Malpighia emarginata DC. bagasse acetone extract: Phenolic compounds and their effect on *Spodoptera frugiperda* (J.E. Smith) (Lepidoptera: Noctuidae)

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ABSTRACT

Annually, several tons of residues that are rich in phenolic compounds are produced during the processing of acerola (*Malpighia emarginata* DC.) juice. Adding value to these residues is of great interest, since they can be a viable solution in the search for natural substances with insecticidal action and low impact on the environment and humans. Taking into account the economic losses from the attacks by the fall armyworm *Spodoptera frugiperda* (J.E. Smith) (Lepidoptera: Noctuidae) in different crops, the objective of this study was to evaluate the effect of the extract of acerola bagasse flour (ABF) against this insect and determine the phenolic compounds in this extract. Bagasse of acerola (BRS238 or Frutacor clon) generated after juice production, was frozen and lyophilized. To obtain the extract, 6 g ABF was mixed with 60 mL acetone:water solution (7:3 v/v), and the extract was lyophilized. *Spodoptera frugiperda* caterpillars, 48 h-old, obtained by the maintenance breeding, were transferred to glass tubes supplied with an artificial diet containing the ABF extract at 0, 250, 500, 1000, and 2000 mg L^{-1} diet. The following variables were evaluated: duration and survival of larval and pupal stages, pupal weight, sex ratio, adult longevity, oviposition period, number of egg masses, and total number of eggs. The ABF extract contained several phenolic compounds including gallic acid, epigallocatechin gallate, catechin, *p*-coumaric acid, salicylic acid, and quercetin. The extract was toxic to *S. frugiperda*, prolonging the pre-pupal stage and increasing the mortality of caterpillars.

Key words: Acerola, fall armyworm, natural product, pest control, toxicity.

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INTRODUCTION

The search for natural products containing active ingredients against insect pests has intensified in recent years. The discovery of new substances as an alternative to synthetic insecticides is of great relevance, because the application of synthetic insecticides, besides causing adverse environmental effects, can result in the select for resistant insect populations, residue accumulation in food, and mortality of natural enemies (Busato et al., 2006; Souza et al., 2014; Zhu et al., 2015).

Among the numerous insect pests of economically important crops, fall armyworm *Spodoptera frugiperda* (J.E. Smith) (Lepidoptera: Noctuidae) is significant. It is a polyphagous and cosmopolitan insect, widely distributed in North and South America, that causes damage to several crops, including cotton, corn, sorghum, and other grasses (Múrua et al., 2009; Casmuz et al., 2010). In corn plantations, this pest causes defoliation of plants and attacks the corncobs, thus reducing production (Busato et al., 2005) depending on cultivar, phenological stage, production system, and planting site (Sarmento et al., 2002). Therefore, the search for new products with insecticidal action against this pest is of great importance. The potential of the products of plant metabolism to control insect pests has been evaluated (Tirelli et al., 2010; Alves et al., 2011; Santos et al., 2013; Alves et al., 2014). Secondary metabolites are involved in plant defense against herbivores and as such have the potential to be used in the control of insect infestations. Thus, phenolic compounds, which include tannins and flavonoids, are known to reduce growth and survival in insects (Schaller, 2008). These compounds, mainly tannins, reduce the palatability of plant tissues to insects because of their astringent taste, causing feeding inhibition, weight reduction, infertility, and biological and nutritional changes in insects (Schoonhoven et al., 2005). Furthermore, War et al. (2012) proposed a theory that these substances form complexes with digestive enzymes present in the intestines of herbivores, reducing the efficiency of protein digestion and ultimately slowing the growth.

In this context, the use of agro-industrial fruit residues is quite promising for the extraction of active ingredients that can be used in insect control. When the residues are discarded, secondary metabolites of great value for potential application as insecticides and/or agrochemicals are also eliminated. Acerola (*Malpighia emarginata* DC.) bagasse is one such example. It is produced after the fruit processing for juice production; according to Marques et al. (2013) acerola bagasse is rich in phenolic compounds, with

recorded content of 10.82 g 100 g^{-1} DM. Hence, the objective of the present study was to evaluate the effect of acetone extract of acerola bagasse flour against *S. frugiperda* and characterize the phenolic compounds and assess the prospect of acerola bagasse as an alternative natural insecticide, thus adding value to the fruit.

MATERIALS AND METHODS

Acerola bagasse flour and preparation of the extracts

Acerola (*Malpighia emarginata* DC.) (BRS 238 or Frutacor clon) bagasse was obtained from plants grown in the municipality of Perdões (21°05'27" S, 45°05'27" W; 848 m a.s.l.), Minas Gerais, Brazil; the local climate according to the Köppen system is classified as Cwa: mild and rainy summers with moderate temperatures, annual average temperature below 21 °C, average annual precipitation of 1529.7 mm, and 76% RH (Emater, 2002). Acerola fruits were used for pulp extraction, and the residual bagasse was provided in three batches by a fruit pulp plant firm located on Perdões. Acerola bagasse (4 kg) was frozen at -18 °C and lyophilized in glass containers protected from light for 7 d to obtain 450 g dry bagasse. After lyophilization, acerola bagasse was homogenized using mortar and pestle and then placed in a hermetically sealed flask, protected from light in a refrigerator at 4 °C.

To obtain the extract, 6 g acerola bagasse flour (ABF) was mixed with 60 mL acetone:water solution (7:3 v/v) (Agostini-Costa et al., 2003) in a round-bottom flask, in three replicates. This mixture was kept at room temperature for 2 h and then vortexed three times for 3 min. The solution was then centrifuged for 10 min, at 5000 g and filtered through glass wool. The residue was dissolved with 60 mL acetone:water solution (7:3 v/v) and the mixture was kept at room temperature for 2 h and finally vortexed three times for 3 min. The residue was discarded and the supernatants from the three extractions were combined and concentrated in a rotary evaporator (801, Fisatom, São Paulo, Brazil) until the complete evaporation of acetone. The material was then frozen, lyophilized, and weighed. Samples for chromatography were prepared by solubilizing 1 g lyophilized extract in 16 mL ultrapure water obtained from a Milli-Q system (EMD Millipore, Billerica, Massachusetts, USA).

Identification and quantification of phenolic compounds

The high performance liquid chromatography (HPLC) was performed using a Shimadzu UHPLC chromatograph (Shimadzu Corporation, Kyoto, Japan) equipped with two LC-20AT high-pressure pumps, an SPD-M20A UV-vis detector, a CTO-20AC oven, a CBM-20A interface, and an automatic injector with an SIL-20A auto sampler. Separations were performed using a Shim-pack VP-ODS-C18 (250 mm × 4.6 mm) column, connected to a Shim-pack Column Holder (10 mm × 4.6 mm) pre-column (Shimadzu).

The mobile phase consisted of the following solutions: 2% acetic acid in water (A) and methanol:water:acetic acid (70:28:2 v/v/v) (B). Analyses were performed for a total time of 65 min at 40 °C, flux of 1 mL min^{-1}, 280 nm wavelength, and injection volume of 20 μL in a gradient-type system (100% solvent A from 0.01 to 5 min; 70% solvent A from 5 to 25 min; 60% solvent A from 25 to 43 min; 55% solvent A from 43 to 50 min; and 0% solvent A for 10 min) until the end of the run. Solvent A was increased to 100%, seeking to maintain a balanced column.

The phenolic standards used were gallic acid, catechin, epigallocatechin gallate, epicatechin, syringic acid, *p*-coumaric acid, ferulic acid, salicylic acid, resveratrol, quercetin, malvidin chloride, pelargonidin chloride, and cyanidin chloride, all obtained from Sigma-Aldrich (St. Louis, Missouri, USA). The stock standard solutions were prepared in methanol (HPLC grade; Sigma-Aldrich) in the following concentration ranges: gallic acid (0.0753-7.5252 mg L^{-1}), catechin (0.1161-11.6120 mg L^{-1}), epigallocatechin gallate (0.1837-18.3360 mg L^{-1}), epicatechin (0.1161-11.6108 mg L^{-1}), syringic acid (0.0793-7.9268 mg L^{-1}), *p*-coumaric acid (0.0657-6.5664 mg L^{-1}), ferulic acid (0.0777-7.7672 mg L^{-1}), salicylic acid (0.0552-5.5248 mg L^{-1}), resveratrol (0.0913-9.1296 mg L^{-1}), quercetin (0.0004-0.0400 mg L^{-1}), malvidin chloride (0.1467-14.6700 mg L^{-1}), pelargonidin chloride (0.1227-12.2680 mg L^{-1}), and cyanidin chloride (0.1291-12.908 mg L^{-1}). Acetic acid and methanol (HPLC grade; Sigma-Aldrich) were used in the preparation of the mobile phase.

The ABF extract and the standards were filtered through a 0.45-μm nylon membrane (EMD Millipore) and directly injected into the chromatographic system, in three replicates. The phenolic compounds in the extract were identified by comparison with retention times of standards. Quantification was performed by the construction of analytical curves obtained by linear regression using Origin 6.1 computer software (OriginLab, Northampton, Massachusetts, USA) and considering the coefficient of determination (R^2) equal to 0.99.

Bioassay with *S. frugiperda* caterpillars

The bioassay was conducted in an acclimatized room at 25 ± 2 °C, 70 ± 10% RH, and a 12-h photophase, using insects reared by laboratory breeding and fed an artificial diet (Parra, 2007). For the bioassay, the lyophilized extract was solubilized in 30 mL distilled water and incorporated into 300 mL artificial diet at 250, 500, 1000, and 2000 mg L^{-1} diet. Once solidified, pieces from the diet were distributed in glass tubes (8 cm high × 2 cm wide).

The experimental design was completely randomized, with 60 replicates per treatment and the control treatment, which consisted of the artificial diet combined with only water

(30 mL). A 48-h-old *S. frugiperda* caterpillar, previously fed an artificial diet free of extract, was transferred into the experimental unit consisting of a glass tube (8 cm high × 2 cm wide) with a piece of diet (3 cm wide × 3 cm high) incorporated with the extract or the control.

Insect mortality was evaluated daily during the larval stage until the pupal stage. The following characteristics were also evaluated: duration of larval stage (d), duration of pre-pupal stage (d), pupal weight (g), duration of pupal stage (d), pupal survival (%) [pupal survival (%) = (number of insects that emerged/total number of pupae) × 100], and sex ratio of adults [sex ratio = ∑ females/(∑ females + males)]. The first 12 couples that emerged from each treatment were separately placed in PVC cages (10 cm high ×10 cm wide) and fed with 10% aqueous solution of honey. Adult longevity (d), oviposition period (d), number of egg masses total, and total number of eggs laid were evaluated daily.

Statistical analysis

Data on larval accumulated mortality and pre-pupal periods were subjected to one-way ANOVA and regression analysis was performed ($p < 0.05$) as a function of extract concentration using R software (R Development Core Team, 2014). Data on larval mortality over time were analyzed with the survival package in the Weibull model (Therneau, 2013). After the selection of the most appropriate mathematical model by residue analysis, a contrast analysis was performed to verify the similarity between the treatments and the formation of congener groups. Lethal concentration (LC_{20} and LC_{30}) and median lethal times (LT_{50}, time required to kill 50% of the insects) were estimated for each group formed by the Probit analysis (Ritz and Strebig, 2015).

Data on larval and pupal periods, pupal weight, pupal survival, adult longevity, oviposition period, the number of ovipositions, and number of eggs were subjected to one-way ANOVA using R (R Development Core Team, 2014).

The results of sex ratio were subjected to the chi-square statistical test (χ^2) at a significance level of $p < 0.05$.

RESULTS AND DISCUSSION

Each 100 g ABF yielded 30 g lyophilized extract (30% yield). The phenolic compounds identified in the ABF extract are presented in the Figure 1, whose areas of the peaks resulted in the following levels (mg 100 g^{-1} extract): gallic acid (14.85 ± 1.03), catechin (14.26 ± 0.95), epigallocatechin gallate (8.98 ± 0.95), *p*-coumaric acid (6.20 ± 0.34), salicylic acid (27.01 ± 0.38), and quercetin (0.80 ± 0.01). Anthocyanin compounds malvidin chloride, pelargonidin chloride, cyanidin chloride, epicatechin, syringic acid, ferulic acid, and resveratrol were not identified in the ABF extract. We observed several other peaks for which the substances were not identified.

Characterization of phenolic compounds in ABF has not been reported; however, a few studies report the identification of these compounds in agro-industrial residues of other fruits. Lafka et al. (2007) identified phenolic compounds such as gallic acid, catechin, epicatechin, caffeic acid, syringic acid, vanillic acid, and *p*-coumaric and *o*-coumaric acids in grape (*Vitis vinifera* L.) residues (skins and seeds) from the winemaking process. In contrast, Melo et al. (2011) studied grape and guava bagasse and found gallic acid, epicatechin, quercetin, isovanillic acid, *p*-coumaric acid, caffeic acid, and resveratrol. Alves et al. (2014) reported gallic acid, gallocatechin, catechin, epicatechin, ellagic acid, and salicylic acid in jabuticaba (*Myrciaria cauliflora* (Mart.) O. Berg) skin flour. These results show that agro-industrial residues from fruits are rich in bioactive substances, and they can be used by the agrochemical and pharmaceutical industries.

The content of each phenolic compound identified in the ABF extracts added to the artificial diet offered to *S. frugiperda* larvae is shown in Table 1. The salicylic acid and gallic were the majority, followed by catechin, epigallocatechin gallate, *p*-coumaric acid and quercetin among the identified phenolic substances. These data show the concentration of each phenolic compound in the diet that hindered the development of *S. frugiperda*.

Figure 1. Chromatogram of acerola bagasse flour extract with peaks identification. 1: Gallic acid (time = 6.126 min); 2: catechin (time = 10.152 min); 3: epigallocatechin gallate (time = 12.211 min); 4: *p*-coumaric acid (19.901 min); 5: salicylic acid (time = 32.547 min); and 6: quercetin (time = 51.187 min).

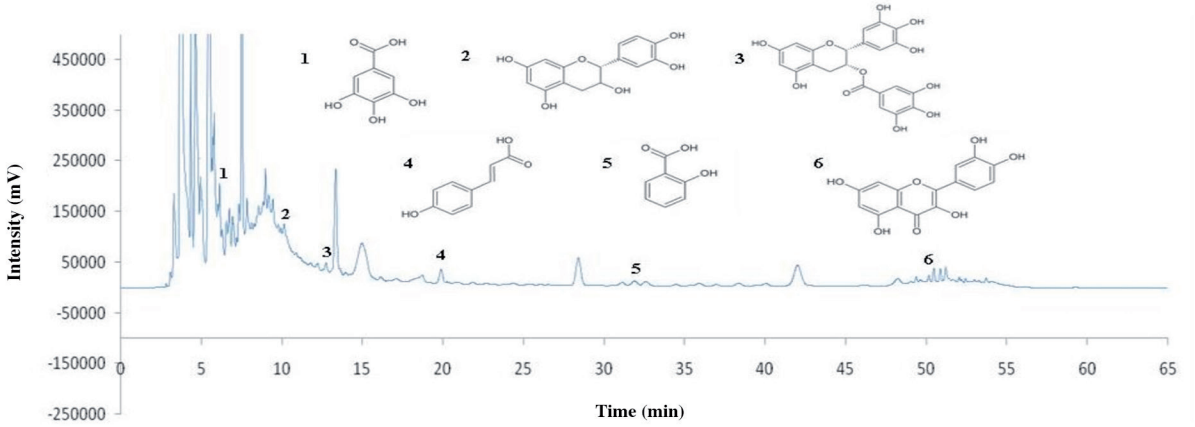


Table 1. Contents of phenolic compounds in acerola bagasse flour (ABF) extract supplemented to the artificial diet of *Spodoptera frugiperda* at different concentrations.

	ABF extract concentration (mg L^{-1} diet)			
Phenolic compound	250	500	1000	2000
Gallic acid	0.037	0.074	0.148	0.296
Catechin	0.036	0.072	0.144	0.288
Epigallocatechin gallate	0.022	0.044	0.088	0.176
p-Coumaric acid	0.016	0.032	0.064	0.128
Salicylic acid	0.068	0.136	0.272	0.544
Quercetin	0.002	0.004	0.008	0.016

Gallic acid in ester form is considered to be hydrolyzable tannin, while oligomers and polymers of catechin and epigallocatechin gallate, formed by polycondensation of two or more flavonoid units, are considered condensed tannins. These compounds are important biological components due to their strong interactions with metal ions and macromolecules such as polysaccharides. Additionally, they are able to form soluble complexes with alkaloids, gelatins, and several other proteins and to inhibit digestive enzymes (Simões et al., 2007), making this class of substances quite toxic to insects.

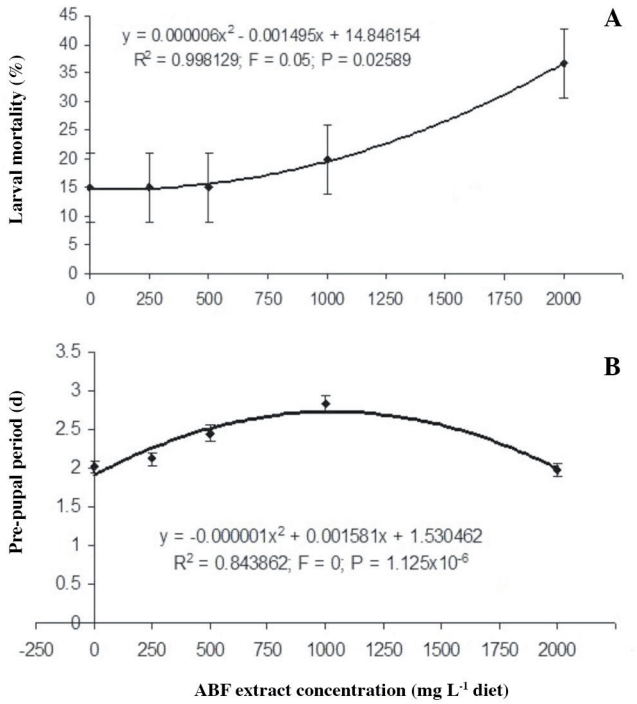
Flavonoids such as quercetin participate in important processes, including development and plant defense, inhibition of the regulation of digestive enzymes, strengthening of plant cell walls, and decrease in the palatability to herbivores (Schoonhoven et al., 2005; Chen, 2008; Filgueiras et al., 2011). Vandock et al. (2012) reported that flavonoids affect enzymes, preventing enzymatic reactions that are vital to the development, and are considered anti-nutritional and highly toxic to insects.

Salicylic and *p*-coumaric acids belong to the class of phenolic acids, which also includes derivatives from hydroxycinnamic and hydroxybenzoic acids. The relationship of these compounds and their role in the protection against herbivory is not known. Acids derived from benzoic acid may undergo substitutions in the meta position, yielding, for example, gallic acid (Mamede and Pastore, 2004), which can be found as hydrolyzable tannins, which are reported to reduce growth and survival of insects.

It was observed that the diet containing the extract at a concentration of 2000 mg L^{-1} caused an increase in accumulated mortality of *S. frugiperda*, while those fed on diets containing the extract at lower concentrations were less affected ($F = 0.05$; $P = 0.02589$) (Figure 2A). Regarding the pre-pupal stage, only the treatment containing 1000 mg L^{-1} of extract caused a lengthening of pre-pupal stage compared to the control ($F = 0$; $P = 1.125 \times 10^{-6}$) (Figure 2B).

The concentrations of ABF extract reduced the insect survival during the larval stage. The mean lethal time (LT_{50}) was determined only at the end of the evaluation period since this time was greater than 336 h (Figure 3). It was not possible to estimate the LC_{50} and LC_{90} because the confidence interval of these values exceeded the ABF extract concentrations employed in this work. Thus, the LC_{20} and LC_{30} of the ABF extract on the larvae of *S. frugiperda* were

Figure 2. Regression analysis of larval accumulated mortality (A) and duration of the pre-pupal period (B) of *Spodoptera frugiperda* larvae fed on artificial diet containing different concentrations of acerola bagasse flour (ABF) extract.

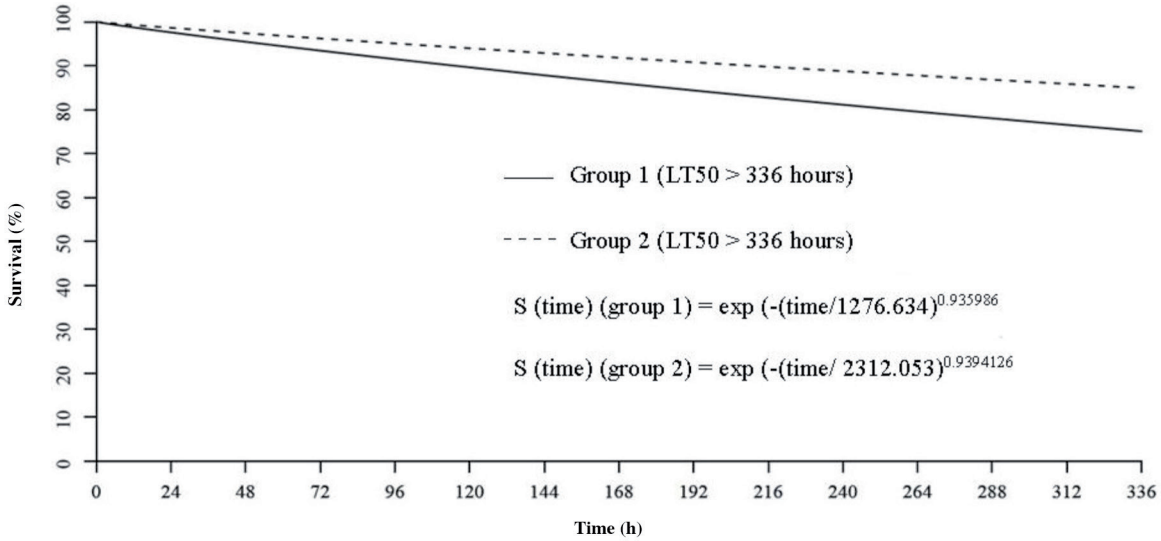


648.18 (95% CI = 229.57 - 996.78) and 1614.35 (95% CI = 477.35 - 2751.3) mg L^{-1}, respectively ($\chi^2 = 242.2$, $P = 0.4122$, $df = 238$), respectively.

The larval mortality of *S. frugiperda* reported in this study may be due to the phenolic compounds (tannins and flavonoids) characterized in ABF, which interfered with the feeding behavior and larval development of this insect. This hypothesis is supported by the fact that the phenolic compounds identified in ABF, such as gallic acid and catechin, affect growth and survival of herbivorous insects. Barbehenn and Constabel (2011) reported that, contrary to early theories, tannins have no direct effect on protein digestion in insect herbivores. Instead, they concluded that their toxicity is derived from reactive oxygen species such as semiquinone radicals and quinones, which are formed by oxidation of tannins in insects' high intestinal pH. Flavonoid quercetin, also identified in ABF, is known to decrease the palatability of plant tissues to herbivores and to inhibit enzymatic reactions that are vital for insect development (Filgueiras et al., 2011; Vandock et al., 2012).

Antifeedant and toxic effects of gallic acid and catechin have already been observed in species of the genus *Spodoptera* (Urrea-Bulla et al., 2004; Rani and Pratyusha, 2013). Mesbah et al. (2007) reported the toxic effect of quercetin on *Spodoptera littoralis* Boisduval (Lepidoptera: Noctuidae), and they attributed this toxicity to insect feeding inhibition, which prevented normal growth and development, causing pupal deformation as well as a reduction in oviposition.

Figure 3. Survival curves of *Spodoptera frugiperda* caterpillars, over time, fed on artificial diet containing acerola bagasse flour (ABF) extract. Group 1: 1000 mg ABF extract L^{-1} diet (n = 60) and 2000 mg ABF extract L^{-1} diet (n = 60). Group 2: control (30 mL water) (n = 60); 250 mg ABF extract L^{-1} diet (n = 60); 500 mg ABF extract L^{-1} diet (n = 60). The curve is represented with the formula S (time) = exp (-(time/δ)α) where δ is shape parameter and α is scale parameter.



Increased mortality at the larval stage was also reported by Rodríguez and Vendramim (1996), who observed that the pupal stage of *S. frugiperda*, compared to the larval stage, was less affected by extracts from various plants when they were incorporated into the artificial diet. Torrecillas and Vendramim (2001) found that changes in the development of *S. frugiperda* occurred only in the larval stage, probably due to higher metabolic activity during this phase.

According to Tirelli et al. (2010) and Jadhav et al. (2012), inhibition of larval growth and duration of the pre-pupal and pupal stages have already been related to the activity of phenolic compounds, especially tannins and flavonoids, which reduce the nutritional value but create the antifeedant effect in food. Alves et al. (2014) studied the effects of acetone extract from jabuticaba (*Myrciaria cauliflora* [Mart.] O. Berg) skin flour on the development of *S. frugiperda* and found that the reduction in their growth was proportional to the concentration and quantity of ingested phenolic compounds.

Tannin activity is not always related to its concentration. Heil et al. (2002) found that in some plant species, the increase in tannin concentration did not lower herbivory. However, a positive correlation between tannin content and reduction in herbivory rate was observed for *Prosopis juliflora* (Sw.) DC. (Fabaceae), *Acacia farnesiana* (L.) Willd. (Fabaceae), and *Leucaena leucocephala* (Lam.) de Wit (Fabaceae). Therefore, authors concluded that the antifeedant effect of tannins cannot always be correlated with the increase in their concentration. These results are consistent with those obtained in the present study, in which it was found that treatment with 1000 mg L^{-1}, although with lower tannin content than the treatment with 2000 mg L^{-1}, had prolonged the most the pre-pupal characteristics of *S. frugiperda* (Figure 2B).

The ABF extract at the evaluated concentrations did not negatively affect the larval period, pupal weight, pupal period, pupal survival, oviposition period, total number of egg masses, number of eggs, male and female longevity (Table 2), and sex ratio (Table 3).

Table 2. Effect of acetone extract from acerola bagasse flour (ABF) at different concentrations on larval period, pupal weight, pupal period, pupal survival, oviposition period, number of egg masses, number of eggs, and male and female longevity of *Spodoptera frugiperda*.

	ABF extract concentration (mg L^{-1} diet)						
	0	250	500	1000	2000	P	F
Larval period, d†	14.00 ± 4.31	13.60 ± 4.24	13.10 ± 3.20	12.80 ± 4.01	12.75 ± 3.93	0.3257	1.1664
Pupal weight, g†	0.219 ± 0.02	0.215 ± 0.03	0.223 ± 0.03	0.218 ± 0.02	0.222 ± 0.02	0.5564	0.7537
Pupal period, d†	7.02 ± 4.03	5.98 ± 4.22	7.70 ± 3.76	6.83 ± 3.88	6.20 ± 4.48	0.1602	1.6561
Pupal survival, %†	75.41 ± 0.43	75.41 ± 0.43	80.00 ± 0.40	65.00 ± 0.48	63.33 ± 0.49	0.0574	2.3154
Oviposition period, d†	9.93 ± 3.91	11.00 ± 3.24	11.13 ± 2.64	10.70 ± 4.45	10.42 ± 4.90	0.9296	0.2139
Number of egg masses†	9.64 ± 3.73	10.33 ± 2.83	10.40 ± 2.23	10.60 ± 4.65	9.58 ± 4.17	0.9344	0.2053
Number of eggs†	2752.93 ± 1197.45	2533.78 ± 733.38	3158.06 ± 1672.09	2215.10 ± 880.76	2504.27 ± 1094.99	0.3891	1.0518
Male longevity, d†	13.64 ± 1.91	14.00 ± 2.06	11.66 ± 2.74	13.55 ± 1.44	12.67 ± 3.01	2.0805	0.0955
Female longevity, d†	14.93 ± 4.62	14.56 ± 4.42	16.27 ± 4.62	15.64 ± 3.96	15.17 ± 5.10	0.9164	0.2368

Data are mean (n = 60) ± standard deviation.
†Nonsignificant difference in means by the one-way ANOVA at 5% probability.

Table 3. Sex ratio in *Spodoptera frugiperda* developed from caterpillars fed artificial diet supplemented with different concentrations of acerola bagasse flour (ABF) extract.

ABF extract concentration (mg L^{-1} diet)	n	Sex ratio†††
0	43	0.44
250	36	0.47
500	49	0.41
1000	40	0.58
2000	38	0.39
P†		0.496
$\chi 2$††		3.38

†*P* value of the chi-square test. ††Chi-square value. †††Nonsignificant difference in means by the one-way ANOVA at 5% probability.

When added to the artificial diet, the phenolic compounds present in ABF caused an increase in larval mortality and lengthening of the pre-pupal stage; however, they did not have a negative effect on other biological characteristics evaluated for *S. frugiperda*. It is possible that the results will be more pronounced if the phenolic substances contained in the ABF extract are used in other concentration ranges.

Given that there are no available research data on the use of ABF in pest control, the results obtained in this study complement those of Santos et al. (2013) and Alves et al. (2014), who used natural products such as cassava *Manihot esculenta* Crantz leaf powder and jabuticaba skin flour, respectively, to control *S. frugiperda* caterpillars.

CONCLUSIONS

Acerola bagasse flour (ABF) acetone extract, in which the phenolic compounds such as gallic acid, catechin, epigallocatechin gallate, *p*-coumaric acid, salicylic acid, and quercetin were identified, decreased the survival of *Spodoptera frugiperda* under laboratory conditions. Future studies should examine their potential use in field conditions and investigate the effect of isolated phenolic compounds identified in the ABF extract.

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