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ORIGINAL PAPER

Antifeedant Activities of Organic Fractions from *Cestrum parqui* Leaves on the Red-Haired Bark Beetle *Hylurgus ligniperda*

Claudia Huanquilef[1,2] · Javier Espinoza[2,3] · Ana Mutis[2,3] · Leonardo Bardehle[2,4] · Emilio Hormazábal[2,3] · Alejandro Urzúa[1] · Andrés Quiroz[2,3]

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Abstract

Hylurgus ligniperda is one of the most important quarantine forest insects associated to pine (*Pinus radiate* D. Don) logs exported from Chile. Methyl bromide fumigation has been the prominent control method. As the use of this synthetic insecticide leads to serious environmental problems, research to find alternative treatments is urgently needed for the Chilean forestry sector. Hence, plant secondary metabolites have been considered as an alternative for its control. Therefore, the objective of this work was to assess the feeding behavior of *H. ligniperda* through the artificial diet supplemented with organic fractions obtained from *Cestrum parqui* leaves. Organic extracts were obtained using a Soxhlet extraction. Non-choice tests were developed for testing the antifeedant activity shown by the organic fractions on adults, and 5th and 6th instar larvae of *H. ligniperda*. All the extracts tested elicited a decrease in the weight of unsexed *H. ligniperda* adults, and the effects were dose-dependent. Male beetle weight gain was reduced strongly by chloroform extracts and ethyl acetate elicited a weight increase of female and from the fifth and sixth instar larvae weight. Saponin extract elicited a reduction of the weight in male and female. Finally, the weight of both instar larvae was reduced by chloroform and saponin extract. *C. parqui* leaf extracts show a great potential for being used for controlling *H. ligniperda* and thus to diminish the use of harmful synthetic pesticide.

Keywords *Cestrum parqui* · *Hylurgus ligniperda* · Chloroform extract · Saponins · Antifeedant activity

1 Introduction

Hylurgus ligniperda Fabricius (Coleoptera: Curculionidae), commonly known as red-haired bark beetle, is an exotic and invasive Eurasian species with a worldwide distribution (Ciesla 1993; Romo et al. 2016), considered one of the main quarantine conifer pests causing significant economic losses in the production of lumber and logs of radiata pine (*Pinus radiata* (D. Don)) (Lanfranco et al. 2002, 2004; Ruiz and Lanfranco 2008). Moreover, it represents an important threat to all coniferous forests around the world (Brockerhoff et al. 2006) because it is a vector for *Ophiostoma*, *Grosmannia*, and *Leptographium* fungi agents implicated in root decline disease and blue-stain (Kim et al. 2011). It has also been observed that *H. ligniperda* adults attack the root collar of 1- or 2-year-old seedlings, which can cause the seedling's death. This type of damage has only been observed in *P. radiata* forest in Chile (Ayres and Lombardero 2000; Lanfranco et al. 2004; Mausel et al. 2007). However, there is no official report about the economic loss caused by this damage. On the other hand, exporting of Chilean forest products, such as sawn wood to Mexico, was suspended in 2006 due to the presence of *H. ligniperda* adults in some shipments. Since, *H. ligniperda* has been recognized as a serious problem for the Chilean forestry sector (Ruiz and Lanfranco 2008), currently, methyl bromide fumigation has been the mostly used method to *H. ligniperda* control. Nevertheless,

✉ Andrés Quiroz
andres.quiroz@ufrontera.cl

[1] Doctorado en Ciencias de Recursos Naturales, Universidad de La Frontera, Av. Francisco Salazar, 01145 Temuco, Chile

[2] Centro de Investigación Biotecnológica Aplicada al Medio Ambiente (CIBAMA), Universidad de La Frontera, Av. Francisco Salazar, 01145 Temuco, Chile

[3] Departamento de Ciencias Química y Recursos Naturales, Facultad de Ingeniería y Ciencias, Universidad de La Frontera, Av. Francisco Salazar, 01145 Temuco, Chile

[4] Departamento de Producción Agropecuaria, Facultad de Ciencias Agropecuarias y Forestales, Universidad de La Frontera, Temuco, Chile

international environmental organisms have banned its use for the next decade because it is considered an ozone-depleting substance (Clare and George 2016). In addition, the use of organophosphates, carbamates, and pyrethroids has low performance controlling this pest. Furthermore, their continuous use provokes the emergence of resistant pests as well as the accumulation of non-biodegradable residues in the environment that are highly toxic to non-target organisms (Yang et al. 2014). Hence, research to find controlling alternative treatments is urgently needed.

Nowadays, plant natural products have attracted much attention as a safe, effective, and environmentally friendly alternative to replace harmful conventional pesticides (Chaieb 2010; De Geyter et al. 2007; D'Incao et al. 2012; Pavela 2016). Many studies have reported that crude extracts or bioactive constituents from medicinal plants exert detrimental effects on survival, growth, and insect reproduction because of their toxic and deterrent activities (Chaieb 2010; De Geyter et al. 2011; Hussein et al. 2005; Saha et al. 2010). These activities are probably due to synergism among the different components present in the extracts (Guo et al. 2013).

In the last decades, pesticide activities associate to *Cestrum parqui* (L'Hér.) (Solanaceae) extracts have been reported (Chaieb 2010). This plant is a 2–3-m-tall perennial shrub, native to Central and South America, which has been introduced throughout the world as an evergreen ornamental plant (Navas 1979). *C. parqui* leaves have been used as an herbal medicine in traditional Mapuche medicine for the treatment of allergies, herpes, impetigo, and headache (Estomba et al. 2005). In addition, several studies have also reported that this plant possess antiplatelet, anticancer, and spermicidal effects (Chenni et al. 2015; Falkenberg et al. 2012; Souad et al. 2007). Moreover, previous phytochemical studies have shown that alkaloids and saponin extracts from leaves of *C. parqui* could be the main active compounds responsible of biological activities (Abdel-Gwad et al. 1997; Baquai et al. 2001; Brevis et al. 1999; Chaieb et al. 2007a; Mosad et al. 2017; Silva et al. 1962). Nevertheless, lignans, neolignans, and sesquilignans (D'Abrosca et al. 2006; Fiorentino et al. 2007); sapogenin (Bianchi et al. 1963; Torres et al. 1988), terpenoids (D'Abrosca et al. 2004a, 2005; Pearce et al. 1992), and phenols (D'Abrosca et al. 2004b) are also present in *C. parqui* leave extracts. In the last decades, diverse reports have demonstrated pesticide activities from *C. parqui* extracts (Chaieb 2010; Zapata et al. 2006). For example, aqueous extracts from *C. parqui* demonstrated a high toxicity to neonate larvae of *Ceratitis capitata* (Wiedemann) (Diptera: Tephritidae) when it was incorporated to a diet at a concentration above 0.6% (Zapata et al. 2006). Additionally, *C. parqui* steroidal crude saponins exerted toxic effects on molting process of *Spodoptera littoralis* (Lepidoptera: Noctuidae) (Chaieb et al. 2007b) and it produced ecdysal disturbance on *Schistocerca gregaria* (Orthoptera: Acrididae) (Barbouche et al. 2001). Moreover, structural modifications at the fat body of *S. littoralis* were observed in histological sections. Similar effect was observed in the foregut and gastric caeca of *S. gregaria* (Chaieb et al. 2007b) and *S. littoralis* larvae (Chaieb et al. 2007c). Because of the good activities showed by *C. parqui* extracts on different insets belonging to different families, we aim to elucidate the antifeedant effect elicited from chloroform, ethyl acetate, and saponin crude extract from *C. parqui* leaves on *H. ligniperda* one of the main pest associated to lumber and logs of radiata pine. This is the first record showing the biological activity of *C. parqui* leaves on a curculionid.

2 Materials and Methods

2.1 Plant Material

C. parqui leaves were collected in October 2015 in Hualpén Botanical Park (36° 48′ S–73° 10′ W) at the Universidad de Concepción, Chile. The identification of the specimen was corroborated in the Departamento de Botánica, Facultad de Ciencias Naturales y Oceanográficas, Universidad de Concepción, Chile. The samples were identified by biologist and botanist MSc. Alicia Marticorena.

2.2 Insects

Both adults and larvae of *H. ligniperda* were hand collected under the bark of infested *P. radiata* logs obtained from sawmills in Pitrufquén (38° 59′ S–72° 38′ W), La Araucanía Region, Chile, in November 2017. The individual beetles were transferred to a small cage (5 cm × 2 cm × 2 cm) and then reared in entomological cages (30 cm × 30 cm × 30 cm) at 26 ± 1 °C under a 16:8 light:dark cycle and 70% humidity. The adults were starving for 48 h before bioassay.

2.3 Obtaining of *C. parqui* Extracts

Extracts were obtained from *C. parqui* leaves following the methodology described by Makkar et al. (2007) with some modifications. Briefly, dried leaves of *C. parqui* (500 g) were milled and defatted three times with dichloromethane in a Soxhlet apparatus, at ~ 50 °C for 24 h. The solvent was removed by a rotary evaporator, under reduced pressure at ~ 40 °C. The defatted plant material was extracted with ethanol by a Soxhlet apparatus, at ~ 50 °C for 24 h and the solvent was removed using a rotary evaporator yielding a dark green ethanolic extract (150 g). Then, it was suspended in 200 ml of distilled water. The aqueous solution was partitioned with chloroform, ethyl acetate, and *n*-butanol to obtain three fractions. Each fraction was dried with anhydrous Na_2SO_4, filtered, and evaporated under reduced pressure on rotary evaporator.

2.4 No-Choice Feeding Bioassays

The feeding bioassays were performed under a no-choice test using an artificial diet, according to Faccoli and Schlyter (2007). Briefly, 500 μl of artificial diet, consiting of 80.4% water, 4.3% cellulose, 4.3% milled pine bark, 2.2% glucose, and 8.7% agar, was added into transparent microcentifugue tubes (Eppendorf, 10-mm diameter × 35-mm length). Then, solutions of chloroform extract, ethyl acetate extract, and saponin-rich fraction (60 μl), diluted in their respective solvent, were added separately, at different concentrations (Table 1), to the artificial diet. Artificial diets (500 μl) supplemented with 60 μl of chloroform, ethyl acetate, and distillated water, respectively, were used as blank. To ensure a homogeneous distribution of the solution, the microcentrifuge tubes were vortexed at 12 rpm continuously for 15 s. The microcentrifuge tubes were incubated overnight at 20 ± 5 °C to remove solvents. Subsequently, insects were weighed (i_w) and then were introduced in each tube, which were closed with a plastic cap. Each bioassay was replicated 15 times with unsexed beetles, 15 times with male beetles, and another 15 times with female beetles. Each insect was used only once. The feeding performance was evaluated at room temperture (20 ± 5 °C) for 7 days under dark conditions. After, the insects were removed from the tubes and were weighed again (*fw*). The feeding performance was evaluated by the weight shift (%), as follows (Toledo et al. 2014; Quiroz et al. 2017; Espinoza et al. 2018): weight shift (%) = $(f_w - i_w)/f_w \times 100$.

Similar feeding bioassay described above was carried out on fifth and sixth instar larvae of *H. ligniperda* using an artificial diet consisting of 79.3% water, 6.9% agar, 3.4% cellulose, 3.4% pine bark, and 3.4% wheat germ. Each bioassay was performed 15 times.

2.5 Statistical Analysis

The statistical software Statistix 10 (Tallahassee, FL, USA) was used to analyze the data. The Shapiro-Wilk test was used to test whether data conform to a normal distribution. When data were normally distributed, the differences in the weight shift of *H. ligniperda* among different artificial diets were analyzed using a one-way ANOVA test ($P \leq 0.05$) with a post hoc Tukey HSD test. On the contrary, when data were not normally distributed, it was analyzed by a nonparametric one-way Kruskal-Wallis test ($P \leq 0.05$) with a post hoc Dunn's test. The results were expressed as means and their corresponding standard errors.

Table 1 Concentrations of the tested extract solutions and concentration of each extract in diet using in the feeding bioassay

Extracts	Concentration in solution (% w/v)	Concentrations in diet matrix (% w/v)
Chloroform, ethyl acetate, and saponin crude	0.20	0.02
	0.40	0.05
	0.60	0.07
	0.80	0.10
	1.00	0.12

3 Results

3.1 *C. parqui* Extracts

Chloroform, ethyl acetate, and *n*-butanol extracts yielded 4.57 g (0.9%), 6.00 g (1.2%), and 9.67 g (1.9%) respectively. Then, the *n*-butanol extract was chromatographed on Sephadex LH-20 column, using MeOH/H2O 1:1 as eluent. Twenty fractions were collected (7 ml each), concentrated, and monitored by thin layer chromatography (TLC) using silica gel–coated aluminum plates (Merck, GF254) developed using BuOH/AcOH/H2O (7:1.15:1.85) and were examined under UV light at 254 and 365 nm. Ehrlich's reagent was used to assess for presence of steroidal saponins. Finally, saponin-rich fractions were combined yielding 1.96 g of a saponin fraction (20.3%).

3.2 Feeding Bioassays of Unsexed Adults Individual

Feeding behavior of the unsexed *H. ligniperda* adults was affected by the extracts added to diet, and the effects were dose dependent. In comparison with their own blank, chloroform extract (Fig. 1A), ethyl acetate extract (Fig. 1B) and saponin extract (Fig. 1C) elicited a significant reduction in the weight gain of the *H. ligniperda* adults. In average chloroform, ethyl acetate and saponin extracts elicited a significant decreasing in the weight of *H. ligniperda* adults by approximately 57%, 28%, and 82% respectively (Fig. 1). In detail, higher antifeedant activity of 66.4%, 73.0, and 75.4% was found with chloroform extract of *C. parqui* at 0.6%, 0.8, and 1% concentrations respectively (Fig. 1A). A lower activity observed with ethyl acetate extract at 32.7%, 32.6%, and 38.5% was found at 0.6%, 0.8%, and 1% concentrations respectively (Fig. 1B). Highest antifeedant activities of 81.0%, 86.0, and 81.3 were found with chloroform extract of *C. parqui* at 0.6%, 0.8, and 1% concentrations respectively (Fig. 1C).

3.3 Feeding Bioassays of Adult Male and Female

Then, saponin-rich extract and chloroform extract were the most active feeding deterrent, but the lowest concentrations (0.2% and 0.4%) were not active. Organic extracts elicited different feeding behavior from both male and female

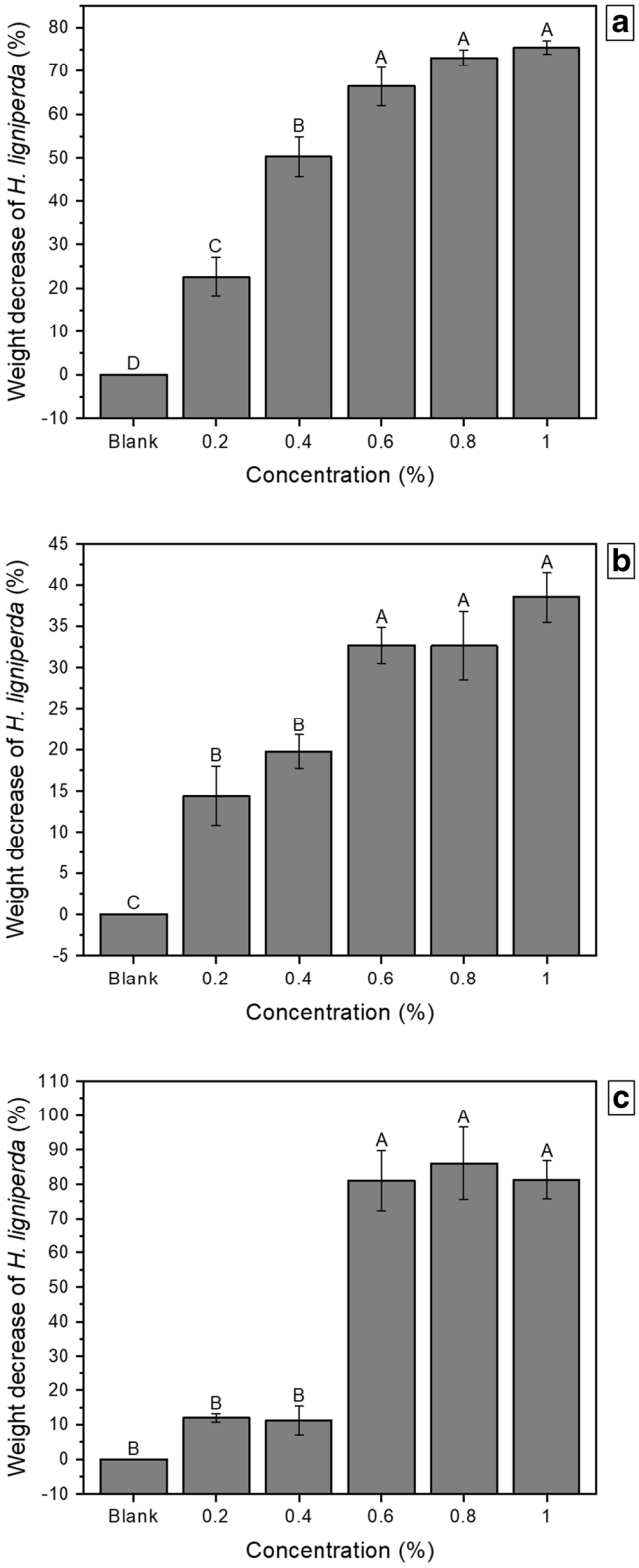

Fig. 1 Weight loss/gain (%) of *H. ligniperda* adults fed with artificial diet supplemented with (A) chloroform extract, (B) ethyl acetate extract, and (C) saponin extract. Blank: artificial diet supplemented with 10 μL of respective solvent. Values indicate mean ± SE. Different letters indicate significant differences ($P \leq 0.05$)

H. ligniperda (Fig. 2). Male beetle weight was reduced by chloroform extract by most of the concentrations, in comparison with the blank. In contrast, female beetle was not affected by this extract (Fig. 2A). The highest decreasing percentage of 195.7%, 280.5%, 298.5, and 286.2% were observed with chloroform extract at 0.4%, 0.6%, 0.8%, and 1.0% concentrations (Fig. 2A). A dissimilar behavior elicited ethyl acetate extract, while in males, it caused an average reduction of 41.9% between 0.6 and 1.0% concentrations, and in females, it elicited an average increase of 220.4% between 0.4 and 1.0% concentrations (Fig. 2B). The highest phagostimulant behavior was observed at 0.8% (262.5%) and 1.0% (283.5%) concentrations. Saponin extract reduced the weight gain of both male and female beetles (Fig. 2C). Significant antifeedant activity of 62.8%, 74.2%, 84.0%, and 93.3% was found with chloroform extract of *C. parqui* at 0.4%, 0.6%, 0.8, and 1% concentrations respectively for males and 46.8%, 74.2%, 53.6%, and 61.4% at the same concentrations were observed for females (Fig. 2C).

3.4 Feeding Bioassays of Larvae

The feeding behavior of 5th and 6th instar larvae was affected by diet in a different manner. Fifth and sixth instar larvae weight was reduced by chloroform extract in concentration-dependent manner, decreasing fifth larval weight by 588.4%, 851.8%, and 1122.6% to concentrations of 0.1%, 0.2%, and 0.4% respectively, and at the same concentrations, the weight of the sixth instar larvae varied between 151.3 and 255.6% (Fig. 3A). Again, the effect of the ethyl acetate extract was dissimilar in comparison with the other two extracts. To the highest concentration, the extract elicited a phagostimulant behavior on both fifth and sixth instar larvae, increasing the fifth and sixth larva weight in 368.0% and 49.3% respectively at 0.4% concentration. On the other hand, at the lowest concentration, it caused a reduction in the weight of larvae of the sixth instar in 29.1% at a concentration of 0.1% (Fig. 3B). Finally, saponin extract reduced the larva of both 5th and 6th instar, being significant only at 0.4% in the 5th instar and at 0.2% and 0.4% in the 6th instar (Fig. 3C). However, only saponin extract at 0.4% reduced the weight of fifth instar larvae by 150.7%. Moreover, a significant weight reduction on sixth instar larvae was observed at 0.2% and 0.4% concentrations respectively. These results show that chloroform extract was the most active feeding deterrent against *H. ligniperda* larvae.

4 Discussion

Most of the antifeedant studies with botanicals extracts have been carried out on Lepidoptera species existing limited information available about antifeedant activity against Coleoptera species. Moreover, to our knowledge, there is no information

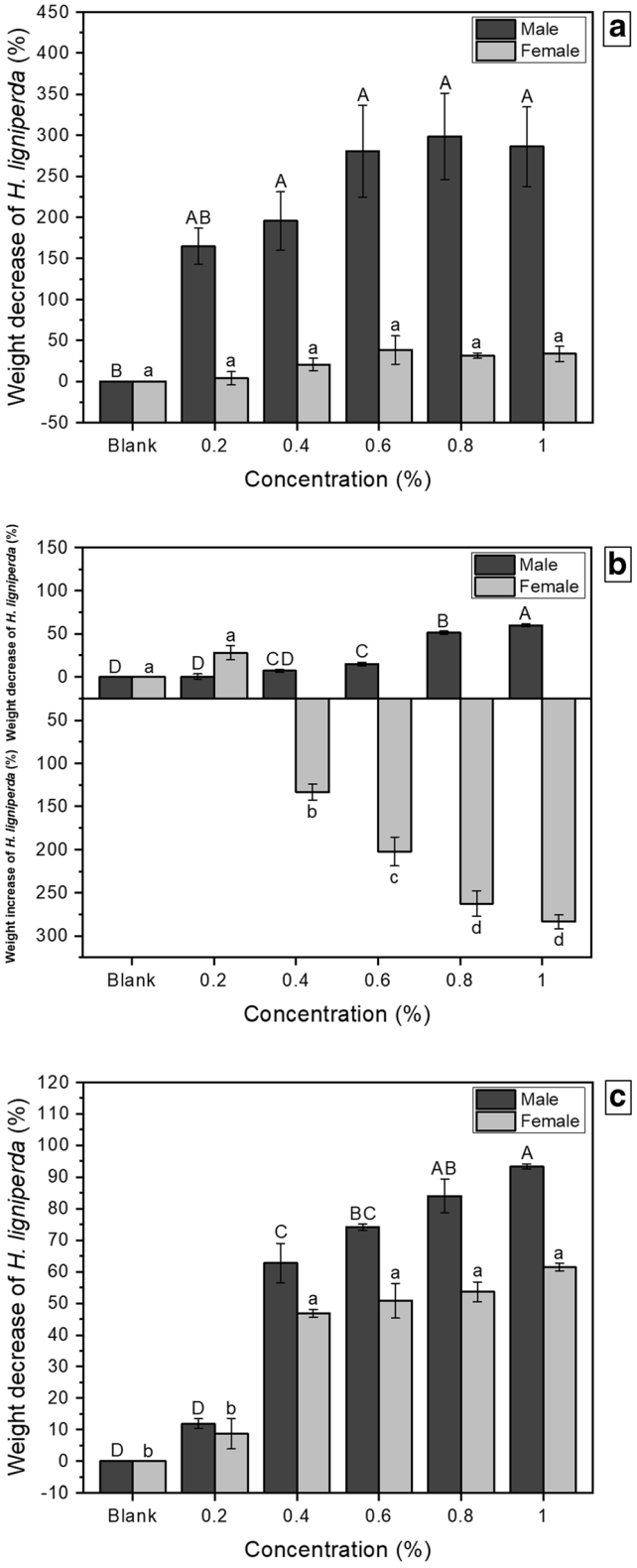

Fig. 2 Weight loss/gain (%) of male and female of *H. ligniperda* fed with artificial diet supplemented with (A) chloroform extract, (B) ethyl acetate extract, and (C) saponin extract. Control: artificial diet supplemented with 10 μL of respective solvent. Values indicate mean ± SE. Different letters indicate significant differences ($P \leq 0.05$)

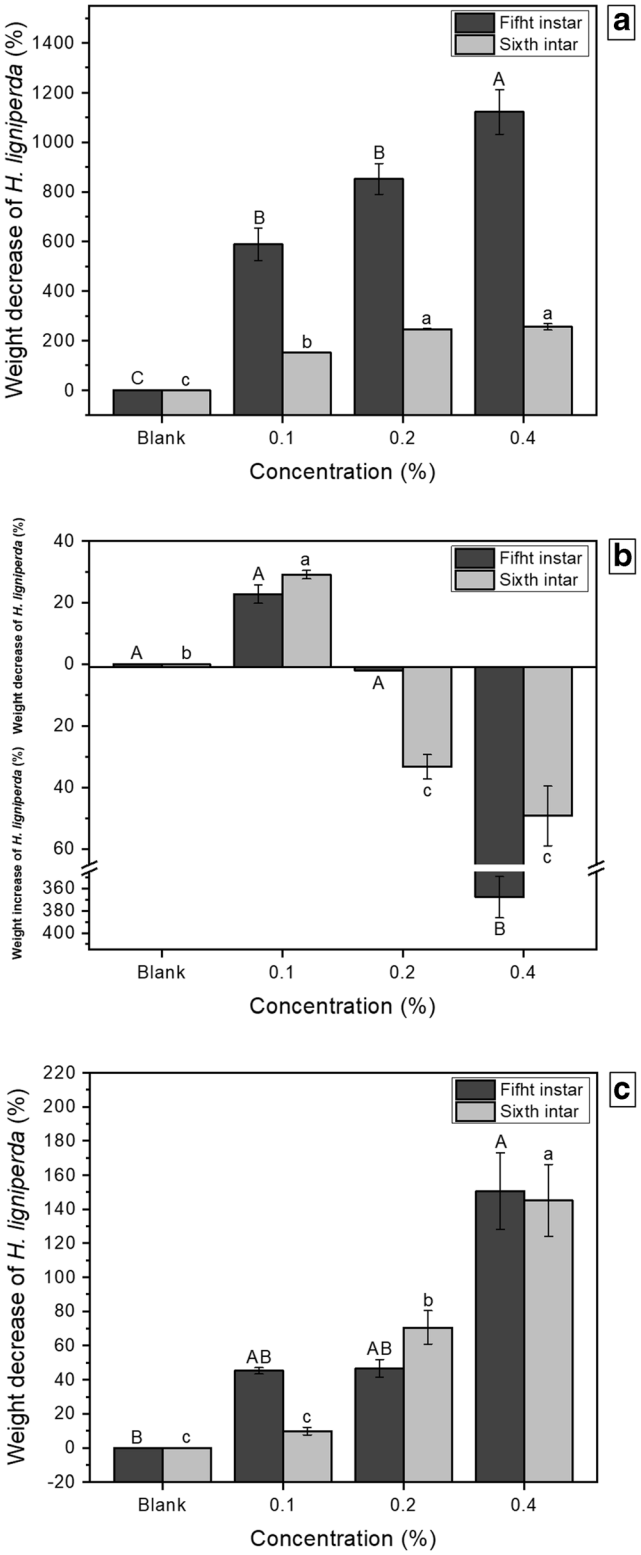

Fig. 3 Weight loss/gain (%) of *H. ligniperda* larvae fed with artificial diet supplemented with (A) chloroform extract, (B) ethyl acetate extract, and (C) saponin extract. Control: artificial diet supplemented with 10 μL of respective solvent. Values indicate mean ± SE. Different letters indicate significant differences ($P \leq 0.05$)

about curculionid. In this study, we demonstrated, for first time, that the chloroform extract and saponin extract from *C. parqui* leaves inhibited the feeding behavior of a curculionid, *H. ligniperda*, acting as feeding deterrent agents. Similarly, Chaieb and Ben Halima (2009) demonstrated that a crude saponin extract of *C. parqui* acted as an antifeedant agent against *Spodopotera littoralis* (Lepidoptera: Noctuidae) and *Tribolium confusum* (Coleoptera: Tenebrionidae) in a feeding bioassay using artificial diet at 2% concentration. In the same way, many chloroform extracts from plants have shown antifeedant activities against Lepidoptera species. For example, chloroform extracts of *Atalantia monophylla* (Baskar et al. 2009), *Caesalpinia bonduc* (Baskar et al. 2018), and *Clerodendrum phlomidis* (Duraipandiyan et al. 2015; Muthu et al. 2015) were antifeedant agents against *Helicoverpa armigera* (Lepidoptera: Noctuidae) at concentrations between 0.02 and 5%. Additionally, chloroform extract of *Gloriosa superba* elicited an antifeedant behavior from *Spodoptera litura* (Lepidoptera: Noctuidae) (Nebapure et al. 2016) at 0.03% concentration. The antifeedancy of chloroform plant extract has been associated with their chemical composition. Secondary metabolites, such as coumarins, flavonoids, phenols, quinones and terpenoid, from *C. bonduc* chloroform extracts exhibited antifeedant activity against *S. litura* (Baskar et al. 2012). For instance, sesquiterpenes from *Pilgerodendron uviferum* heartwood produced antifeedant effects on *Hylastinus obscurus* (Coleoptera: Curculionidae) (Espinoza et al. 2018). Similarly, red clover root isoflavonoids and long chain fatty acids acted as feeding deterrents against *H. obscurus* (Quiroz et al. 2017; Toledo et al. 2014), demonstrating that several classes of secondary metabolites from plants can act as antifeeding agents against insects.

At the present study, males were more susceptible to chloroform and saponin fraction than females. Similar result was reported by Faccoli et al. (2005) against *Ips typographus* (Coleoptera: Curculionidae), who evaluated the feeding performance in a bioassay using an artificial diet very similar to use in this study but supplemented with terpenes. Moreover, the authors indicated that *I. typographus* males and females reduced feeding in concentration-dependent manner, at concentrations between 0.3 and 1.0%. Therefore, different compounds and doses have distinct effects on feeding responses of males and females, where sex is an important factor to consider.

On the other hand, many saponin compounds elicit a reduction of the food intake for several insect species producing nutritive deficiency that can cause death or inhibit the evolution for next stage (Chaieb 2010; De Geyter et al. 2007; Dowd et al. 2011; Gao et al. 2010). Adel et al. (2000) suggested that the addition of saponins to artificial diet is the cause that sterols are not uptake in the intestine, causing low nutrient availability for the normal development and survival of the insect. Also, it has been suggested that saponins could interact with digestive enzymes forming a complex with them causing a decrease in enzyme digestive capabilities (De Geyter et al. 2007; Singh et al. 2017; Taylor et al. 2004). In this context, Pedersen et al. (1976) attributed the high resistance of six alfalfa cultivars against pea aphids to high contents of saponins. Golawska and Lukasik (2009) reported the preference of aphids for alfalfa plant with low saponin levels, and Shinoda et al. (2002) found that the resistance of the wild crucifer *Barbara vulgaris* to the diamond back moth, *Plutella xylostella* (Lepidoptera: Yponomeutidae), was due to the presence of triterpene glycoside in the plant. Szczepanik et al. (2004) reported that saponins from three *Medicago* species, *Medicago arabica*, *M. hybrida*, and *M. murex*, showed feeding deterrent effects on *Leptinotarsa decemlineata* (Coleoptera: Chrysomelidae) larvae. Additionally, saponins produced also a reduction of feeding intake, larval growth, and survival against larvae (Szczepanik et al., 2001). Accordingly, in our research, saponin extract exhibited antifeedancy against *H. ligniperda* males, females, and larvae. In contrast, Hussein et al. (2005) reported the effect of saponins from alfalfa roots on *Tropinota squalida* (Coleoptera: Scarabaeidae), commonly named hairy rose beetle, demonstrating that adults fed with saponins lead to a high mortality, but the food consumption rate was not affected. On the other hand, Nielsen et al. (2010) determined that hederagenin cellobioside and oleanolic acid cellobioside saponins from *Barbarea vulgaris* inhibited feeding of the flea beetle, whereas the effect of oleanolic acid cellobioside was much weaker. However, the aglycones (sapogenins) were inactive. Therefore, glycoside chains play a significant role in the antifeedant activities of saponins.

Antifeedant activity elicited from ethyl acetate extract evaluated in this work was lower than the other *C. parqui* extracts. However, it has been demonstrated that ethyl acetate extracts from plants exhibit antifeeding activities against Lepidoptera species of economic importance (Baskar et al. 2011, 2012; Duraipandiyan et al. 2011; Raja et al. 2005; Pavunraj et al. 2011). Active concentrations ranged from 0.1 to 5.0%. For example, significant antifeedant activity (56.06%) was observed at 5.0% concentration when ethyl acetate leaf extract of *Aristolochia tagala* Cham. were tested against *S. litura* Fab. (Baskar et al. 2011). In the same way, ethyl acetate extract from *Hyptis suaveolens* L. leaves exhibited an antifeedant activity of 65.3% and 71.0% at 1% concentration against *H. armigera* and *S. litura* (Raja et al. 2005). Moreover, ononitol monohydrate and 6-(4,7-hydroxy-heptyl)-quinine isolated from ethyl acetate extract of *Cassia tora* L. exhibited a strong antifeedant activity of 74.57% and 69.05% against *H. armigera* and *S. litura*, respectively at 1% concentration and the activity was statistically significant over control (Baskar and Ignacimuthu 2012).

5 Conclusions

In the present study, we demonstrated that chloroform, ethyl acetate, and saponin extracts affected the feeding behavior of *H. ligniperda* adults as well as larvae, in a concentration-dependent manner. Chloroform extract elicited the highest antifeedant behavior, reaching more than 1000% of weight reduction at 0.4% concentration in fifth instar larvae and more than 195.7% in adult male at the same concentration. Our results are consistent with those from previous studies that showed deterrent activity of different plant extracts against bark beetle and, principally, Lepidoptera species at similar or lowest concentrations. The strong antifeedant activity shown by the chloroform extract of *C. parqui* leaves at low concentrations makes it a powerful alternative control tool for *H. ligniperda*. The relatively simple and cheap obtainment of this extract is very promising for its commercial application.

Code Availability Not applicable.

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Data Availability Not applicable.

Compliance with Ethical Standards

Ethics Approval Not applicable.

Consent to Participate The authors declare consent to participate in this work.

Consent for Publication The authors declare consent to publish this work.

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