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ORIGINAL PAPER

Kaolin nano-powder effect on insect attachment ability

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Abstract

The present study investigates under controlled conditions the effect of kaolin particle film on reduction of insect attachment ability. Two economically important polyphagous insect pests characterized by different attachment devices were tested, the Southern green stink bug *Nezara viridula* (Heteroptera: Pentatomidae) and the Mediterranean fruit fly *Ceratitis capitata* (Diptera: Tephritidae). We performed traction force experiments with females pulling on treated (covered with kaolin particle film) and untreated (control) natural (leaf surfaces with different morphological traits) and artificial (hydrophilic and hydrophobic glass) surfaces. The data demonstrated that insect adhesion is heavily affected by kaolin particle film in both tested species. The degree of reduction of insect adhesion to the treated substrates compared with the untreated ones differed according to the kind of treated substrate owing to its initial wettability and morphology (presence of trichomes). To unravel the insect adhesion reduction mechanism of kaolin particle film, we evaluated the safety factor for females before and after walking on treated surfaces and analyzed under cryo-SEM the tarsal attachment devices of *N. viridula* and *C. capitata* after walking on treated surfaces. We observed contamination by the kaolin nanoflakes in both the smooth pads of the bug and the hairy pads of the fly. The present study can help to better understand the mechanism of action of kaolin particle film and can contribute to develop future physical control barriers against pest insects, particularly relevant owing to the need to reduce the negative impacts of pesticides on environment and human health.

Keywords Particle film · Natural product · Bioadhesion · Friction · Southern green stink bug · Mediterranean fruit fly

Key message

- The study investigates under controlled conditions the effect of kaolin particle film in reducing attachment ability of *Nezara viridula* (Heteroptera: Pentatomidae) and *Ceratitis capitata* (Diptera: Tephritidae).
- Kaolin particle film strongly reduces insect attachment ability with a degree of reduction depending on the initial wettability and morphology of treated surfaces.
- Figuring out the biomechanics of the interaction between the particle film and insect attachment devices can help to develop non-chemical methods for pest control.

Introduction

Control of insects with inert dusts represents an important economically sustainable and environmentally safe alternative to the use of synthetic pesticides (Glenn et al. 1999; Glenn and Puterka 2005). This technology (particle film technology) is based on coating plants in orchards and field crops with chemically inert mineral particles producing a porous film which does not interfere with gas exchange between the leaf and the atmosphere and can be easily removed from harvest (see review in Glenn and Puterka 2005). Among the different minerals investigated, kaolin, a white, non-swelling, non-abrasive, fine-grained,

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nanoplate-like aluminosilicate mineral ($Al_4Si_4O_{10}(OH)_8$) that easily disperses in water and is chemically inert over a wide pH range, attracted much attention (Glenn and Puterka 2005). Kaolin powder, suspended in water and applied with traditional sprayers on fruits and leaves, creates a continuous coating, once the water evaporates. It is beneficial for plants because it (1) can improve plant health protecting it from weather conditions like heat stress and sunburn (Glenn et al. 2002; Jifon and Syvertsen 2003; Melgarejo et al. 2004; Gindaba and Wand 2007) and (2) can protect the plant from different insect pests. In particular, kaolin particle films revealed to be effective against many arthropod pests affecting crops, including hemipterans, coleopterans, lepidopterans, dipterans (Puterka et al. 2000; Saour and Makee 2004; Daniel et al. 2005; Glenn and Puterka 2005; Saour 2005; Bostanian and Racette 2008; Pascual et al. 2009; D'Aquino et al. 2011; Lo Verde et al. 2011; Nateghi et al. 2013; Silva and Ramalho 2013; Tacoli et al. 2017a, b, 2019). Laboratory and field investigations reported oviposition and feeding deterrence, with a consequent reduced survival rate of adults or young stages and a longer developmental time (Knight et al. 2000; Unruh et al. 2000; Cottrell et al. 2002; Puterka et al. 2005; Barker et al. 2006; Lapointe et al. 2006; Bostanian and Racette 2008; Tacoli et al. 2017a, b, 2019) and reduced mating success of adults (Knight et al. 2000; Puterka et al. 2005).

The main basic action of kaolin particle film against pest insects is due to its interference during the host plant location and acceptance process by the insect. Indeed, plants covered with white particle films are altered from the visual and tactile point of view and also the taste and smell of the host plant can change inducing repellence from treated foliage and a consequent reduced feeding and oviposition (Glenn and Puterka 2005). In a series of laboratory investigations testing the effect of kaolin particle film on the biology and behavior of *Cacopsylla pyri* (L.) (Hemiptera: Psyllidae) (Puterka et al. 2005), a reduction in the insect ability to grasp the plant has been highlighted as a simply "fall-off" the leaves of the host plant covered by kaolin powder. A reduction in the walking speed in the larvae of *Cydia pomonella* (L.) (Lepidoptera: Tortricidae) on kaolin film covered plants has been reported (Unruh et al. 2000). Puterka et al. (2005) hypothesized that the particle attachment to insects owing to the loosely bound nature of kaolin film could differ depending on arthropod species and their adaptations to grasp the plant surfaces.

The present study investigates in detail under controlled conditions the effect of kaolin particle film on reduction of insect attachment ability. We performed traction force experiments on treated (covered with kaolin particle film) and untreated (control) natural (leaf surfaces with different morphological traits) and artificial (hydrophilic and hydrophobic glass) surfaces. In order to unravel the mechanism of action of the kaolin particle film in impeding insect adhesion, we studied the initial wettability of the tested surfaces through measurements of water contact angle prior to the treatment, characterized the roughness of treated and untreated glass substrates using the white light interferometer and examined the natural surfaces and treated hydrophilic glass in cryo-SEM. To estimate the possible contamination effect of kaolin particles, we evaluated the attachment ability and observed the tarsal attachment devices of insects after walking on treated surfaces.

We tested two economically important polyphagous insect pests belonging to two different orders, represented by the southern green stink bug *Nezara viridula* L. (Heteroptera: Pentatomidae) and the Mediterranean fruit fly *Ceratitis capitata* Wiedemann (Diptera: Tephritidae). Among the various types of leg attachment devices, which insects evolved to adhere to the surfaces [see review by Gorb (2001)], these two species are characterized by different tarsal attachment structures, whose morphology and attachment ability have been studied in detail in ultrastructural and behavioral investigations, on natural and artificial substrates (Rebora et al. 2018; Salerno et al. 2017, 2018a, b; Salerno et al. 2019, submitted). The attachment structures of *N. viridula* comprise two sclerotized claws, a pair of smooth flexible pulvilli and a hairy adhesive pad located at the ventral side of the basitarsus (Rebora et al. 2018; Salerno et al. 2017), while *C. capitata* has claws and hairy pulvilli (Salerno et al. 2019 submitted).

Materials and methods

Insects

Individuals of *N. viridula* were collected in the field close to Bastia Umbra (Perugia, Umbria region, Italy) in June 2018 and reared in a controlled climate chamber (14:10 light–dark rhythm, at a temperature of 25 ± 1 °C and a relative humidity of 70 ± 10%) inside transparent plastic food containers (300 mm × 195 mm × 125 mm) with mesh-covered holes with a diameter of 5 cm. Sunflower seeds (*Helianthus annus* L.) and French beans (*Phaseolus vulgaris* L.) were used to feed the insects.

C. capitata adults were obtained from pupae reared in the laboratory of the Dipartimento di Scienze delle Produzioni Agroalimentari e dell'Ambiente (University of Florence, Italy) (Granchietti et al. 2012) and kept in a controlled condition chamber (14 h photophase, temperature of 25 ± 1 °C; RH of 60 ± 10%) inside net cages (300 mm × 300 mm × 300 mm). Adult insects were provided with water and a solid diet consisting of sucrose, yeast, and enzymatic yeast hydrolysate in a 6:2:1 ratio (Cavalloro and Girolami 1969).

Only adult females of both species have been tested.

Plants

Two plant species having either visually smooth, shiny leaves [cherry laurel *Prunus laurocerasus* L. 'caucasica' (Rosaceae)] or conspicuous hairy leaves [common sunflower *Helianthus annuus* L. (Asteraceae)] were used in the study. Cherry laurel is an evergreen shrub with leathery shiny leaves. It originated from Asia Minor and has become naturalized widely. Common sunflower is an annual herb with a single large inflorescence atop an unbranched stem. It is grown as a crop and as an ornamental plant in domestic gardens. The plant originated from North and Middle America, where it was first domesticated.

In both plant species, only the adaxial leaf side was tested in this study.

Cryo-scanning electron microscopy (cryo-SEM)

The shock-frozen samples of the leaf surfaces, of treated (with kaolin particle film) hydrophilic glass and of the insect tarsi (after walking on treated hydrophilic glass) were studied in a scanning electron microscope (SEM) Hitachi S-4800 (Hitachi High-Technologies Corp., Tokyo, Japan) equipped with a Gatan ALTO 2500 cryo preparation system (Gatan Inc., Abingdon, UK). For details of sample preparation and mounting for cryo-SEM, see Gorb and Gorb (2009). Whole mounts of leaf surface pieces, insect tarsi and glass were sputter-coated in frozen conditions with gold–palladium (thickness 10 nm) and examined at 3 kV acceleration voltage and temperature of − 120 °C at the cryo stage within the microscope.

Force measurements

The experiments were performed using a traction force experimental setup. Prior to the force measurements, adults of *N. viridula* and *C. capitata* were weighed on a microbalance (Mettler Toledo AG 204 Delta Range, Greifensee, Switzerland). Experimental insects were anaesthetized with carbon dioxide for 60 s and made incapable of flying by carefully gluing their wings together using a small droplet of melted wax. One end of about 15-cm-long human hair was fixed on the insect thorax with a droplet of molten wax (Fig. 1a, inset). Before starting experiments, insects were left to recover for 30 min. All the experiments were performed during the daytime at 25 ± 1 °C temperature and 60 ± 10% relative humidity.

The traction force experimental setup consisted of a force sensor FORT-10 (10 g capacity; World Precision Instruments Inc., Sarasota, FL, USA) connected to a force transducer MP 100 (Biopac Systems Ltd, Goleta, CA, USA) (Gorb et al. 2010). Data were recorded using AcqKnowledge 3.7.0 software (Biopac Systems Ltd, Goleta, CA, USA). The insect was attached to the force sensor by means of the hair glued to its thorax and was allowed to move on the substrate in a direction perpendicular to the force sensor. The force

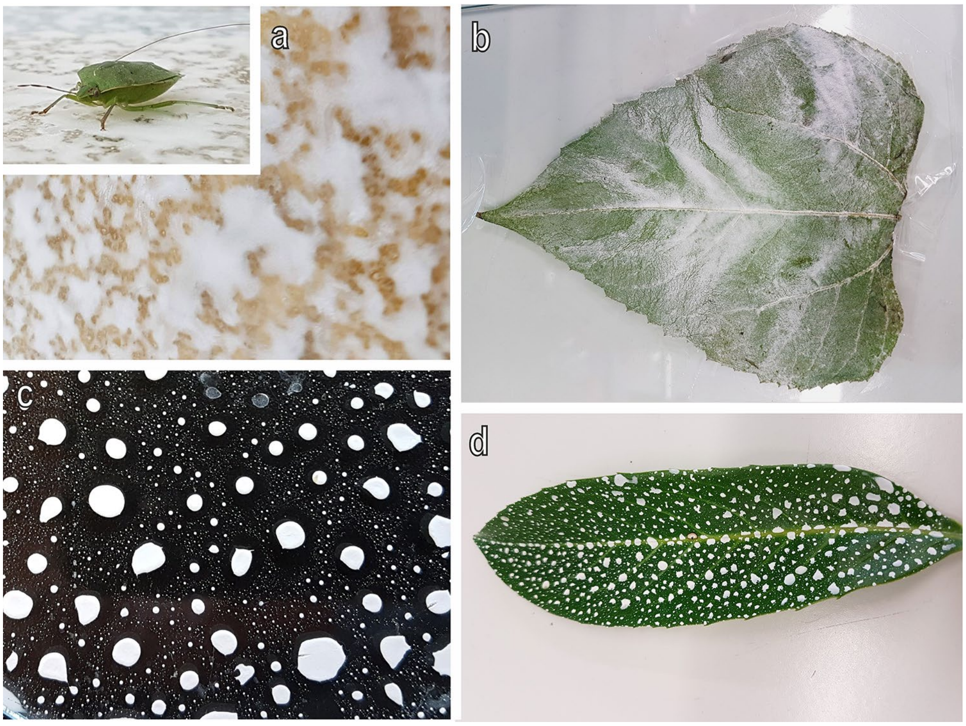

Fig. 1 Treated surfaces: hydrophilic glass (**a**), *Helianthus annuus* (**b**), hydrophobic glass (**c**), and *Prunus laurocerasus* (**d**). Inset in **a** shows a female of *Nezara viridula* during a traction force experiment. Note the different distribution (see Table 3) of kaolin particle film (dry droplets) on the tested surfaces

generated by the insect walking horizontally on the test substrates was measured. Force–time curves were used to evaluate the maximal pulling force produced by walking insects (friction). A treated or untreated leaf of the plant to be tested was cut and attached with double-sided tape to a horizontal glass slide with its adaxial side upward. The leaf surfaces were changed after every three tested insects to avoid plant dehydration. Each insect walked from the proximal to the distal portion of the tested leaf.

N. viridula adult females were tested according to the following sequences:

- on untreated hydrophilic glass, treated hydrophilic glass, untreated hydrophilic glass;
- on untreated hydrophilic glass, untreated hydrophobic glass, treated hydrophobic glass, untreated hydrophilic glass;
- on untreated hydrophilic glass, untreated *P. laurocerasus* leaf, treated *P. laurocerasus* leaf, untreated hydrophilic glass;
- on untreated hydrophilic glass, untreated *H. annuus* leaf, treated *H. annuus* leaf, untreated hydrophilic glass.

Forty *N. viridula* adult females (10 for each sequence) were tested.

C. capitata adult females were tested according to the following sequence:

- on untreated hydrophilic glass, treated hydrophilic glass, untreated hydrophilic glass.

Ten *C. capitata* adult females were tested.

Force–time curves were used to evaluate the maximal friction force produced by pulling insects on the different test surfaces for each individual run.

Substrate preparation and characterization

The natural and artificial treated surfaces have been prepared by spraying them with a water suspension of kaolin powder commonly available in Italy (“I consigli dell’esperto”, Civitavecchia, Italy) at the rate of 4% (W/V). The suspension was applied on horizontal surfaces with a hand-sprayer until runoff. After spraying, the surfaces were left to dry at room temperature. The wettability of untreated surfaces (hydrophilic glass, hydrophobic glass and leaf surfaces) and of the hydrophilic glass covered by kaolin particle film was characterized by measuring the contact angles of water (aqua Millipore, droplet size = 1 µl, sessile drop method) using a high-speed optical contact angle measuring instrument OCAH 200 (Dataphysics Instruments GmbH, Filderstadt, Germany). Ten measurements ($n = 10$) were taken for each substrate.

The roughness of treated and untreated hydrophilic glass substrates has been characterized (as reported in Salerno et al. 2018b) using the white light interferometer NewView 6000 (Zygo Middlefield, CT, USA) with the objectives 5 × and 50 × (window size 1400 × 1050 and 140 × 105 µm, respectively). Five individual measurements ($n = 5$) at different sites were taken for each substrate. The distribution of kaolin particle film on treated surfaces was evaluated using the open source image processing program ImageJ (Schneider et al. 2012). The evaluated parameter was the coverage area (%).

Statistical analysis

In the traction force experiments testing the attachment ability of *N. viridula* and *C. capitata* females to hydrophilic glass covered by kaolin particle film, the safety factors (friction force divided by the insect weight) were analyzed with two-way repeated measures ANOVA, considering the treatment of the surface and the insect species as factors. In the traction force experiments, testing the attachment ability of *N. viridula* females to hydrophilic and hydrophobic glass and to the adaxial leaf sides of *P. laurocerasus* and of *H. annuus*, the safety factors were analyzed with two-way repeated measures ANOVA, considering the treatment of the surface and the surfaces as factors. For the surface factor, Tukey’s HSD post hoc test for multiple comparisons between means was used. The safety factor obtained on the treated (covered by kaolin particle film) surfaces was compared with that obtained on the corresponding untreated surface using the Student’s *t* test for dependent samples. The percentage of reduction of the safety factor obtained on treated hydrophilic glass in relation to the untreated hydrophilic glass was analyzed using the Student’s *t* test for independent samples. One-way ANOVA followed by Tukey’s HSD post hoc test for multiple comparisons between means was used for comparing the percentage of reduction of the safety factor obtained on different surfaces (Statistica 6.0, Statsoft Inc. 2001). Before the analysis, all the data were subjected to Box–Cox transformations, in order to reduce data heteroscedasticity (Sokal and Rohlf 1998).

Results

Characterization of tested surfaces

Untreated surfaces

The tested surfaces were represented by artificial (hydrophilic and hydrophobic glass) and natural surfaces (adaxial side of *H. annuus* and *P. laurocerasus* leaves). The hydrophilic glass shows a water contact angle of 61.63° ± 7.86°

Table 1 Contact angles of water on tested surfaces

Tested surfaces	Contact angle (°)
Hydrophilic glass	61.63 ± 7.86
Hydrophobic glass	92.94 ± 1.07
Prunus laurocerasus (adaxial leaf surf.)	104.76 ± 2.20
Helianthus annuus (adaxial leaf surf.)	36.00 ± 7.30
Hydrophilic glass treated with kaolin	10.04 ± 1.65

Data are presented as mean ± SEM

(Table 1) and roughness at a sub-nanometer scale (Fig. 2a, b, Table 2). Hydrophobic glass is characterized by a contact angle of 92.94° ± 1.07° (Table 1).

Plane adaxial leaf surface of *P. laurocerasus* is slightly uneven at both small and high magnifications (Fig. 3a–c). Such surface appearance is caused by the 2D epicuticular wax film/layer with very flat, plate-like surface irregularities having various irregular shapes (Fig. 3c). Also solitary wax projections (3D wax), membranous platelets, protruding perpendicularly from the surface and differing in both shape and size (length: 3.90 ± 1.92 µm, $n = 25$; thickness: 0.13 ± 0.04 µm, $n = 5$), are very sparsely scattered over the adaxial leaf surface (Fig. 3b,c).

The adaxial side of *P. laurocerasus* leaf has a contact angle of 104.76° ± 2.20° (Table 1).

A dense pubescence of the adaxial leaf surface of *H. annuus* is composed by three types of trichomes: (1) non-glandular ones dispersed regularly over the surface; (2) linear glandular ones occurring more densely on leaf veins and more sparsely at intercostals regions, and (3) capitate glandular ones associated with only intercostals regions [classification according to Aschenbrenner et al. (2013)] (Fig. 3d, e). The total trichome density is ca. 25 mm^{-2}, with the non-glandular, linear glandular, and capitate glandular types accounting for ca. 46, 50, and 4% of trichomes, respectively. The non-glandular trichomes are highly variable in length (ca. 0.2–0.6 mm; 0.27 ± 0.11 mm, $n = 15$), perpendicular or slightly inclined, uniseriate, tapered cone-shaped, with sharp tip and multi-cellular base (Fig. 3e, f). The trichome surface is distinctly sculptured (nodose) in both basal and shaft parts (Fig. 3f). The linear glandular trichomes are ca. 0.5 mm long (0.10 ± 0.01 mm, $n = 15$), curved, uniseriate (6–11 cells), finger-shaped, with unicellular base (Fig. 3e, f). Here, the

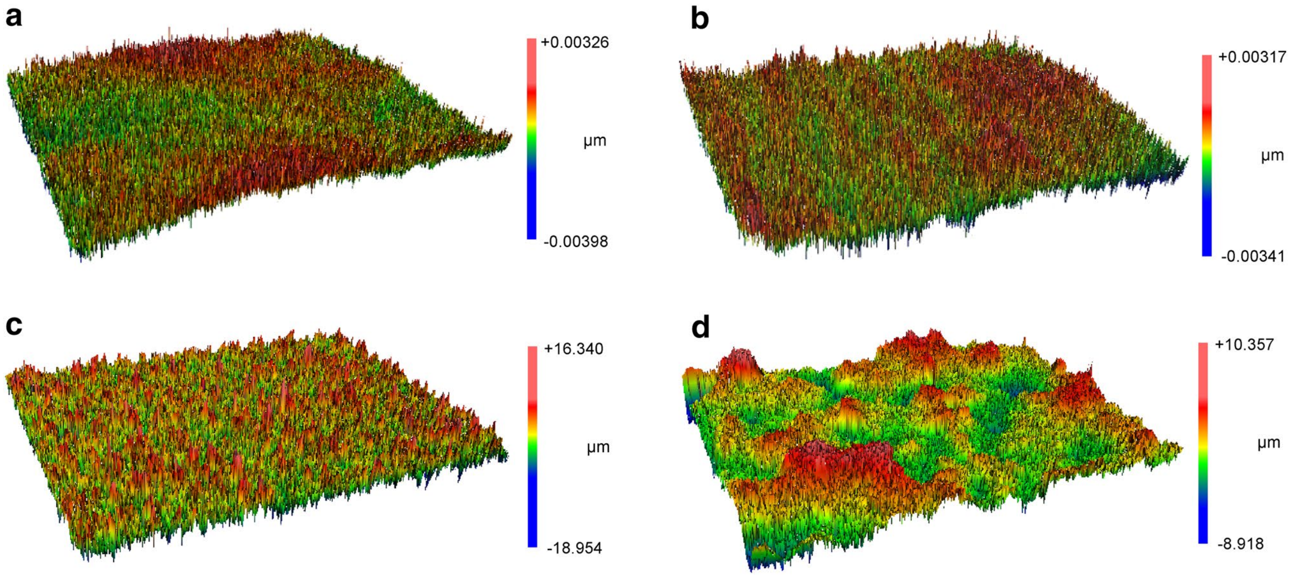

Fig. 2 3D surface profiles of untreated (**a**, **b**) and treated (with kaolin particle film) hydrophilic glass (**c**, **d**). The long side length equals to 1400 µm for (**a**, **c**) and 140 µm for (**b**, **d**)

Table 2 Roughness characteristics of untreated and treated (with kaolin particle film) hydrophilic glass

Surfaces	R_a (5×) (µm)	R_a (50×) (µm)	RMS (5×) (µm)	RMS (50×) (µm)
Hydrophylic glass	$0.551 \pm 0.029 \times 10^{-3}$	$0.516 \pm 0.011 \times 10^{-3}$	$0.700 \pm 0.040 \times 10^{-3}$	$0.653 \pm 0.014 \times 10^{-3}$
Hydrophilic glass treated with kaolin	2.004 ± 0.078	1.537 ± 0.089	2.575 ± 0.102	1.929 ± 0.113

Data are presented as mean ± SEM

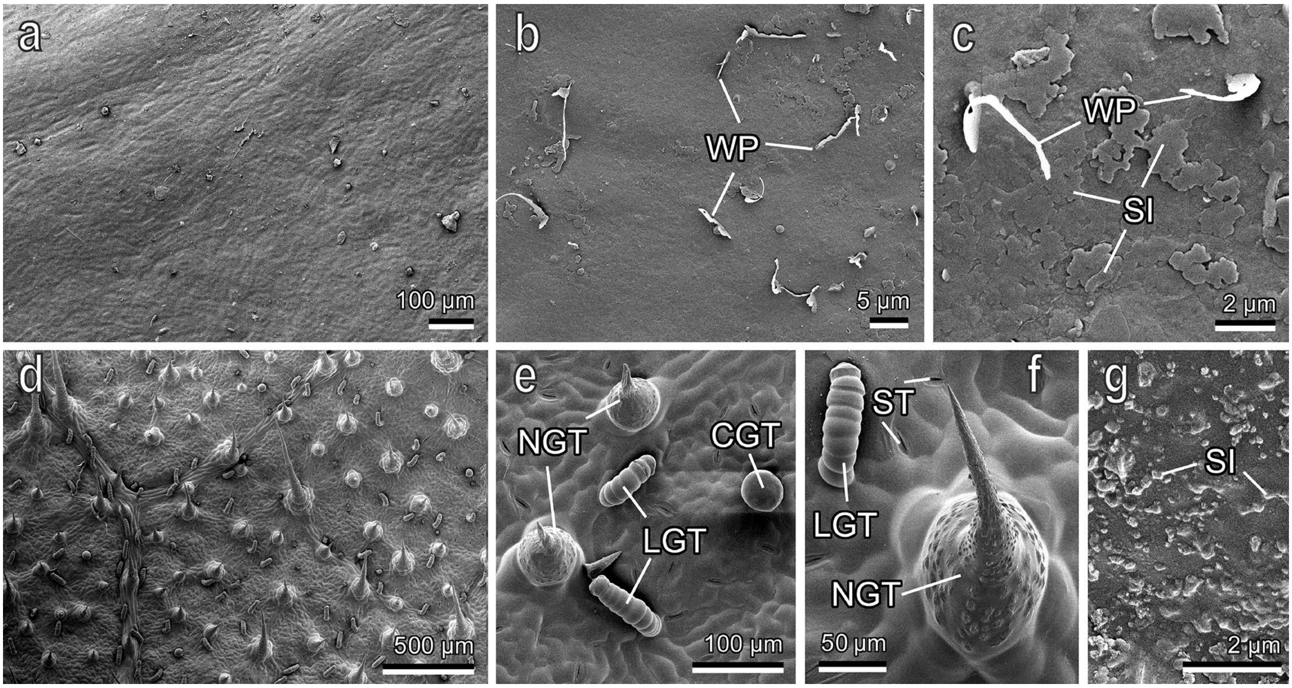

Fig. 3 The adaxial leaf surface of *Prunus laurocerasus* 'caucasica' (**a**–**c**) and *Helianthus annuus* (**d**–**g**) in cryo-SEM. *CGT* capitate glandular trichome; *LGT* linear glandular trichome; *NGT* non-glandular trichome; *SI* surface irregularity; *ST* stoma; *WP* wax platelet

trichome surface is smooth (Fig. 3f). Each capitate glandular trichome bears a spherical, unicellular glandular head (diameter: 48.86 ± 2.54 µm, $n = 5$) (Fig. 3e). Also stomata are regularly scattered among the trichomes (Fig. 3f). The cuticle between trichomes is covered by the 2D epicuticular wax film/layer with numerous densely, but non-uniformly, distributed very small (< 0.5 µm) granule-like surface asperities (Fig. 3g). The latter, although often being half-spherical or half-ellipsoid, vary greatly in shape and size.

The adaxial side of *H. annuus* leaf has a water contact angle of 36.00° ± 7.30° (Table 1). During contact angle measurements, the water drops quickly spread over the surface and become partly sucked by the hairy coverage.

Treated surfaces

The kaolin particle film on the hydrophilic glass (Fig. 1a) appears as a film of irregularly shaped dry droplets close to each other, resulting in a percentage of covered area of about 70% of the glass surface (Table 3). The kaolin particle film on the hydrophobic glass (Fig. 1c) appears as a film of round-shaped dry droplets (most of them are very small and well separated one from the other, few are large), resulting in a percentage of covered area of about 18% of the glass surface (Table 3). The kaolin particle film on the adaxial side of *H. annuus* leaf (Fig. 1b) forms wide covered areas (percentage of covered area of about 65% of the leaf surface, Table 3), whereas in the case of *P. laurocerasus* (Fig. 1d), likewise on hydrophobic glass, it forms round-shaped dry droplets, resulting in a percentage of covered area of about 18% of the leaf surface (Table 3).

Table 3 Distribution of kaolin particle film on treated surfaces

Tested surfaces	Area coverage (%)
Hydrophilic glass	70.82 ± 3.03
Hydrophobic glass	17.96 ± 0.92
Prunus laurocerasus (adaxial leaf surf.)	21.03 ± 0.42
Helianthus annuus (adaxial leaf surf.)	65.61 ± 8.85

Data are presented as mean ± SEM

The area (hydrophilic glass) covered by the kaolin particle film visualized with cryo-SEM appears as a uniform film (Fig. 4a, b), which at high magnification reveals to be constituted of many layers of hexagonal- or pseudo-hexagonal-shaped, horizontally placed plates (Fig. 4c, d). The most superficial plates measure 0.05 ± 0.01 µm^2 ($n = 42$) and lay on the underlying layers constituted of larger plates with an area of 0.34 ± 0.10 µm^2 ($n = 9$).

The kaolin particle film (applied on hydrophilic glass) is characterized by a contact angle of 10.04° ± 1.65° (Table 1) with water drops spreading very quickly over the surface and completely absorbed in a short time and by a roughness of about 2 µm (Fig. 2c, d, Table 2).

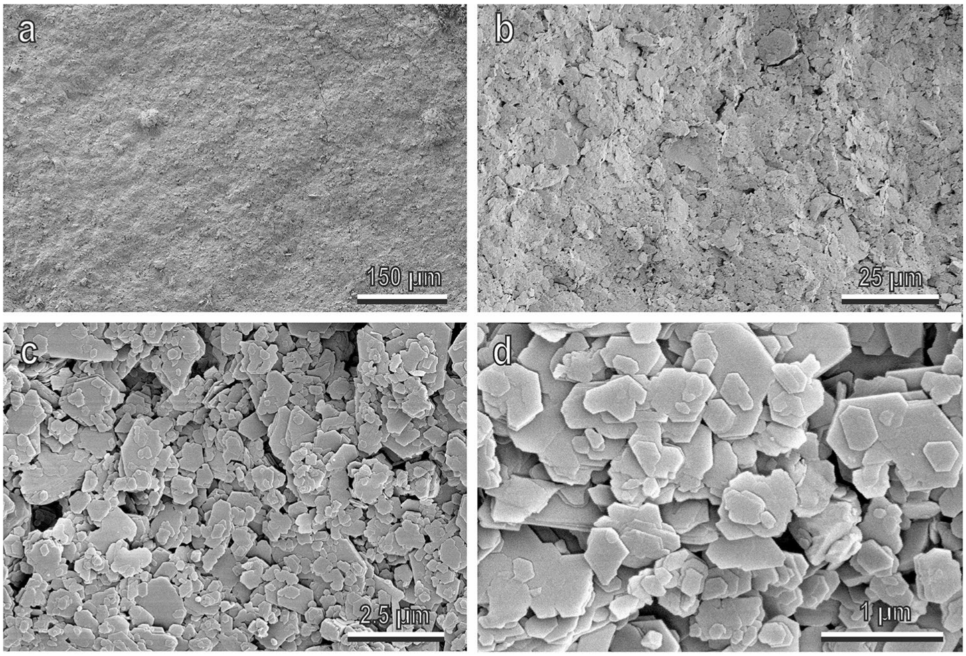

Fig. 4 Kaolin particle film visualized with cryo-SEM at progressively higher magnifications (**a**–**d**). Note in **c** and **d** the layers of hexagonal- or pseudo-hexagonal-shaped, horizontally placed plates. The most superficial plates are smaller than those located in the underlying layers

Attachment ability of *Nezara viridula* and *Ceratitis capitata* females to untreated and treated surfaces

In the traction force experiments testing the attachment ability of *N. viridula* and *C. capitata* females to hydrophilic glass covered by kaolin particle film, the safety factors (friction force divided by the insect weight) generated by females varied significantly depending on insect species and treatments (Fig. 5, table inset). Also, the interaction between treatments and insect species was significant. In particular, the safety factor was significantly higher in *C capitata* than in *N. viridula* and on the untreated than on the treated surfaces. This last effect was significant for both species (*N. viridula*: $t = 11.51$, $df = 9$, $p < 0.0001$; *C. capitata*: $t = 10.38$, $df = 9$, $p < 0.0001$), but, in agreement with the significance of the interaction, the percentage of reduction of the safety factor due to the kaolin coverage was significantly different being higher in *N. viridula* than in *C. capitata* ($t = -5.86$, $df = 18$, $p < 0.0001$) (Fig. 5).

In the traction force experiments, testing the attachment ability of *N. viridula* females to hydrophilic and hydrophobic glass and to the adaxial leaf sides of *P. laurocerasus* and of *H. annuus*, the safety factors varied significantly depending on surfaces and treatments. Also, the interaction between treatments of the surfaces and the surfaces varied significantly (Fig. 6, table inset). In particular, the safety factor was significantly higher on *H. annuus* than on the other surfaces that did not differ among them (Fig. 6). The significance of the interaction between treatment and surface indicates that the extent of the kaolin effect was influenced by surfaces as shown by the fact that the differences between untreated and treated surfaces were significant on hydrophilic glass ($t = 11.51$, $df = 9$, $p < 0.0001$), hydrophobic glass ($t = 3.72$, $df = 9$, $p = 0.0048$) and *P. laurocerasus* ($t = 3.30$, $df = 9$, $p = 0.0092$), but not on *H. annuus* ($t = 2.14$, $df = 9$, $p = 0.0605$) (Fig. 6).

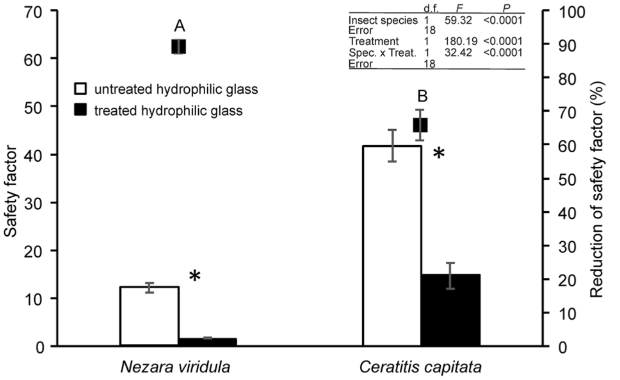

Fig. 5 Safety factor (friction force divided by the insect weight) obtained in friction force experiments with *Nezara viridula* and *Ceratitis capitata* females on treated (covered by kaolin particle film) and untreated hydrophilic glass. Columns indicate the means ± SEM. Asterisk (*) indicates significant difference at $p < 0.05$, Student's *t* test for dependent samples. Black squares indicate the percentage of reduction of the safety factor on hydrophilic glass covered by kaolin particle film in relation to the untreated hydrophilic glass. Squares with different letters are significantly different at $p < 0.05$, Student's *t* test for independent samples. Table inset shows the statistical parameters of two-way repeated measures ANOVA

The percentage of reduction of the safety factor of *N. viridula* females on treated surfaces (hydrophilic and hydrophobic glass, adaxial leaf surfaces of *P. laurocerasus*

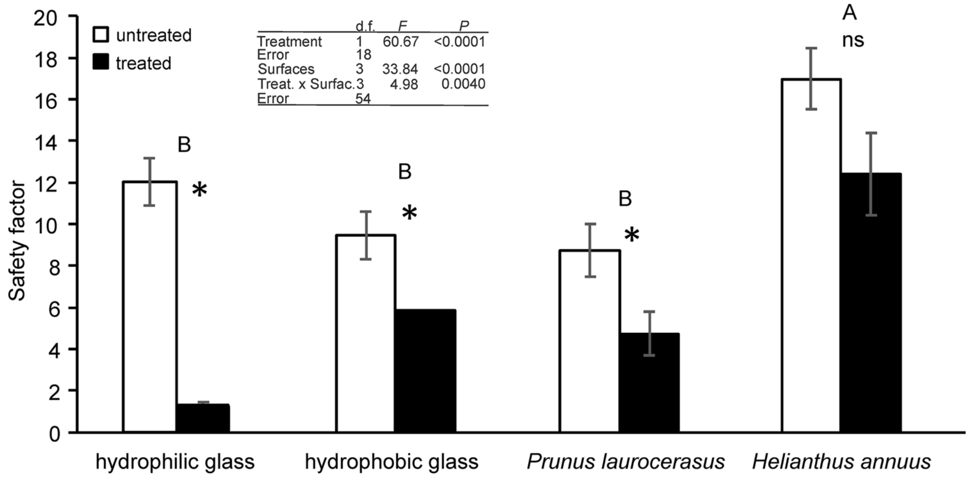

Fig. 6 Safety factor (friction force divided by the insect weight) obtained in friction force experiments with *Nezara viridula* females on treated (covered by kaolin particle film) and untreated surfaces. Columns indicate the means ± SEM. Asterisk (*) means significantly different at $p < 0.05$ and ns means not significant, Student's *t* test for dependent samples. Columns with different letters are significantly different at $p < 0.05$, ANOVA, Tukey HSD post hoc test. Table inset shows the statistical parameters of two-way repeated measures ANOVA

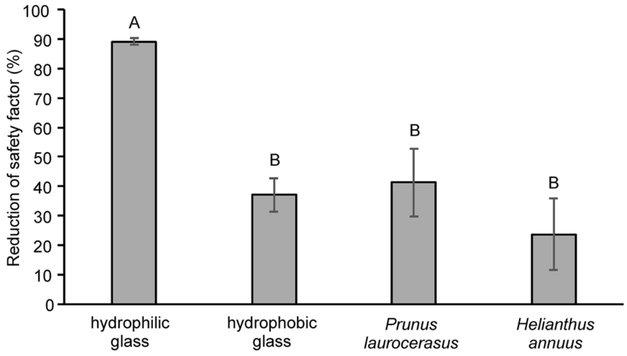

Fig. 7 Percentage of reduction of the safety factor obtained in friction force experiments with *Nezara viridula* females on treated (covered by kaolin particle film) surfaces in relation to the untreated ones. Columns indicate the means ± SEM. Columns with different letters are significantly different at $p < 0.05$, one-way ANOVA, Tukey HSD post hoc test

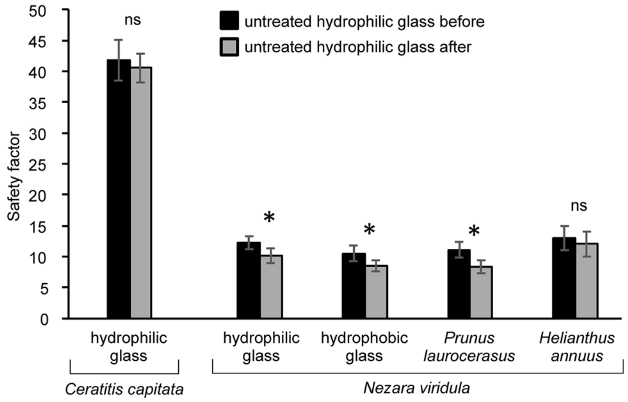

Fig. 8 Safety factor (friction force divided by the insect weight) obtained with *Ceratitis capitata* and *Nezara viridula* females on hydrophilic glass before and after walking on treated surfaces. Columns indicate the means ± SEM. Asterisk (*) means significantly different at $p < 0.05$ and ns means not significant, Student's *t* test for dependent samples

and *H. annuus*) in relation to the untreated ones was significantly higher on hydrophilic glass than on all the other tested surfaces (one-way ANOVA: $F_{3,36} = 13.08$, $p < 0.0001$) (Fig. 7). There was no significant difference in the percentage of reduction of the safety factor of *N. viridula* females on treated surfaces compared with the untreated ones among hydrophobic glass, *P. laurocerasus* adaxial leaf side and *H. annuus* adaxial leaf side.

Evaluation of the contaminating effect of kaolin particles

In the traction force experiments with *N. viridula* and *C. capitata* females on hydrophilic glass before and after walking on treated surfaces, no significant difference has been recorded in the safety factor of *C. capitata* after they walked on a hydrophilic glass covered with kaolin particle film (Student's *t* test for dependent samples: $t = 0.79$, $df = 9$, $p = 0.4490$) (Fig. 8). In *N. viridula,* no significant difference in the safety factor has been found after walking on the treated adaxial side of *H. annuus* (Student's *t* test for dependent samples: $t = 1.21$, $df = 9$, $p = 0.2579$), while the insect safety factor was significantly lower on the second test on glass than on the first one after the females walked on the treated hydrophilic glass ($t = 2.35$; $df = 9$; $p = 0.0435$), treated hydrophobic glass (Student's *t* test for dependent samples: $t = 5.94$, $df = 9$, $p = 0.0002$) and treated adaxial side of *P. laurocerasus* leaf (Student's *t* test for dependent samples: $t = 4.17$, $df = 9$, $p = 0.0024$) (Fig. 8).

Cryo-SEM investigations of the tarsal attachment devices of *N. viridula* and *C. capitata* females after they walked on treated hydrophilic glass revealed that both the smooth pads of the stink bug (Fig. 9a, b) and the hairy pads of the medfly (Fig. 9c, d) are contaminated by the kaolin plates. In particular, large areas of the smooth pulvilli in *N. viridula* appear covered by crusts formed by kaolin powder plates

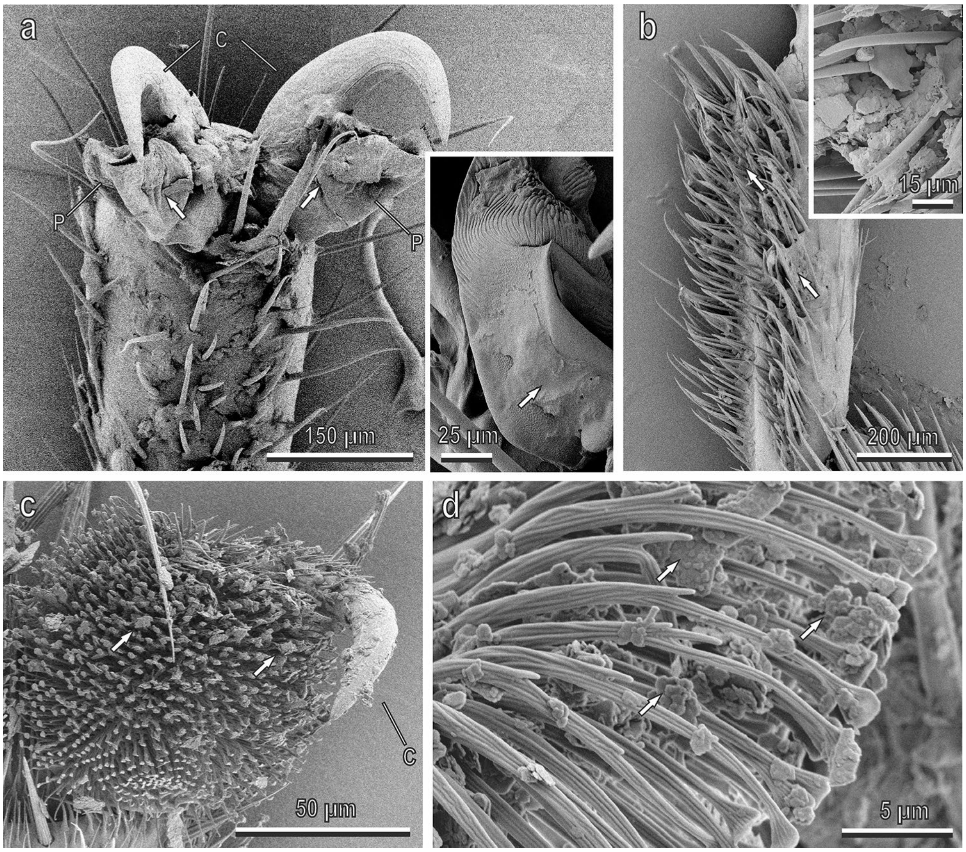

Fig. 9 Tarsal attachment devices of *Nezara viridula* (**a**, **b**) and *Ceratitis capitata* (**c**, **d**) females after walking on treated hydrophilic glass (cryo-SEM). **a** Ventral surface of smooth pulvilli contaminated by the kaolin powder (arrows). Note in the inset the large area of pulvilli surface covered by crusts (arrows) formed by kaolin powder plates glued together. **b** Kaolin powder plates (arrows) accumulated among the adhesive setae of the basitarsal hairy pad. **c** Hairy pulvillus with kaolin plates (arrows) adhering to the terminal plate of some of the tenent setae. **d** Some kaolin plates (arrows) visible also between the tenent setae. C, claws; P, smooth pulvilli

glued together (Fig. 9a), while these last accumulate among the adhesive setae of the hairy pad located at the ventral side of the basitarsus (Fig. 9b). In *C. capitata,* kaolin plates are visible adhering to the terminal parts of some of the tenent setae (Fig. 9c) and also between setae (Fig. 9d).

Discussion

Kaolin particle film reduces insect attachment ability

The present investigation testing with traction force experiments the effect of kaolin particle film in reducing the attachment ability of two important insect pests on natural and artificial surfaces demonstrates that adhesion is heavily impacted by the presence of the film in both *N. viridula* and *C. capitata*.

As far as *C. capitata* is concerned, the kaolin powder film represents an effective alternative to conventional insecticides in integrated and traditionally managed orchards. Different studies demonstrated in the field and in laboratory conditions the role of the kaolin particle film in reducing medfly female landing and oviposition on treated fruit surfaces (Mazor and Erez 2004; D'Aquino et al. 2011; Lo Verde et al. 2011). It is assumed that the main mechanism of action of kaolin against the medfly and other Tephritidae is related to its interference with the insect host selection process, since it renders fruit surface white and alters the visual cues, which are important for these insects to locate hosts at a short distance (Katsoyannos 1987; Saour and Makee 2004; Villanueva and Walgenbach 2007). In *Bactrocera oleae* (Rossi) (Diptera: Tephritidae) (Saour and Makee 2004), it has been hypothesized that gravid females that visited particle-treated olives were repelled for behavioral reasons due to the tactile unsuitable surface texture of such olives. In addition to these mechanisms of action mainly based on the modified sensory cues emitted from the host fruits, is possible that the reduction in adhesion to the fruit surface, which is clearly shown by our results, could contribute to the success of kaolin particle film in reducing Tephritidae oviposition.

To our best knowledge, no field study has been performed so far to evaluate the role of kaolin particle film in controlling the Southern green stink bug, but some data are available regarding effective management in the field with kaolin particle film treatments against other pentatomid species such as *Chinavia hilaris* Say, *Euschistus servus* (Say) and *E. tristigmus* (Say) (Lalancette et al. 2005). Further investigations in the field should be performed to test the effect of kaolin particle film to control pentatomid bugs in consideration of the high impact of the "particle film technology" in reducing the adhesion of *N. viridula* evidenced by our results, a reduction in adhesion even higher than that recorded in *C. capitata*.

Kaolin particle film-induced reduction of insect attachment ability depends on the treated surface

In our experiments, the percentage of reduction of insect adhesion on the treated substrates compared with the untreated ones changed according to the kind of substrate in relation to its initial wettability and morphology. Indeed, kaolin particle film could reduce insect adhesion on both hydrophilic and hydrophobic artificial surfaces, but the degree of force reduction in *N. viridula* was significantly higher on hydrophilic glass than on hydrophobic one. This can be related to the different distribution of kaolin particle film, which on hydrophilic glass can reach a percentage of covered area of about 70% of the surface, while on hydrophobic glass results in only 18%. A low percentage of covered area (about 21%) occurs also on treated hydrophobic natural surfaces such as the adaxial leaf side of *P. laurocerasus* showing 2D epicuticular wax film/layer, where kaolin film forms round-shaped dry droplets. In the case of *H. annuus,* the dense pubescence of the adaxial leaf side composed by three types of trichomes together with rather hydrophilic nature of the leaf surface makes the surface rather absorptive, where water drop quickly spreads over the surface and is partly sucked by the hairy coverage. This allows obtaining a percentage of covered area of about 65% but, notwithstanding this, there is no significant reduction of *N. viridula* friction force on the treated leaf of this plant. This is undoubtedly due to the presence of trichomes. Indeed, a recent study on the attachment ability of adults of the Southern green stink bug to plant leaves with different morphological features (Salerno et al. 2018a) revealed the best attachment on plants with dense pubescence, such as *Solanum melongena* L., suggesting that the trichomes present on both leaf surfaces of this plant are probably used by insect claws to improve attachment during pulling. Although one of the main function of plant trichomes is to contribute to the plant defense mechanism against herbivores (Gorb and Gorb 2013), it was reported for some insects on some plants that trichomes may serve as additional "foothold" promoting insect attachment to the surface (Southwood 1986; Stork 1980; Gorb and Gorb 2002; Voigt et al. 2007). The possibility to use trichomes as clinging sites for the tarsal claws could highly decrease the effect of the kaolin powder film in reducing insect adhesion to the plant surface, which is mainly due to pulvilli contamination (see below). In agreement with this result, we observed no significant difference in the safety factor of *N. viridula* on untreated glass surfaces before and after walking on the adaxial leaf side of *H. annuus* treated with kaolin, differently from what was recorded in cases of other kaolin-treated surfaces (i.e., hydrophilic glass, hydrophobic glass and *P. laurocerasus*), where insects used mainly pulvilli.

Mechanism of action of kaolin particle film in reducing insect attachment ability

To unravel the mechanism of reducing effect action of kaolin particle film on insect attachment ability, we evaluated the safety factor for females before and after walking on treated surfaces and analyzed under cryo-SEM the tarsal attachment devices of *N. viridula* and *C. capitata* females after walking on treated surfaces. We observed contamination by the kaolin plates on both the smooth pads of the stink bug and the hairy pads of the medfly. This contamination is particularly evident in *N. viridula*, where large areas on the ventral surface of smooth pulvilli are covered by crusts of kaolin plates, which were glued together probably due to the presence of tarsal fluids secreted into the contact zone between the adhesive pad and the substrate (Peisker et al. 2014; Rebora et al. 2018). These glued together kaolin particles covering the pads could reduce the ability of pulvilli to adhere to the substrate surface profile, which results in a significant decrease of the insect safety factor not only on a treated surface, but also on a clean one after walking on a treated surface, as shown in our experiments with *N. viridula*.

Attachment of kaolin particle films to the insect's body parts has been reported earlier (Glenn et al. 1999; Glenn and Puterka 2005), but we provide the first detailed description of kaolin particle film on insect tarsal attachment devices, which can interfere with its adhesion to the surface. Such a mechanism of action of kaolin particle film in reducing insect adhesion is similar to that described in the "contamination hypothesis" proposed to explain the effect of epicuticular plant waxes on reducing insect attachment (Gorb and Gorb 2002; Gorb et al. 2014). It is well known that plant surfaces bearing 3D wax coverage can greatly reduce insect attachment (Stork 1980; Atkin and Hamilton 1982; Gaume et al. 2002; Gorb et al. 2004, 2008; Salerno et al. 2018a). Different mechanisms have been proposed to explain this phenomenon (see review in Gorb and Gorb 2013), among which the contamination hypothesis stating that the detached wax projections serve as a separation layer between plant surface and insect attachment pads (Gorb and Gorb 2002; Gorb et al. 2014). Another mechanism of action of waxes in reducing insect adhesion is suggested by the "fluid-absorption hypothesis" stating that the absorption of the pad fluid due to the structured wax coverage reduces insect attachment ability (Gorb et al. 2017, 2019). We cannot exclude this mechanism of action also for kaolin particle film owing to is high hydrophilicity and adsorption ability. On the contrary, from the explanations of the mechanisms of action of kaolin particle film, we can reduce the role of roughness according to the "roughness hypothesis" (Gorb and Gorb 2002; Scholz et al. 2010) stating that on plants covered by wax projections, the adhesion reducing effect is due to the specific surface microroughness. Tarsal smooth or hairy pads in many insects, among which *N. viridula* (Salerno et al. 2017, 2018b) and *C. capitata* (Salerno et al. 2019, submitted), can generate sufficient contact mainly with smooth substrates or smooth islands within rough substrates, whereas there is a great reduction of the contact area with small surface irregularities having 0.3–1.0 µm size (Gorb 2001; Peressadko and Gorb 2004; Voigt et al. 2008; Wolff and Gorb 2012; Zhou et al. 2014; Zurek et al. 2017; Kovalev et al. 2018). Since kaolin particle film exhibits much courser microroughness than the "critical" microrough surfaces mentioned above [$R_a = 0.074–0.20$ µm, RMS = 0.09–0.24 µm according to Peressadko and Gorb (2004) and Salerno et al. (2017, 2018b)], we can exclude strong influence of this mechanism of insect adhesion reduction for treated surfaces tested here.

Conclusions

The present investigation highlighted the effective role of kaolin particle film in reducing insect adhesion to artificial and natural substrates characterized by different surface features. Figuring out the biomechanics of the interaction between the kaolin particle film covered surfaces and attachment devices of insect legs can lead to the development of new physical control methods, such as physical barriers that protect crops from pest infestation. Management of agricultural insects with physical control methods is particularly relevant owing to the need to decrease the negative impacts of pesticides on the environment and human health (Vincent et al. 2003).

Author contributions

The study was designed by all the authors. S.G. and E.G. performed the cryo-scanning electron microscopy investigations. G.S. and M.R. performed the traction force experiments. A.K. and E.G. characterized the tested surfaces. The manuscript was written by M.R., G.S. and E.G. All authors discussed the analysis and interpretation of the results and participated in the final editing of the manuscript.

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Compliance with ethical standards

Conflict of interest All the authors declare that they have no conflict of interest.

Ethical approval All applicable international, national, and/or institutional guidelines for the care and use of animals were followed. This article does not contain any studies with human participants performed by any of the authors.

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