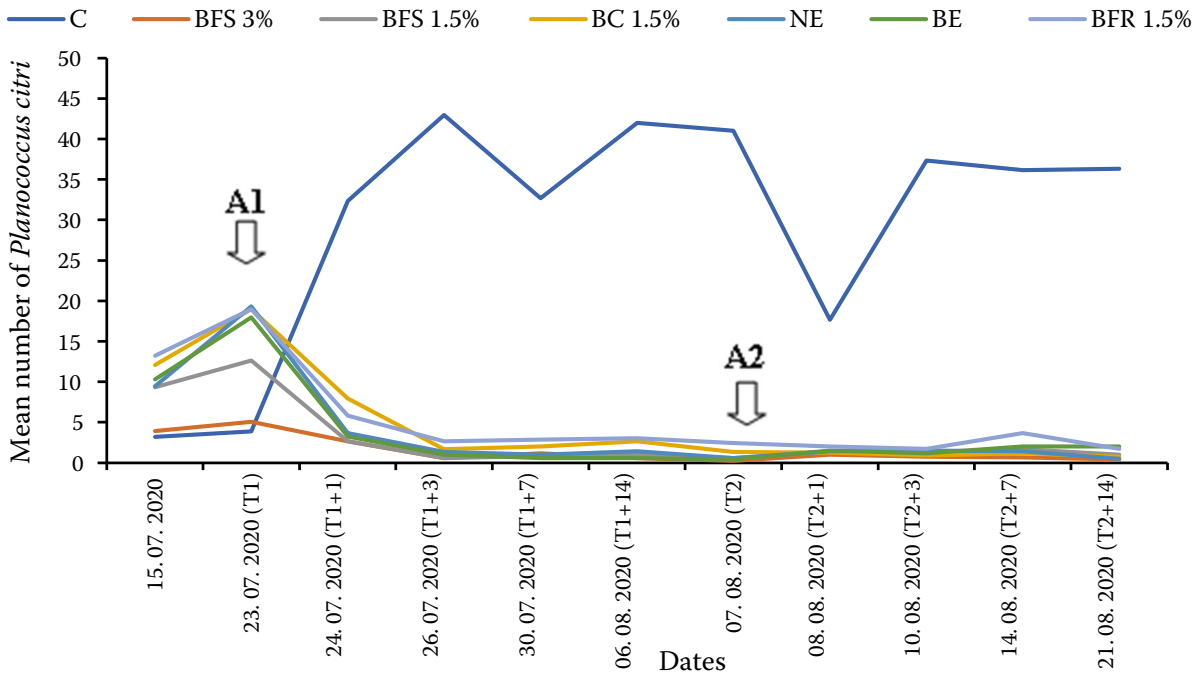

Figure 8. Monitoring alive *Planococcus citri* depending on treatments

A1 – 1st application; A2 – 2nd application; C – control; BFS 3% – basalt as a foliar spray at 3%; BFS 1.5% – basalt as a foliar spray at 1.5%; BE – bitter orange extract with 50 g/L; NE – nerium extract with 50 g/L; BFR – basalt as a fertiliser at 1.5%; BC 1.5% – combined basalt application as a foliar spray and as a fertiliser in the ground at 1.5%

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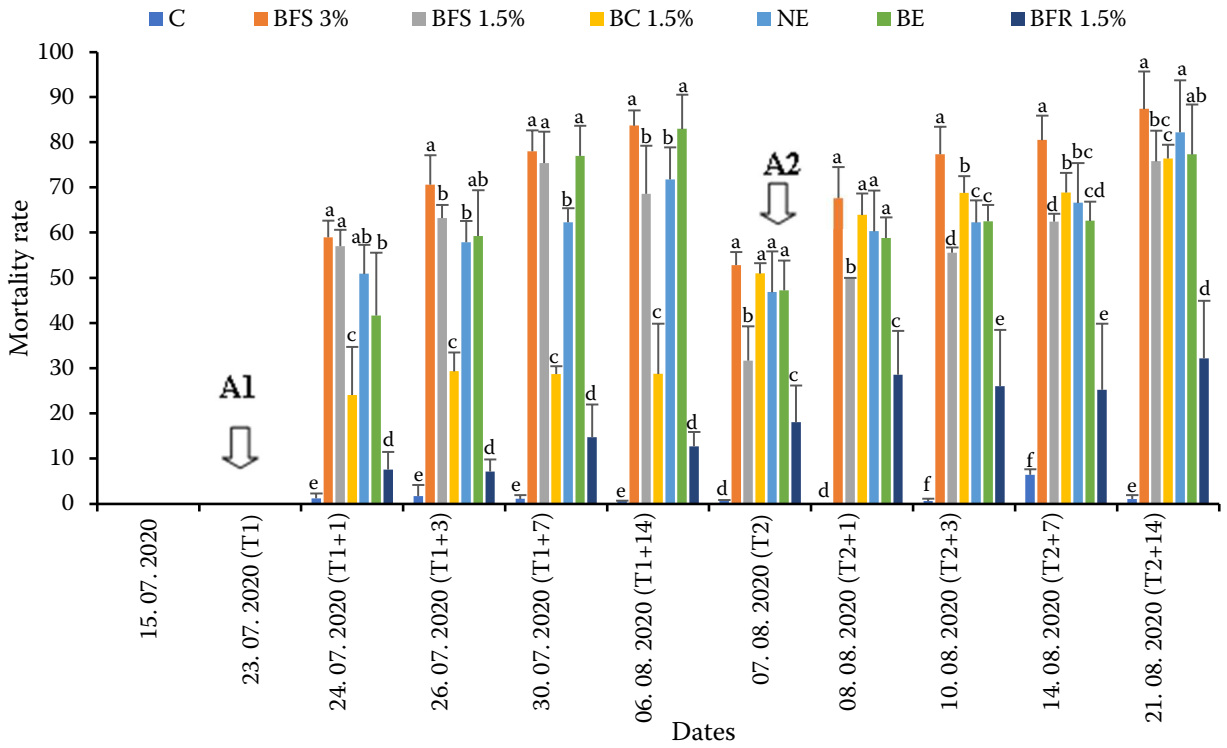

Figure 9. Mortality rates of *Planococcus citri* depending on treatments

A1 – 1st application; A2 – 2nd application; C – control; BFS 3% – basalt as a foliar spray at 3%; BFS 1.5% – basalt as a foliar spray at 1.5%; BE – bitter orange extract with 50 g/L; NE – nerium extract with 50 g/L; BFR – basalt as a fertiliser at 1.5%; BC 1.5% – combined basalt application as a foliar spray and as a fertiliser in the ground at 1.5%

Means followed by the same letter are not significantly different at $P \leq 0.05$

DISCUSSION

This study has proven the efficacy of basalt powder and both botanical aqueous extracts of *C. aurantium* and *N. oleander* against *A. punicae* and *P. citri* on pomegranate fruits.

Our findings highlight recent works that revealed basalt powder's role in enhancing crops' tolerance to pests (Rouz et al. 2020; Elimem et al. 2021). Indeed, basalt powder has been used to control insects and diseases in crops and stored products (Fawzy et al. 2012; Isnugroho et al. 2018; Elimem et al. 2020; Elimem et al. 2021). Mewis and Ulrichs (2001) demonstrated that silicon dioxide is the active ingredient that gives basalt powder its pesticide activity. Inert flours or powders like basalt caused the desiccation of pests and disease agents. Basalt causes insects' death by absorbing the waxy layer surrounding the exoskeleton, which causes death by desiccation (Ebeling 1971; Elimem et al. 2021). Also, they have repellent activity against insects (Ebeling 1971). For example, Guèvel et al. (2007) affirmed that basalt powder decreased the severity of powdery mildew disease due to its richness in silicon. Groth et al. (2017) have shown the effectiveness of basalt flour in reducing pest pop-

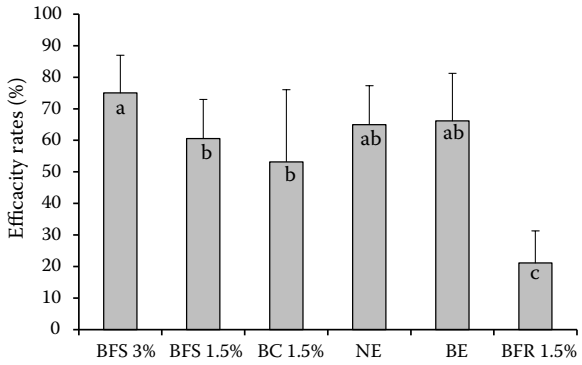

Figure 10. Efficacy rates of different applied treatments against *Planococcus citri*

BFS3 % – basalt as a foliar spray at 3%; BFS 1.5% – basalt as a foliar spray at 1.5%; BE – bitter orange extract with 50 g/L; NE – nerium extract with 50 g/L; BFR – basalt as a fertiliser at 1.5%; BC 1.5% – combined basalt application as a foliar spray and as a fertiliser in the ground at 1.5%

Means followed by the same letter are not significantly different at $P \leq 0.05$

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ulation dynamics on lettuce. Cremonez et al. (2020) demonstrated basalt powder's efficacy in controlling *Tribolium castaneum* in stored canola. That makes basalt powder an interesting product to consider in Integrated Pest Management programs against pests and diseases of crops and stored products.

Several plants contain natural insecticidal products that can kill pests at a very moderate dose. For example, phenols are secondary metabolites that exhibit an important role in protecting plants against insects (War et al. 2012). They are attractive alternatives to synthetic chemicals because they are highly effective, safe and ecological (Siskos et al. 2009). The HPLC–PDA/ESI–MS analysis allowed the identification of seven flavonoid derivatives in *C aurantium* extracts, where hesperidin was the major. Our results affirmed the abundance of flavonoids in *Citrus* plants (Shi et al. 2007; Scordino et al. 2011; El-Sayed et al. 2017; Baron et al. 2021; Wen et al. 2021). Overall, the results indicated that the aqueous extract of *C. aurantium* was toxic to *A. punicae* and *P. citri*. In line with that, War et al. (2012) reported that flavonoids protect plants against pests by influencing insect behaviour, growth, and development. Our study's findings agree with those of Zarrad et al. (2017), affirming that *Citrus* species are a source of botanical insecticides. The richness of the extracts in hesperidin can be the origin of this activity. Indeed, this flavanone, predominantly found in *Citrus* (Noshy & Azouz 2021), has a high insecticide activity (Wang et al. 2016). Concerning *N. oleander* extracts, six compounds were identified, with quercetin rutinoside (rutin) as the most important one. These compounds were also detected by Ayouaz et al. (Ayouaz et al. 2021) in Algerian *N. oleander* extracts, which are characterised by the abundance of chlorogenic acid, rutin, and quinic acid esters. This plant species has been known worldwide as poisonous and can be used to control pests (Bari et al. 2020). Rutin is well-known as a plant flavonoid mediating insect-plant relationship. It causes multiple effects on insect behaviour depending on the insect and plant species involved (Stec et al. 2021). On the other hand, other studies demonstrated that *C. aurantium* extracts and essential oils showed an insecticidal impact and a repellent effect on several economic pests such as *C. capitata* and *Bactrocera oleae* Rossi (1790) (Diptera, Tephritidae) (Siskos et al. 2009), *Spodoptera frugiperda* Smith (1797) (Lepidoptera: Noctuidae) (Villafane et al. 2011), the whitefly *Bemisia tabaci* Gennadius (1889) (Hemiptera; Aleyrodidae) (Zarrad et al. 2015) and scale insects such as mealybugs (Majeed et al. 2018). Moreover, it must be noted that flavedo of standard bitter contains secondary metabolites with insecticidal activity (Siskos et al. 2009). Regarding *Nerium*, many studies proved its insecticidal and bactericidal effects (Ali et al. 2008). According to several studies, *N. oleander* has been shown to cause mortality of several insect species, such as *Acanthoscelides obtectus* Say (1831) (Coleoptera, Bruchidae) (Laib 2014), *Paederus fuscipes* Curtis 1826 (Coleoptera, Staphylinidae) (Gupta et al. 2017), and the processionary moth *Thaumetopoea wilkinsoni* Tam (1924) (Lepidoptera, Notodontidae) (Semiz 2017).

CONCLUSION

The objective of this study is to test and prove the effectiveness of two application modes of basalt powder (foliar and in the ground, with two doses) and two botanical aqueous extracts of *C. aurantium* and *N. oleander* against *A. punicae* and *P. citri* on pomegranate fruits. The results obtained demonstrated the efficacy of the mineral product and the botanical aqueous extracts in the control of the pest. They highlighted that the methods of application of the basalt powder significantly influence its role in crop protection. Moreover, identified compounds of both plants' extracts may be purified and employed to protect crops from other pests.

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