

Original article (Orijinal araştırma)

Physicochemical properties of local diatomaceous earths and their effects of water-suspension treatments against *Callosobruchus maculatus* (Fabricius, 1775) (Coleoptera: Chrysomelidae) on chickpeas

Yerel diatom toprakların fiziko-kimyasal özellikleri ve nohutlarda sulu süspansiyon uygulamalarına *Callosobruchus maculatus* (Fabricius, 1775) (Coleoptera: Chrysomelidae)'a karşı etkileri

Hüseyin BOZKURT^{1*}

Abstract

This study evaluated the chemical and physical characteristics of two locally sourced diatomaceous earth (DE) formulations (Detech[®]-s and Detech[®]-m) alongside the commercial product SilicoSec[®] to assess their effects on adult mortality, lethal time (LT₉₉), and progeny reduction of the cowpea weevil, *Callosobruchus maculatus* (Fabricius, 1775) (Coleoptera: Chrysomelidae) in stored chickpeas. Conducted in 2023 at the Entomology Laboratory of Kahramanmaraş Sütçü İmam University, the research found Detech[®]-m to be the most effective formulation. It achieved 99% mortality in the shortest time (LT₉₉=70.65 hours at 1000 ppm) and the highest progeny suppression (98.1%). This superior performance was attributed to its finer particle size (d₅₀=7.34 µm) and larger surface area (60.45 m²/g), which enhanced adsorption and cuticle disruption. Although SilicoSec[®] contained a higher percentage of silicon dioxide (85.7% SiO₂) than Detech[®] (80.6% SiO₂), its broader particle size distribution and lower Brunauer-Emmett-Teller (BET) surface area reduced its effectiveness. These findings highlight the critical role of particle size and surface area in optimizing DE formulations for *C. maculatus* control. Overall, our results suggest that refining DE formulations for water-suspension applications can improve stored-legume pest management by enhancing adhesion, uniformity, and persistence while minimizing dust-related health risks.

Keywords: Cowpea weevil, diatom powder, particle size, slurry application, surface area

Öz

Bu çalışmada, yerel olarak temin edilen iki diatom toprağı (DE) formülasyonunun (Detech[®]-s ve Detech[®]-m) ve ticari formülasyon SilicoSec[®]'in kimyasal ve fiziksel özelliklerini değerlendirerek, bunların depolanmış nohutlardaki börülce tohum böceğı, *Callosobruchus maculatus* (Fabricius, 1775) (Coleoptera: Chrysomelidae) üzerindeki ergin ölümleri, lethal süre (LT₉₉) ve yeni nesil ergin sayısındaki azaltılmasına etkileri incelenmiştir. Çalışma, 2023 yılında Kahramanmaraş Sütçü İmam Üniversitesi'nin Entomoloji Laboratuvarı'nda gerçekleştirilmiştir. Sonuçlar, Detech[®]-m'nin en etkili olduğunu, en kısa sürede (LT₉₉=1000 ppm'de 70.65 saat) %100 ölüm oranına ve en yüksek yeni nesil ergin baskılamasına (%98.1) ulaştığını göstermiştir. Daha ince partikül boyutu (d₅₀=7.34 µm) ve yüksek yüzey alanı (60.45 m²/g), Detech[®]-m formülasyonunun adsorpsiyon ve böcek kütikulasını bozma özelliklerini artırarak üstün etkinlik göstermesine katkıda bulunmuştur. SilicoSec[®]'in daha yüksek silisyum dioksit içeriğine (%85.7) sahip olmasına rağmen, Detech[®]-m (%80.6) ile karşılaştırıldığında daha geniş partikül boyutu dağılımı ve daha düşük Brunauer-Emmett-Teller (BET) yüzey alanı nedeniyle daha düşük bir etki göstermiştir. Bu bulgular, *C. maculatus* mücadelesinde DE formülasyonlarının etkinliğini optimize etmede partikül boyutunun ve yüzey alanının kritik rolünü vurgulamaktadır. Sonuç olarak, bu çalışmada elde edilen bulgular su süspansiyon uygulamaları için DE formülasyonlarının yapışma, homojenlik ve kalıcılığı artırarak optimize edilmesi depolanmış tahıl zararlılarının yönetimini iyileştirebileceğini ve toz kaynaklı sağlık risklerini en aza indirebileceğini göstermektedir.

Anahtar sözcükler: Börülce tohum böceğı, diatom tozu, partikül büyüklüğü, sulu bulamaç uygulaması, yüzey alanı

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Introduction

Stored-product insect pests pose a significant threat to global food security by causing substantial post-harvest losses in cereals, legumes, and other agricultural commodities (Phillips & Throne, 2010). These pests, including beetles (Coleoptera) and moths (Lepidoptera), inflict direct grain damage, reduce weight, and contaminate products with feces, webbing, and frass, ultimately diminishing quality and marketability (Lorini et al., 2007). To mitigate these losses, conventional chemical insecticides such as organophosphates and pyrethroids, have been widely used (Arthur, 1996; Kljajic & Peric, 2007; Agrafioti et al., 2015; Vassilakos et al., 2015; Rumbos et al., 2018; Mutlu et al., 2019). However, concerns over insecticide resistance, residue accumulation, and environmental toxicity have driven the search for alternative control methods (Arthur, 1996; Athanassiou et al., 2007a). Among these eco-friendly alternatives, diatomaceous earth (DE) has gained attention as a promising insecticidal agent due to its physical mode of action rather than chemical toxicity (Korunić, 1998). DE formulations are considered as safe, effective, and sustainable for stored-grain pest management, making them a viable substitute for synthetic pesticides (Subramanyam & Roesli, 2000; Athanassiou et al., 2011). However, their efficacy depends on physicochemical properties such as particle size, surface area, chemical composition, and hydrophobicity (Korunić, 1997, 2013; Korunić et al., 2021; Mewis & Ulrichs, 2001; Ogreten et al., 2023; Henteş & Işıkber, 2024). Understanding how these factors influence insecticidal activity is essential for optimizing DE-based formulations for practical use.

Diatomaceous earth (DE) is a naturally occurring sedimentary material composed primarily of fossilized diatom remains, with a high silica (SiO_2) content ranging from 80% to 93% (Korunić, 1998). Depending on the deposit source, it may also contain clay minerals, organic matter, quartz, calcium carbonate, and magnesium oxide (Stathers et al., 2004). The unique porous structure of DE particles provides a high surface area, enhancing their strong adsorption properties, which contribute to their insecticidal action (Mewis & Ulrichs, 2001, Saçlam et al., 2017). DE's effectiveness against insect pests stems from its ability to disrupt the cuticle, leading to desiccation and death. The particles adhere to the exoskeleton and absorb lipid layers from the epicuticle, causing excessive water loss and dehydration (Ebeling, 1971; Subramanyam & Roesli, 2000). Additionally, its abrasive nature creates micro abrasions on the insect's body, further accelerating water loss (Korunić, 2013). Unlike synthetic insecticides, which act through neurotoxic mechanisms, DE remains effective against insect populations regardless of resistance development (Athanassiou et al., 2007b).

While DE is widely recognized as an effective non-chemical insecticide, its efficacy varies significantly based on multiple physicochemical factors. Understanding these properties is essential for optimizing DE formulations for stored-product protection. Particle size plays a crucial role in DE's ability to adhere to the insect cuticle and remove its protective wax layer (Athanassiou et al., 2003). Smaller particles generally exhibit higher insecticidal activity due to their greater surface area and enhanced adsorption capacity (Korunić, 2013). However, excessively fine particles can pose dust hazards and increase inhalation risks, raising concerns about worker safety in grain storage facilities (Subramanyam et al., 1994). The chemical composition of natural DE deposits also influences insecticidal properties. A higher amorphous silica content is generally associated with greater efficacy, whereas impurities such as clay and quartz may reduce effectiveness by diluting the active components (Stathers et al., 2004). Additionally, studies indicate that calcined DE, which undergoes high-temperature treatment, differs in physical characteristics from natural DE, affecting both its adsorptive capacity and insecticidal activity (Athanassiou et al., 2005).

In recent years, researchers have compared the effectiveness of aqueous and powder formulations of various commercial DE products against stored-grain pests. Studies have shown that dust applications, where DE is sprayed onto grain, are more effective at controlling insect and mite pests than wet applications (Field & Korunić, 2000; Athanassiou et al., 2006; Collins & Cook, 2006a; Wakil et al., 2006; Athanassiou & Korunić, 2007c). Similarly, research indicates that when DE formulations are applied to different surfaces,

dust formulations exhibit higher efficacy than aqueous formulations (Bridgeman, 1994; McLaughlin, 1994; Collins & Cook, 2006b). A study comparing the reduced effectiveness of aqueous DE formulations on wheat to dust formulations found that diatom particles adhere more readily to cereal grains in wet applications (Johnson et al., 2014). This phenomenon is attributed to the formation of capillary bridges between DE particles and the grain surface due to residual surface water (Israelachvili, 2011). The adhesion force resulting from capillary bridging is primarily driven by water's surface tension. Additionally, studies have shown that DE particles fail to adhere effectively to the insect cuticle upon contact, also due to water's high surface tension.

Although Türkiye has substantial diatom deposits, the potential use of locally derived DE for insect control has only recently been evaluated. Most studies have tested DE preparations in powder form against various pests in grain storage (Doğanay, 2013; Alkan et al., 2019; Şen et al., 2019; Ertürk et al., 2020; Öztekin & Mutlu, 2020; Kılıç, 2022; Sağlam et al., 2022a; Henteş & Işıkber, 2024). However, powder applications often present challenges, including difficulty achieving homogeneous distribution, poor adherence to surfaces, and powder drift during application. In many cases, DE is applied as water suspension, but its effectiveness against stored-grain pests is known to be somewhat reduced compared to powder applications (Field & Korunić, 2000; Athanassiou et al., 2006; Collins & Cook, 2006b; Wakil et al., 2006; Athanassiou & Korunić, 2007c). While the challenges of powder application are well-documented, research on the effects of mixing locally sourced DE with surfactants for spray applications remains limited (Ertürk et al., 2020; Kılıç, 2022). Therefore, there is a need for a locally sourced DE formulation with a flowable, easy-to-apply suspension suitable for spray application on insect-infested products. This study aimed to characterize the physicochemical properties of two Turkish-origin DE formulations (Detech[®]-s and Detech[®]-m) and the commercial product SilicoSec[®] while evaluating their insecticidal efficacy in water-suspension treatments against the cowpea weevil, *Callosobruchus maculatus* (Fabricius, 1775) (Coleoptera: Chrysomelidae: Bruchinae) in stored chickpeas. Additionally, the study sought to identify key formulation properties influencing DE performance in aqueous applications.

Materials and Methods

Test insect rearing

In the biological tests, adult cowpea seed beetles, *C. maculatus* were used. The initial insect population was obtained from chickpea samples collected from legume warehouses in the Mersin and Adana provinces. The adults were reared in the Laboratory of the Plant Protection Department, Faculty of Agriculture, Kahramanmaraş Sütçü İmam University, where a stock culture was established for routine experiments. The "Azkan" variety of chickpeas, *Cicer arietinum* L. (Fabales: Fabaceae), with a moisture content of 10±1%, served as both the culture medium and the test diet. Before use in experiments, chickpeas were stored at -20°C for 7-10 days to eliminate potential contamination. For rearing, 300 g of chickpeas were placed in 1-liter glass jars, and 100-150 mixed-sex adults (1-2 days old) were introduced. The jars were covered with mesh to allow airflow. The jars were kept in a cooled incubator (Lovibond TC-135S, Germany) under controlled conditions: 65±5% relative humidity, 30±1°C temperature, and complete darkness. To facilitate mating and egg-laying, adults remained in the jars for two days before being removed using a 2 mm mesh metal sieve (Retsch[®], Germany). The eggs laid on the chickpeas hatched, and the emerging larvae to penetrate the grains. The larvae and pupae completed their development inside the chickpeas, and the cultures were maintained under the same incubator conditions until the new generation of adults emerged. Once the new adults emerged, they were collected from the culture jars using a 2 mm mesh metal sieve and used in subsequent biological tests.

Diatomaceous earth formulations

Two forms of the local diatomaceous earth (DE) formulation, Detech[®], were used in the biological tests: the standard (Detech[®]-s) and the micronized (Detech[®]-m) powder formulations. The standard formulation (Detech[®]-s), characterized by a relatively coarse particle size distribution, was produced by grinding DE deposits using a laboratory-scale ball mill (RAM1107; Rantek Test Solutions, Ankara). In contrast, the micronized formulation (Detech[®]-m), distinguished by its finer particle size distribution, was prepared using a lab-scale fluidized bed opposed jet mill (AFG 100, Hosokawa Alpine AG, Germany). Detech[®] is a blend of freshwater DE deposits sourced from three reserves in Middle Anatolia, Türkiye. It primarily consists of naturally occurring amorphous silica, has a white-grey color (Figure 1a), and is formulated by Entoteam R&D Food Agriculture Co. as a pest control-grade insecticide. For comparison, the commercial DE formulation SilicoSec[®] was used as a positive control in the biological tests. SilicoSec[®], a freshwater diatomaceous earth formulation manufactured by Biofa GmbH (Münsingen, Germany), is derived from fossilized single-celled diatoms and has a white color (Figure 1b).



Figure 1. Powder formulations of a) the local standard diatomaceous earth (Detech[®]), and b) the commercial diatomaceous earth (SilicoSec[®]) used in biological tests.

Physicochemical characterization of tested diatomaceous earth powder formulations

Quantitative mineral analyses of three diatomaceous earth (DE) powder formulations (Detech[®]-s, Detech[®]-m, and SilicoSec[®]) were conducted at the Accredited Mineralogy and Petrography Laboratory of the General Directorate of Mineral Research and Exploration of Türkiye (MTA). Elemental composition was determined using atomic absorption spectroscopy (AAS) following a melting and acid removal process. Particle size and distribution analyses were performed using a Mastersizer 2000 laser diffraction particle size analyzer (Malvern Instruments) with wet dispersion. Measurements were taken in triplicate using a micro-volume (200 mg) sample feeder at 2-second intervals, with obscuration limits set between 5% and 15%. The Brunauer-Emmett-Teller (BET) method was used to assess the specific surface area by measuring nitrogen multilayer adsorption relative to pressure. This technique evaluates both external and pore surface areas, providing crucial data on total specific surface area (m²/g) and offering insights into factors affecting particle size and surface porosity in various applications. BET surface area measurements were conducted using a Quantachrome[®] ASiQwin[™] (version 5.23) instrument (Quantachrome Instruments, Boynton Beach, FL, USA) at the MTA laboratory. Before measurement, samples were dried at 150°C for 24 hours and degassed at 200°C for 5 hours under vacuum.

To examine the morphological and structural characteristics of the silica-based remains of diatoms—single-celled algae with intricate, species-specific silica frustules—Scanning Electron Microscopy (SEM) analyses were conducted at the Scientific and Technological Research Implementation and Research Center of Tekirdağ Namık Kemal University (NABILTEM). Water and oil absorption capacities were

determined using the gravimetric method. Before testing, DE samples were conditioned at 80°C for 24 hours in an air-circulating oven. Oil absorption capacity was measured according to the ASTM D1483 standard, in which linseed oil was titrated onto a 1 g DE sample until free oil appeared (ASTM, 2012). Similarly, water absorption capacity was measured following the ASTM D570 standard, where water was titrated onto a 1 g DE sample until free water appeared (ASTM, 2012). Tapped bulk density analysis was performed by placing 10 g of DE in a 100 mL graduated cylinder. The cylinder was tapped 100 times on a hard rubber surface, and the final volume was recorded. Results were expressed in grams per liter (g/L) (Korunić, 1997). All measurements were conducted in triplicate. The pH was determined by stirring 10 g of DE with 90 mL of double-distilled water for six hours, followed by measurement using a Hanna pH-211 meter. pH measurements were also performed in triplicate.

Commodity

For biological tests and insect cultures, the Azkan variety of edible chickpea, widely used in Türkiye, was selected. The moisture content of the Azkan chickpeas used in the experiments was measured at 10%±1% using a KETT PM-600 portable moisture analyzer (Kett Electric Laboratory, Japan).

Biological test with diatomaceous earth (DE) formulations

In the biological tests, diatomaceous earth (DE) formulations (Detech[®]-s, Detech[®]-m, and SilicoSec[®]) were applied to chickpea grains as water suspensions at concentrations of 500 ppm and 1000 ppm (mg DE/kg chickpea). The DE suspensions were prepared by mixing 0.5 g and 1 g of DE with 10 mL of water, respectively, in a 250 mL beaker using a magnetic stirrer. One kilogram of chickpea grains was spread in a single layer on a flat surface, and 10 mL of DE suspension at 500 ppm or 1000 ppm was evenly sprayed onto the grains using an HSENG Airbrush AS18 spray compressor (Ningbo Haosheng Pneumatic Machinery Co., Zhejiang, China). As a control treatment, only pure water was applied. After spraying, the chickpeas were left to dry at room temperature for one hour. Sub-samples of 50 g of DE-treated chickpeas were taken from the 1 kg batch and transferred to 100 mL glass vials (8.3 cm × 4.5 cm). Thirty mixed-sex adult insects (aged 1-2 days) were introduced into each vial. The vial openings were covered with gauze to prevent insect escape while ensuring airflow. Each DE treatment was replicated four times, with the vials placed inside 80 L plastic storage containers (26 cm × 36,5 cm × 15 cm) with lockable lids. To maintain 65±3% relative humidity, the containers were conditioned with a saturated sodium nitrate solution (Greenspan, 1976), and plastic grids were placed at the bottom. The containers were then stored in a refrigerated incubator set at 25±1°C.

The biological tests followed a randomized parcel design, with mortality assessed at 24-hour intervals over 6- and 7-day exposure to DE treatments. During each assessment, chickpea grains were sieved using a 2 mm sieve, and the number of dead and live adults was recorded. To evaluate *C. maculatus* F₁ progeny emergence, the glass vials were stored in a dark, climate-controlled room at 25±1°C and 65±5% relative humidity for 45 days. At the end of this period, the grains from both DE-treated and control samples were sieved using a 2 mm sieve, and the number of newly emerged adults was recorded.

Data processing and analysis

In the biological tests, mortality rates in DE treatments were corrected using Abbott's correction formula when mortality occurred in the control group by the end of the exposure period (Abbott, 1925). Percentage mortality at the end of each exposure period for spray applications was arcsine-transformed to normalize heteroscedastic treatment variances and analyzed using one-way analysis of variance (ANOVA), with DE formulation as the main factor. Transformed data were used for the analysis, whereas raw data were used in the figures. The analysis was performed using Minitab 21 (Minitab Inc., USA), and differences between means were determined using Tukey's Honestly Significant Difference (HSD) test at a 5% significance level.

Differences in the number of adult emergences in the new generation compared to the control were evaluated using Dunnett's test ($p=0.05$). Adult progeny counts for each concentration-DE formulation combination were compared to those in the control vials using Dunnett's test at a 5% significance level. Percentage reduction in progeny emergence for each concentration was analyzed using one-way ANOVA, with DE formulation as the main factor. Mean percentage reductions in new-generation adult emergence for each concentration and DE formulation were compared using Tukey's HSD test at a 5% significance level.

Percentage mortality data over different exposure times for each DE formulation were subjected to probit analysis using the PC-POLO program (Leora Software, 1994). Based on the probit analysis, lethal time (LT_{50} , LT_{90} , and LT_{99}) values were calculated for each DE formulation at 500 and 1000 ppm concentrations. Differences among LT_{50} , LT_{90} , and LT_{99} values were determined by examining the overlap of their lower and upper confidence intervals.

Results

Physicochemical characterization of tested diatomaceous earth powder formulations

Scanning Electron Microscopy (SEM) images of the Detech[®] formulation (Figure 2a) reveal numerous cylindrical and rod-shaped diatom skeletons, whereas the commercial DE formulation SilicoSec[®] consists of fossilized single-celled diatoms with a rod-like structure (Figure 2b).

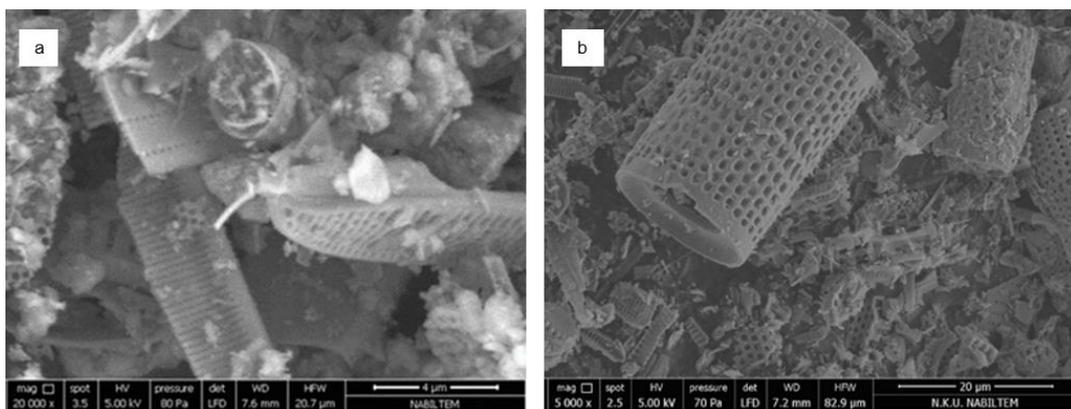


Figure 2. Scanning Electron Microscopy (SEM) images of diatom frustules in a) the local standard DE formulation (Detech[®]) at 20,000x magnification, and b) the commercial DE formulation (SilicoSec[®]) at 5,000x magnification.

The chemical composition of the diatomaceous earth formulations used in biological tests is provided (Table 1). Notably, SilicoSec[®] has a higher silicon dioxide (SiO_2) content (85.7%) than Detech[®] (80.6%). Figure 3 presents the particle size distributions (by volume) of the three DE formulations: Detech[®]-s, Detech[®]-m, and SilicoSec[®]. The particle sizes (in μm) at the 10th, 50th, 90th, and 99th percentiles of the cumulative size distribution (d_{10} , d_{50} , d_{90} , and d_{99}) are provided in Table 2. Detech[®]-m and SilicoSec[®] are relatively fine powders, with d_{50} values of approximately 7.34 μm and 12.69 μm , respectively, and d_{99} values ranging from 41.82 to 42.79 μm . In contrast, Detech[®]-s consists of coarser particles, as indicated by its cumulative frequency distribution, where 50% of particles measure 24.89 μm and 99% reach 280.39 μm . Uniformity analysis showed that Detech[®]-s (1.20) and Detech[®]-m (0.69) had similar uniformity values, while SilicoSec[®] (0.53) exhibited a much lower uniformity value, indicating a narrower particle size distribution with more consistent particle sizes.

Table 1. Chemical composition of the diatomaceous earths used in biological tests

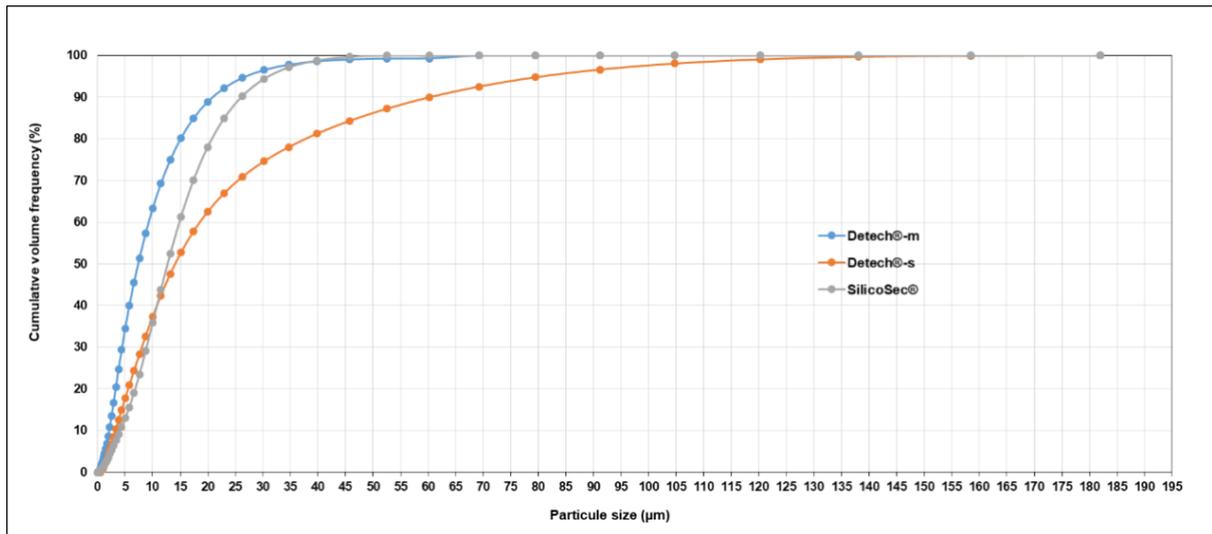
DE samples	Compound weight percentage (%)							
	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	TiO_2
Detech [®]	80.6	4.7	1.5	4.75	0.85	0.4	0.5	<0.01
SilicoSec [®]	85.7	4.45	1.35	0.45	0.25	0.35	0.35	0.2

Table 2. Particle size distribution and Brunauer-Emmett-Teller (BET) specific surface area of three diatomaceous earth formulations (Detech[®]-s, Detech[®]-m, and SilicoSec[®])^{*}

DE formulations	Particle size distribution				Uniformity	BET specific surface area (m ² /g)
	d (10) (μm)	d (50) (μm)	d (90) (μm)	d (99) (μm)		
Detech [®] -s	3.22±0.11ab	24.89±0.42 a	135.84±1.61 a	280.39±5.13 a	1.20±0.072 a	40.35±1.88 b
Detech [®] -m	2.08±0.18 b	7.34±0.69 c	20.83±1.74 b	41.82±2.59 b	0.69±0.13 a	60.45±2.30 a
SilicoSec [®]	4.07±0.56 a	12.69±0.95 b	26.05±1.50 b	42.79±2.45 b	0.53±0.067 b	43.81±2.09 b
<i>F</i> and <i>p</i> value [*]	<i>F</i> _{2,6} =8.44 <i>p</i> =0.018	<i>F</i> _{2,6} =24.28 <i>p</i> =0.001	<i>F</i> _{2,6} =175.62 <i>p</i> < 0.001	<i>F</i> _{2,6} =155.34 <i>p</i> < 0.001	<i>F</i> _{2,6} =12.61 <i>p</i> =0.007	<i>F</i> _{2,6} =24.50 <i>p</i> =0.001

^{*} A one-way analysis of variance (ANOVA) was conducted, and differences between means were determined using Tukey's HSD test at a 5% significance level. The different letters in the column indicate statistically significant differences.

The Brunauer-Emmett-Teller (BET) analysis revealed that Detech[®]-m had a significantly greater specific surface area (SSA) (60.45 m²/g) compared to Detech[®]-s and SilicoSec[®] (*F*_{2,6}=24.50, *p*=0.001). Detech[®]-s (43.81 m²/g) and SilicoSec[®] (40.35 m²/g) had nearly identical BET specific surface area values.

Figure 3. Particle size distribution (by volume) of three diatomaceous earth formulations (Detech[®]-s, Detech[®]-m, and SilicoSec[®]).

There were significant differences in oil and water absorption values among the tested DE formulations (*F*_{2,6}=293.1, *p*< 0.0001; *F*_{2,6}=68.5, *p*< 0.0001, respectively). SilicoSec[®] exhibited the highest oil absorption (173.6 g/mL), which was significantly greater than that of both Detech[®] formulations (Table 3).

Table 3. Physical properties of three diatomaceous earth formulations (Detech[®]-s, Detech[®]-m, and SilicoSec[®])^{*}

DE formulation	Oil absorption value (g/mL)	Water absorption value (g/mL)	Mean pH (n=3)	Compressed density (g/L)
Detech [®] -s	127.5±1.3 c	119.6±0.8 b	8.6±0.0 a	441.3±6.5 b
Detech [®] -m	141.6±1.2 b	124.0±1.5 b	7.8±0.0 b	340.9±3.8 a
SilicoSec [®]	173.6±1.5 a	147.0±2.5 a	7.7±0.0 b	337.1±3.8 a
<i>F</i> and <i>p</i> value [*]	<i>F</i> _{2,6} =293.1 <i>p</i> < 0.0001	<i>F</i> _{2,6} =68.5 <i>p</i> < 0.0001	<i>F</i> _{2,6} =124.4 <i>p</i> < 0.0001	<i>F</i> _{2,6} =143.9 <i>p</i> < 0.0001

^{*} A one-way analysis of variance (ANOVA) was conducted, and differences between means were determined using Tukey's HSD test at a 5% significance level.

Detech[®]-m had an intermediate absorption value (141.6 g/mL), while Detech[®]-s had the lowest (127.5 g/mL). Similarly, SilicoSec[®] had the highest water absorption (147 g/mL), followed by Detech[®]-m (124 g/mL) and Detech[®]-s (119.6 g/mL). In terms of pH, Detech[®]-s had the highest value (8.66), which was significantly different from both Detech[®]-m and SilicoSec[®] (*F*_{2,6}=124.4, *p*< 0.0001). Detech[®]-m (7.88) and

SilicoSec® (7.72) had similar pH values but remained statistically distinct from Detech®-s. While SilicoSec® and Detech®-m were mildly alkaline and close to neutral, Detech®-s was more alkaline, suggesting a higher potential for distinct chemical and biological interactions. Detech®-s also had the highest compressed density (441.3 g/L), whereas Detech®-m (340.9 g/L) and SilicoSec® (337.1 g/L) had significantly lower densities ($F_{2,6}=143.9$, $p < 0.0001$).

Mortality

The mean percentage mortality (\pm standard error) of *C. maculatus* adults exposed to 500 ppm of three DE formulations (Detech®-s, Detech®-m, and SilicoSec®) over a 7-day period is presented in Figure 4.

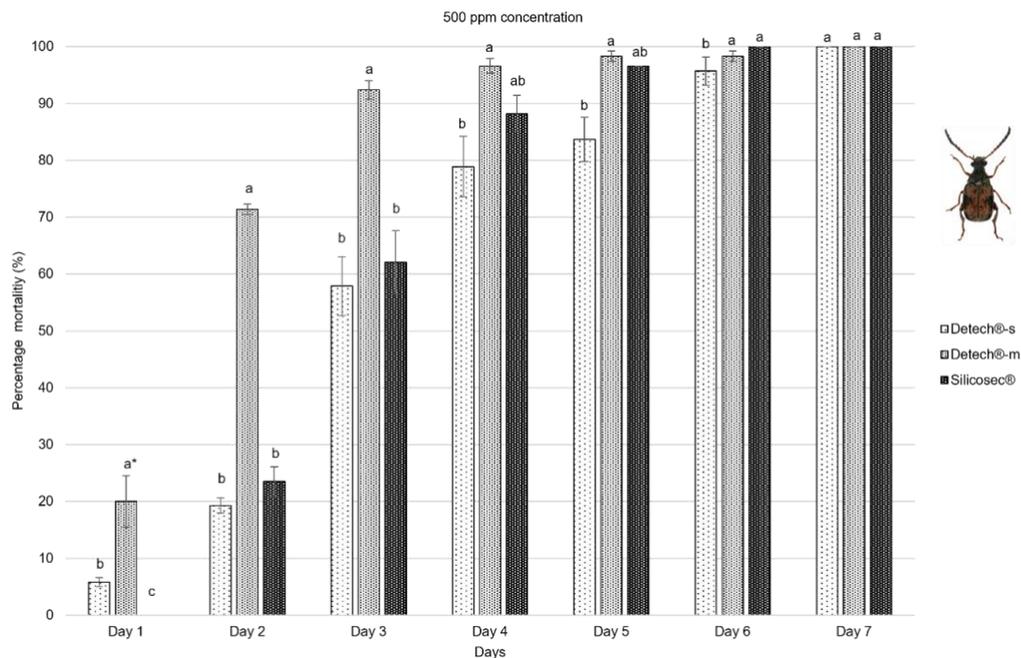


Figure 4. Mean percentage mortality (\pm standard error)* of *Callosobruchus maculatus* adults exposed to chickpeas treated with a water suspension of three diatomaceous earth (DE) formulations (Detech®-s, Detech®-m, and SilicoSec®) at 500 ppm over a 7-day period.

* A one-way analysis of variance (ANOVA) was conducted, and differences between means were determined using Tukey's HSD test at a 5% significance level. Bars with different letters indicate statistically significant differences and each day is compared within each day.

On Day 1, Detech®-m exhibited the highest initial mortality (20%), which was significantly higher than Detech®-s (5.8%) and SilicoSec® (0%) ($F_{2,9}=33.8$, $p < 0.0001$). Detech®-s was significantly more effective than SilicoSec®, while Detech®-m remained the most effective overall. By Day 2, Detech®-m showed a sharp increase in mortality (71.4%), whereas Detech®-s (19.3%) and SilicoSec® (23.5%) had significantly lower mortality rates ($F_{2,9}=218.6$, $p < 0.0001$). On Day 3, Detech®-m maintained the highest mortality (92.4%), followed by Detech®-s (57.9%) and SilicoSec® (62.1%). Significant differences persisted between Detech®-m and the other two DE formulations ($F_{2,9}=22.8$, $p < 0.0001$). On Day 4, Detech®-m approached 100% mortality, while Detech®-s and SilicoSec® continued increasing (80-88%). However, Detech®-m still trended slightly higher. By Day 5, Detech®-s reached at least 85% mortality, while Detech®-m and SilicoSec® achieved nearly complete mortality (>96%). By Day 6, all DE formulations had reached 100% or nearly 100% mortality. On Day 7, no significant differences remained between the formulations, as all achieved complete mortality. Overall, Detech®-m was significantly more effective than Detech®-s and SilicoSec® during the early exposure period (Days 1-3), while SilicoSec® was the least effective. However, by the end of the study, all DE formulations reached 100% mortality (Figure 4).

The mean percentage mortality (\pm standard error) of *C. maculatus* adults exposed to 1000 ppm of three DE formulations (Detech[®]-s, Detech[®]-m, and SilicoSec[®]) over a 6-day period is presented in Figure 5. On Day 1, Detech[®]-m exhibited the highest initial mortality (66.6%), which was significantly higher than SilicoSec[®] (2.5%). However, there was no significant difference between Detech[®]-m and Detech[®]-s, despite Detech[®]-s resulting in a slightly lower mortality rate (46.6%) ($F_{2,9}=35.3$, $p < 0.0001$). By Day 2, mortality remained high for both Detech[®]-m (94.1%) and Detech[®]-s (76.4%), while SilicoSec[®] had a significantly lower mortality rate (32.7%) ($F_{2,9}=21.9$, $p < 0.0001$). On Day 3, SilicoSec[®] showed a sharp increase in mortality (77.3%), but its mortality rate was still significantly lower than that of Detech[®]-m (98.3%) ($F_{2,9}=8.9$, $p < 0.0001$). By Day 4, Detech[®]-m achieved 100% mortality of *C. maculatus* adults, while SilicoSec[®] and Detech[®]-s reached complete mortality on Days 5 and 6, respectively. Overall, Detech[®]-m was significantly more effective than SilicoSec[®] during the early exposure period (Days 1-3). However, by Days 5 and 6, all DE formulations achieved 100% mortality.

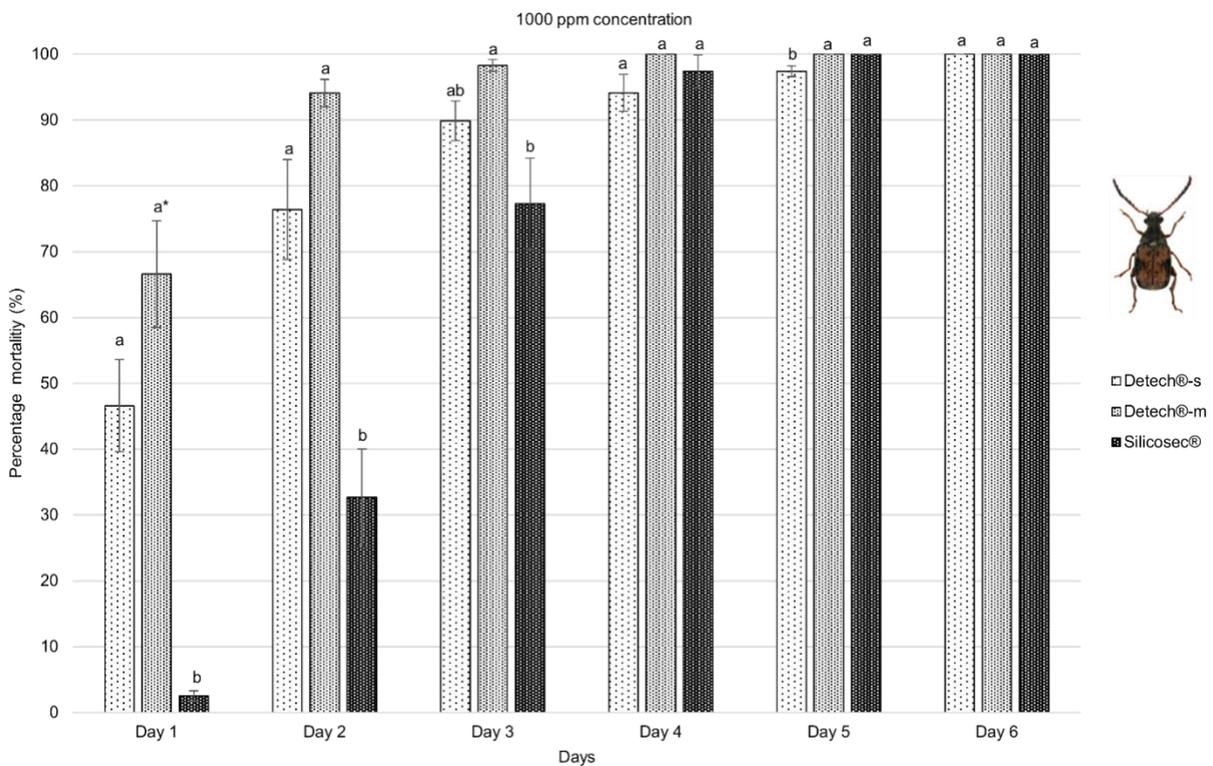


Figure 5. Mean percentage mortality (\pm standard error)* of *Callosobruchus maculatus* adults exposed to chickpeas treated with a water suspension of three diatomaceous earth (DE) formulations (Detech[®]-s, Detech[®]-m, and SilicoSec[®]) at 1000 ppm over a 6-day period.

* A one-way analysis of variance (ANOVA) was conducted, and differences between means were determined using Tukey's HSD test at a 5% significance level. Bars with different letters indicate statistically significant differences and each day is compared within each day.

Lethal time values

The lethal times (LT₅₀ and LT₉₉) and probit analysis parameters for three DE formulations—Detech[®]-s, Detech[®]-m, and SilicoSec[®]—when applied to *C. maculatus* adults at 500 ppm are presented in Table 4. In all formulations, the results of the probit analysis indicate that the χ^2 tests demonstrated a good fit to the data ($p > 0.05$). There were statistically significant differences in the exposure times required to kill 50% (LT₅₀) and 99% (LT₉₉) of *C. maculatus* adults on chickpeas treated with water suspensions of the three DE formulations. Among them, Detech[®]-m exhibited the fastest action, with the lowest LT₅₀ (36.55 h) and LT₉₉

(105.22 h). Importantly, its confidence intervals (CIs) did not overlap with those of Detech[®]-s (LT₅₀=72.80 h, LT₉₉=154.66 h) or SilicoSec[®] (LT₅₀=67.31 h, LT₉₉=122.73 h), confirming the statistical significance of the differences. These findings indicate that Detech[®]-m acts significantly faster than the other formulations, while Detech[®]-s is the slowest, and SilicoSec[®] exhibits an intermediate rate of mortality progression. Furthermore, SilicoSec[®] demonstrated the best probit model fit ($\chi^2=24.26$, $p < 0.05$, $H=1.10$), indicating higher reliability in its mortality response predictions. While Detech[®]-m showed moderate variability ($\chi^2=37.19$, $p < 0.05$, $H=1.49$), its model still provided a good fit. Regarding the rate of mortality progression, SilicoSec[®] had the steepest slope, meaning its mortality rate increased more sharply after exposure. In contrast, Detech[®]-m had a moderate slope, aligning with its faster LT₅₀ compared to SilicoSec[®]. These results highlight the distinct modes of action of the tested DE formulations, with Detech[®]-m being the most rapid, SilicoSec[®] acting moderately fast, and Detech[®]-s being the slowest.

Table 4. Lethal time (LT₅₀ and LT₉₉) values and probit analysis parameters for *Callosobruchus maculatus* adults exposed to a water suspension of three diatomaceous earth formulations (Detech[®]-s, Detech[®]-m, and SilicoSec[®]) at 500 ppm

DE formulation	n ^a	Slope±SE	Intercept	t value	LT ₅₀ (hour) (Lower-upper confidence interval) ^b	LT ₉₉ (hour) (Lower-upper confidence interval) ^b	χ^2 ^c (df) ^d p value	H ^e
Detech [®] -s	840	0.028±0.002	-2.069	16.934	72.80 (67.40 - 77.96)	154.66 (143.61- 169.22)	39.47 (26) $p < 0.05$	1.52
Detech [®] -m	840	0.034±0.003	-1.239	11.363	36.55 (29.00 - 42.44)	105.22 (94.50 - 121.33)	37.19 (25) $p < 0.05$	1.49
SilicoSec [®]	840	0.042±0.003	-2.826	15.397	67.31 (63.67 - 70.88)	122.73 (115.30 - 132.35)	24.26 (22) $p < 0.05$	1.10

^a: Total number of individuals tested, SE: Standart error ^b: Lower-upper confidence interval (at 5% significance level), ^c: Chi-square value, ^d: Degree of freedom, ^e: Heterogeneity value.

The lethal times (LT₅₀ and LT₉₉) and probit analysis parameters for three DE formulations—Detech[®]-s, Detech[®]-m, and SilicoSec[®]—when applied to *C. maculatus* adults at 1000 ppm are presented in Table 5. There were statistically significant differences in the exposure times required to kill 50% (LT₅₀) and 99% (LT₉₉) of *C. maculatus* on chickpeas treated with water suspensions of the three DE formulations. Among them, Detech[®]-m exhibited the fastest action, with the lowest LT₅₀ (12.42 h) and LT₉₉ (70.65 h). Importantly, its confidence intervals (CIs) did not overlap with those of SilicoSec[®] (LT₅₀=57.53 h, LT₉₉=101.02 h) or Detech[®]-s (LT₅₀=22.59 h, LT₉₉=123.50 h), except for a partial overlap in LT₅₀ CIs between Detech[®]-s and Detech[®]-m. This confirms that the difference in LT₉₉ values is statistically significant, while the LT₅₀ difference between Detech[®]-s and Detech[®]-m requires careful interpretation due to overlapping confidence intervals. These findings indicate that Detech[®]-m acts significantly faster than the other formulations, while SilicoSec[®] is the slowest, and Detech[®]-s exhibits an intermediate rate of mortality progression based on LT₅₀ values. Furthermore, Detech[®]-m demonstrated the best probit model fit ($\chi^2=18.73$, $p < 0.05$, $H=0.85$), indicating higher reliability in its mortality response predictions. In contrast, SilicoSec[®] showed high variability ($\chi^2=32.16$, $p > 0.05$, $H=3.43$), suggesting a less consistent mortality response. Regarding mortality progression rates, SilicoSec[®] had the steepest slope, meaning its mortality rate increased more sharply after exposure. In contrast, Detech[®]-m had a moderate slope, aligning with its faster LT₅₀ compared to SilicoSec[®]. These results highlight the distinct modes of action of the tested DE formulations, with Detech[®]-m being the most rapid, Detech[®]-s acting moderately fast, and SilicoSec[®] being the slowest.

Table 5. Lethal time (LT₅₀ and LT₉₉) values and probit analysis parameters for *Callosobruchus maculatus* adults exposed to a water suspension of three diatomaceous earth formulations (Detech[®]-s, Detech[®]-m, and SilicoSec[®]) at 1000 ppm

DE formulation	n ^a	Slope±SE	intercept	t value	LT ₅₀ (hour) (Lower-upper confidence interval) ^b	LT ₉₉ (hour) (Lower-upper confidence interval) ^b	χ ^{2c} (df) ^d p value	H ^e
Detech [®] -s	720	0.023±0.002	-0.521	10.391	22.59 (8.82 - 32.03)	123.50 (108.20 - 147.98)	34.69 (22) p< 0.05	1.58
Detech [®] -m	720	0.040±0.006	-0.496	6.734	12.42 (1.65 - 18.99)	70.65 (61.67 - 86.21)	18.73 (22) p< 0.05	0.85
SilicoSec [®]	720	0.053±0.004	-3.066	13.820	57.53 (53.34 - 61.65)	101.18 (93.03 - 113.02)	32.16 (18) p > 0.05	3.43

^a: Total number of individuals tested, SE: Standart error ^b: Lower-upper confidence interval (at 5% significance level), ^c: Chi-square value, ^d: Degree of freedom, ^e: Heterogeneity value.

Progeny production

Dunnnett's test showed that *C. maculatus* progeny production in the control group was significantly higher than in all DE-treated chickpea formulations at 500 ppm (Table 6). Among the treatments, Detech[®]-m had the lowest adult progeny emergence (17.6), indicating the highest suppression of reproduction. In contrast, Detech[®]-s was the least effective, with the highest progeny emergence (181.7), though still significantly lower than the control. Progeny emergence in SilicoSec[®] (109.7) was higher than in Detech[®]-m but lower than in Detech[®]-s, confirming its intermediate effectiveness. Detech[®]-m provided the highest suppression rate (96.7%), significantly reducing progeny production. Meanwhile, Detech[®]-s was the least effective (64.9% suppression), allowing significantly more progeny to emerge. SilicoSec[®] showed a suppression rate of 78.8%, which was significantly higher than Detech[®]-s but lower than Detech[®]-m ($F_{2,9}=49.72$, $p < 0.0001$).

Table 6. Mean number of progeny emergence (± S.E.) and percentage reduction in progeny production for *Callosobruchus maculatus* 45 days after the removal of parental adults from chickpea grains treated with 500 ppm of three diatomaceous earth (DE) formulations (Detech[®]-s, Detech[®]-m, and SilicoSec[®])

F ₁ progeny emergence <i>Callosobruchus maculatus</i> (500 ppm)*			
DE Formulation	Number of progeny emergence±S.E	p value***	Percentage reduction in progeny production (%)±S. E.
Detech [®] -s	181.7±18.7 (518.7±39.3)**	0.0001	64.9±3.6 C
Detech [®] -m	17.6±3.6 (518.7±39.3)	0.0001	96.7±0.7 A
SilicoSec [®]	109.7±6.8 (518.7±39.3)	0.0001	78.8±1.3 B
F and p value*		-	F _{2,9}=49.72 p< 0.0001}

* A one-way analysis of variance (ANOVA) was conducted, and differences between means were determined using Tukey's HSD test at a 5% significance level. Different letters indicate statistically significant differences.

** The values in parentheses represent the average number of new-generation adults obtained from the control treatment.

*** Dunnnett's test was used to compare the number of new-generation adults from the biological tests with the control group's average.

According to Dunnnett's test, *C. maculatus* progeny production in the control group was significantly higher than in all DE-treated chickpea formulations at 1000 ppm (Table 7). Among the treatments, Detech[®]-m had the lowest adult progeny emergence (9.5), indicating the highest suppression of reproduction. In contrast, Detech[®]-s was the least effective, with the highest progeny emergence (107), though still significantly lower than the control. Progeny emergence in SilicoSec[®] (87.5) was higher than in Detech[®]-m but lower than in Detech[®]-s, confirming its intermediate effectiveness. Detech[®]-m provided the highest suppression rate (98.1%), significantly reducing progeny production. Meanwhile, Detech[®]-s was the least effective (79.3% suppression), allowing significantly more progeny to emerge. SilicoSec[®] showed a suppression rate of 83.1%, which was statistically in the same group as Detech[®]-s but significantly lower than Detech[®]-m ($F_{2,9}=8.46$, $p=0.009$).

Table 7. Mean number of progeny emergence (\pm S.E.) and percentage reduction in progeny production for *Callosobruchus maculatus* 45 days after the removal of parental adults from chickpea grains treated with 1000 ppm of three diatomaceous earth (DE) formulations (Detech[®]-s, Detech[®]-m, and SilicoSec[®])

F ₁ progeny emergence <i>Callosobruchus maculatus</i> (1000 ppm)*			
DE formulation	Number of adult emergence \pm S. E	<i>p</i> value***	Percentage reduction in progeny production (%) \pm S. E
Detech [®] -s	107 \pm 30.4 (518.7 \pm 39.3)**	0.000	79.3 \pm 5.8 B
Detech [®] -m	9.5 \pm 3.5 (518.7 \pm 39.3)	0.000	98.1 \pm 0.7 A
SilicoSec [®]	87.5 \pm 1.8 (518.7 \pm 39.3)	0.000	83.1 \pm 0.3 B
<i>F</i> and <i>p</i> value*	-	-	<i>F</i> _{2,9} =8.46 <i>p</i> =0.009

* A one-way analysis of variance (ANOVA) was conducted, and differences between means were determined using Tukey's HSD test at a 5% significance level. Different letters indicate statistically significant differences.

** The values in parentheses represent the average number of new-generation adults obtained from the control treatment.

*** Dunnett's test was used to compare the number of new-generation adults from the biological tests with the control group's average.

Discussion

The mode of application—water suspension (slurry) vs. dry dust—significantly affects the effectiveness of diatomaceous earth (DE) formulations. Dry DE relies on direct contact with insect exoskeletons, where its fine abrasive particles attach to the cuticle, absorb lipids, and cause dehydration (Athanassiou et al., 2003). However, dust applications present challenges such as displacement, reduced adhesion to grains, and inhalation risks for workers (Fields & Korunić, 2000). In contrast, slurry applications involve suspending DE in water and spraying it onto stored products. As the water evaporates, a uniform DE layer remains on the surface, ensuring better adhesion to grains and insect bodies (Ertürk et al., 2020). This method minimizes dust-related health concerns and may enhance insecticidal activity by providing broader coverage and longer persistence (Stathers et al., 2004). In this study, the water suspension of Detech[®]-m at 1000 ppm resulted in complete mortality of *C. maculatus* by the fourth day and reduced progeny production by 98.1%, indicating near-total reproductive inhibition. A previous study reported that dry application of Detech[®] at 1000 ppm achieved 100% mortality by the fifth day and reduced progeny production by 88.7% (Sağlam et al., 2022b). The efficacy of Detech[®] and SilicoSec[®] in controlling *C. maculatus* varied depending on whether they were applied as dry powder or water suspension. Similarly, in the same study, dry SilicoSec[®] at 1000 ppm achieved full mortality by day five, but at 500 ppm, mortality was only 77% after seven days (Sağlam et al., 2022a). In the present study, water-suspension application of SilicoSec[®] at 500 ppm required six days to reach complete mortality, while at 1000 ppm, full mortality was achieved in a shorter time. With dry application, SilicoSec[®] at 500 ppm reduced progeny production by 48.7%, whereas at 1000 ppm and above, suppression exceeded 80.6%. However, in the water-suspension treatment, SilicoSec[®] at 500 ppm provided 78.8% progeny suppression, and at 1000 ppm, it achieved 83.1%. Overall, comparing dry and water-suspension applications suggests that water suspensions result in higher mortality, particularly at lower concentrations, and require slightly less exposure time. Additionally, water suspensions provide better progeny suppression at lower concentrations, likely due to more uniform grain coverage. This application method improves coverage and adhesion to grain surfaces, reducing dust displacement and increasing persistence (Korunić, 1998; Stathers et al., 2004). Our findings align with previous research demonstrating that slurry-applied DE formulations prolong insecticidal activity due to better adhesion to treated surfaces (Fields & Korunić, 2000).

This study also examined the relationship between the chemical and physical properties of different diatomaceous earth (DE) formulations and their effects on adult mortality, lethal time (LT₉₉), and progeny reduction of *C. maculatus*. The primary component of DE is silicon dioxide (SiO₂), which constitutes a significant portion of its composition. Among the formulations analyzed, SilicoSec[®] contains 85.7% SiO₂,

while Detech® contains 80,6% SiO₂. The higher SiO₂ content in SilicoSec® suggests a greater abundance of diatom frustules, which are critical for the abrasive action that causes insect desiccation. Studies indicate that the insecticidal properties of DE are influenced by its chemical composition. Formulations with higher SiO₂ concentrations have been associated with greater efficacy against pests such as *C. maculatus* (Korunić, 1998), as well as other stored-product insects, including the rice weevil, *Sitophilus oryzae* (L., 1763) and the red flour beetle, *Tribolium castaneum* (Herbst, 1797) (Athanassiou et al., 2005). However, DE efficacy is not determined solely by SiO₂ concentration. A study by Arnaud et al. (2005) assessed the insecticidal efficacy of various DE formulations with differing oxide compositions, including SiO₂, Al₂O₃, Fe₂O₃, CaO, and MgO. The findings revealed that higher SiO₂ content did not always correlate with increased insecticidal activity. Instead, the presence and proportions of other oxides, such as Al₂O₃ and Fe₂O₃, influenced the hydrophilic properties and abrasiveness of DE particles, ultimately affecting their overall efficacy against stored-product insects. These results suggest that insecticidal performance is a multifaceted trait influenced by the complete chemical composition of DE, rather than SiO₂ concentration alone.

The physical attributes of DE, particularly particle size distribution and specific surface area (SSA), play a crucial role in its effectiveness as an insecticide (Korunić, 1997; Robinson, 2005; Vayias et al., 2009; Ziaee et al., 2013; Henteş & Işıkber, 2024). Detech®-m, with a median particle size (d₅₀) of approximately 7.34 µm and a specific surface area of 60.45 m²/g, exhibited superior insecticidal performance. Several studies have also demonstrated that smaller DE particles enhance insecticidal activity. For instance, Vayias et al. (2009) found that DE formulations with particles smaller than 45 µm were more effective against *Rhizopertha dominica* (Fabricius, 1792) (Coleoptera: Bostrichidae), *Sitophilus oryzae* A.Hustache, 1930 (Coleoptera: Dryophthoridae), and *Cryptolestes ferrugineus* (Stephens, 1831) (Coleoptera: Laemophloeidae) compared to formulations with larger particles. The increased efficacy of finer particles is attributed to their greater adherence to the insect cuticle, leading to enhanced desiccation and higher mortality rates. However, particle size alone does not solely determine efficacy. Baliota & Athanassiou (2020) reported that particle shape also plays a crucial role, with certain shapes enhancing DE's insecticidal properties. Their research suggests that while smaller particles tend to be more effective, overall shape and structure are also critical factors influencing DE's performance as an insecticide. Moreover, a higher specific surface area (SSA) provides a greater interaction area between DE particles and insect exoskeletons. This increased contact enhances the abrasive action of DE, effectively disrupting the insect's protective waxy cuticle and accelerating desiccation. In contrast, Detech®-s, with a coarser particle size (d₅₀=24.89 µm) and a lower specific surface area (40.35 m²/g), was less effective. The larger particle size and reduced surface area likely resulted in diminished contact with the insect cuticle, reducing desiccation efficiency. This finding aligns with previous research, which indicates that finer DE particles with higher surface areas are more effective for stored-product insect pest control (Athanassiou et al., 2004).

The efficacy of DE formulations is often assessed by measuring the time required to achieve a specific mortality rate, such as LT₉₉ (the time needed to kill 99% of the target population). Unlike many other stored-product insect pests, adult *C. maculatus* have a short lifespan, typically surviving only 5 to 10 days in stored pulses under tropical conditions (Rees, 2004). During this period, they do not feed or directly damage stored grains; instead, their primary function is egg-laying. Fast-acting DE formulations with high SSA and optimal particle size can kill adults before oviposition, preventing infestation without chemical pesticides (Arthur, 2000). The key objective is to minimize LT₅₀ and LT₉₉ values, ensuring *C. maculatus* adults perish before significant egg-laying occurs. In this study, at 500 ppm, Detech®-m exhibited a significantly lower LT₉₉ (105.22 hours) than SilicoSec® (122.73 hours) and Detech®-s (154.66 hours). At 1000 ppm, the same trend persisted, with Detech®-m (70.65 hours) acting more rapidly than Detech®-s (123.50 hours) and SilicoSec® (101.18 hours). These results confirm that Detech®-m provides faster and more effective mortality against *C. maculatus* adults due to its fine particle size, higher BET surface area, and superior adsorption properties. Studies have shown that DE products with enhanced SSA and finer particles induce higher mortality in stored-product pests like *R. dominica* and *C. maculatus* within shorter time frames than

formulations with lower SSA (Athanassiou et al., 2005; Kabir & Wulgo, 2014). Although SilicoSec[®] contains a higher SiO₂ content, it exhibited a longer LT₉₉ (101.18 hours) under similar conditions. This disparity may be due to its broader particle size distribution and lower BET surface area, which could reduce consistent contact with the insect cuticle. These findings highlight the importance of optimizing physical properties, such as SSA, particle size and distribution, to enhance the insecticidal efficiency of DE formulations.

Beyond immediate adult mortality, an effective DE formulation should also disrupt the reproductive cycle of pests to prevent population resurgence (Arthur, 2000; Subramanyam & Roesli, 2000; Athanassiou et al., 2005). Detech[®]-m exhibited a substantial reduction in progeny production, with a suppression rate of 98.1% at 1000 ppm. This result suggests that exposure to Detech[®]-m not only causes high adult mortality but also adversely affects the reproductive capabilities of surviving individuals, possibly by interfering with oviposition or egg viability. In contrast, Detech[®]-s, with its coarser particles and lower surface area, achieved a lower progeny suppression rate (79.3%) under the same conditions. The reduced efficacy in progeny reduction may result from insufficient disruption of the insect's protective cuticular layer, allowing some individuals to survive and reproduce. These findings align with Stathers et al. (2004), who reported that DE treatments significantly reduce oviposition and inhibit larval development in *C. maculatus*. Furthermore, DE water-suspension applications may enhance efficacy by ensuring more uniform grain coverage, preventing larvae from burrowing and developing (Athanassiou et al., 2003).

Other physical characteristics, such as oil and water absorption capacities, pH, and compressed density, can influence the performance of DE formulations (Korunić, 1997 & 1998; Losic & Korunić, 2017). SilicoSec[®] exhibited the highest oil absorption (173.6 g/mL) and water absorption (147 g/mL), which may enhance its desiccating effect on insects. However, its lower uniformity value (0.53) suggests a narrower particle size distribution, which might limit its overall efficacy due to reduced coverage and contact with the insect cuticle. The pH of DE formulations can also affect their insecticidal activity. Detech[®]-s had the highest pH value (8.66), making it more alkaline compared to Detech[®]-m (7.88) and SilicoSec[®] (7.72). The pH of DE may influence its interaction with insect cuticles, as a neutral to slightly alkaline pH is considered optimal for maintaining stability, dispersion, and effectiveness of DE particles (Athanassiou et al., 2005). However, the direct impact of DE pH on insect mortality remains unclear and requires further investigation.

Conclusion

This study clearly demonstrates that water-suspension (slurry) application significantly enhances the insecticidal efficacy of diatomaceous earth (DE) formulations against *C. maculatus* in stored chickpeas compared to dry dust applications. Slurry treatments improved DE adhesion, reduced dust-related health risks, and achieved higher mortality and progeny suppression at lower concentrations. Among the evaluated formulations, Detech[®]-m exhibited the highest effectiveness, achieving 99% adult mortality and 98.1% progeny suppression, attributable to its fine particle size (7.34 µm) and elevated specific surface area (60.45 m²/g), which enhanced cuticular abrasion and desiccation. Although SilicoSec[®] contained a higher SiO₂ content, its broader particle size distribution and lower BET surface area diminished its performance relative to Detech[®]-m. These findings reinforce that the physical characteristics of DE, particularly particle size and surface area, are more critical determinants of efficacy than chemical composition alone. Moreover, slurry applications further augmented these properties by ensuring more uniform coverage and prolonged persistence on treated grains.

The results advocate for refining DE formulations by targeting their physicochemical attributes to maximize efficacy and minimize health risks. Water-suspension methods emerge as a promising strategy to develop sustainable, non-chemical pest management practices for stored legumes. Future research should investigate the interaction between DE formulations and storage environment variables, such as humidity and temperature, to optimize their practical application under diverse conditions. The strategic optimization of diatomaceous earth formulations and application techniques thus represents a pivotal advancement toward sustainable, non-chemical preservation of global food security.

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References

- Abbott, W. S., 1925. A method of computing the effectiveness of an insecticide. *Journal of Economic Entomology*, 18 (28): 65-267.
- Agrafioti, P., G. C. Athanassiou, T. M. Vassilakos, G. Viontzos & F. H. Arthur, 2015. Using a lethality index to assess susceptibility of *Tribolium confusum* and *Oryzaephilus surinamensis* to insecticides. *PLoS One*, 10: e0142044 (1-19).
- Alkan, M., T. Atay, S. Ertürk & İ. Kepenekci, 2019. Comparison of bioactivities of native diatomaceous earth against Turkestan cockroach [*Blatta lateralis* Walker (Blattodea: Blattidae)] nymphs. *Applied Ecology and Environmental Research*, 17 (3): 5987-5994.
- Arnaud, L., S. Navarro & F. Fleurat-Lessard, 2005. Efficacy of diatomaceous earth formulations admixed with grain against populations of *Sitophilus oryzae* (L.) (Coleoptera: Curculionidae) and *Tribolium castaneum* (Herbst) (Coleoptera: Tenebrionidae) in different storage conditions. *Journal of Stored Products Research*, 41 (2): 121-130.
- Arthur, F. H., 1996. Grain protectants: Current status and prospects for the future. *Journal of Stored Products Research*, 32 (4): 293-302.
- Arthur, F. H., 2000. Toxicity of diatomaceous earth to red flour beetles and confused flour beetles: effects of temperature and relative humidity. *Journal of Economic Entomology*, 93 (2): 526-532.
- ASTM, 2012. ASTM D1483 - 12 Standard Test Method for Oil Absorption of Pigments by Gardner Coleman Method. *Annual Book of ASTM Standards*, Vol. 06.03 ASTM International, West Conshohocken, PA., 1104 pp.
- Athanassiou, C. G. & Z. Korunić, 2007c. Evaluation of two new diatomaceous earth formulations, enhanced with abamectin and bitterbarkomycin, against four stored-grain beetle species. *Journal of Stored Products Research*, 43 (4): 468-473.
- Athanassiou, C. G., N. G. Kavallieratos & C. M. Meletsis, 2005. Insecticidal effect of three diatomaceous earth formulations against adults of *Sitophilus oryzae* and *Tribolium confusum* on stored wheat and maize. *Journal of Stored Products Research*, 41 (1): 57-66.
- Athanassiou, C. G., N. G. Kavallieratos & C. M. Meletsis, 2007a. Insecticidal effect of three diatomaceous earth formulations, applied alone or in combination, against three stored-product beetle species on wheat and maize. *Journal of Stored Products Research*, 43 (4): 330-334.
- Athanassiou, C. G., N. G. Kavallieratos & N. S. Andris 2004. Insecticidal effect of three diatomaceous earth formulations against adults of *Sitophilus oryzae* (Coleoptera: Curculionidae) and *Tribolium confusum* (Coleoptera: Tenebrionidae) on oat, rye and triticale. *Journal of Economic Entomology* 97 (6): 2160-2167
- Athanassiou, C. G., N. G. Kavallieratos & Z. Korunić, 2007b. Evaluation of diatomaceous earth formulations against stored-grain insects: Effect of dose rate, temperature, and relative humidity. *Journal of Economic Entomology*, 100 (2): 599-603.
- Athanassiou, C. G., N. G. Kavallieratos, B. J. Vayias, Z. Tomanović, A. Petrović, V. Rozman, C. Adler, Z. Korunić & D. Milovanović, 2011. Laboratory evaluation of diatomaceous earth deposits mined from several locations in central and southeastern Europe as potential protectants against coleopteran grain pests. *Crop Protection*, 30 (3): 329-339.
- Athanassiou, C. G., N. G. Kavallieratos, F. C. Tsaganou, B. J. Vayias, C. B. Dimizas & C. T. Buchelos, 2003. Effect of grain type on the insecticidal efficacy of SilicoSec against *Sitophilus oryzae* (L.) (Coleoptera: Curculionidae). *Crop Protection*, 22 (10): 1141-1147.

- Athanassiou, C. G., N. G. Kavallieratos, J. B. Tsakiri, S. N. Xyrafidis & B. J. Vayias, 2006. Effect of temperature and humidity on insecticidal effect of SilicoSec against *Ephesia kuehniella* (Lepidoptera: Pyralidae) larvae. *Journal of Economic Entomology*, 99 (4): 1520-1524.
- Baliota, G. V. & C. G. Athanassiou, 2020. Evaluation of a Greek diatomaceous earth for stored product insect control and techniques that maximize its insecticidal efficacy. *Applied Sciences*, 10 (18): 6441.
- Bridgeman, B. W. & P. J. Collins, 1994. "Integrated Pest management in the Grainco, Queensland, Australia, Storage System, 910-914". In: *Proceedings 6th International Conference on Stored-Product Protection (17-23 April 1994, Canberra, Australia)*, 1274 pp.
- Collins, D. A. & D. A. Cook, 2006a. Laboratory studies evaluating the efficacy of diatomaceous earths, on treated surfaces, against stored-product insect and mite pests. *Journal of Stored Products Research*, 42 (1): 51-60.
- Collins, D. A. & D. A. Cook, 2006b. The efficacy of applied diatomaceous earths for the control of stored-product insect pests in dry food processing environments. *Journal of Stored Products Research*, 42 (2): 197-206.
- Doğanay, Ş. İ., 2013. Determination of Efficiency of some Diatomaceous Earths against to Stored-Grain Insects, *Sitophilus granarius* (L.) and *Rhyzopertha dominica* (F.). Kahramanmaraş Sütçü İmam University, Institute of Natural and Applied Science, Plant Protection Department (Unpublished) Master Thesis, 55 pp (In Turkish with abstract in English).
- Ebeling, W., 1971. Sorptive dusts for pest control. *Annual Review of Entomology*, 16 (1): 123-158.
- Ertürk, S., T. Atay, U. Toprak & M. Alkan, 2020. The efficacy of different surface applications of wettable powder formulation of Detech® diatomaceous earth against the rice weevil, *Sitophilus oryzae* (L.) (Coleoptera: Curculionidae). *Journal of Stored Products Research*, 89 (2020): 101725.
- Fields, P. G. & Z. Korunić, 2000. The effect of grain moisture content and temperature on the efficacy of diatomaceous earths from different geographical locations against stored-product beetles. *Journal of Stored Products Research*, 36 (1): 1-13.
- Henteş, S. & A. A. Işıkber, 2024. Insecticidal efficacy of local diatomaceous earth compositions with different particle sizes against stored grain pests. *Turkish Journal of Entomology*, 48 (3): 353-365.
- Israelachvili, J. N., 2011. *Intermolecular and Surface Forces*. 3rd Edition. Academic, Burlington, MA. 676 pp.
- Johnson, J. A., K. A. Valero & S. Wang, 2014. Physical properties of stored grains and insect pests in relation to radio frequency and microwave treatments. *Applied Engineering in Agriculture*, 30 (6): 949-957.
- Kabir, B. G. J. & M. A. Wulgo, 2014. "Efficacy of four diatomaceous earth formulations against *Callosobruchus maculatus* (F.) (Coleoptera: Bruchidae) on cowpea, 798-806". In: *Proceedings 11th International Working Conference on Stored-Product Protection (24-28 November 2014, Chiang Mai, Thailand)*, 1034 pp.
- Kılıç, N., 2022. Efficacy of dust and wettable powder formulation of diatomaceous earth (Detech®) in the control of *Tyrophagus putrescentiae* (Schrank) (Acari: Acaridae). *Insects*, 13 (10): 857.
- Kljajic, P. & I. Peric, 2007. Effectiveness of wheat-applied contact insecticides against *Sitophilus granarius* (L.) originating from different populations. *Journal of Stored Products Research*, 43 (4): 523-529.
- Korunić, Z., 1997. Rapid assessment of the insecticidal value of diatomaceous earths without conducting bioassays. *Journal of Stored Products Research*, 33 (3): 219-229.
- Korunić, Z., 1998. Diatomaceous earth a group of natural insecticides. *Journal of Stored Products Research*, 34 (2-3): 87-97.
- Korunić, Z., 2013. Diatomaceous earths - Natural Insecticides. *Pesticides & Phytomedicine (Belgrade)*, 28 (2): 77-95.
- Korunić, Z., C. G. Athanassiou, & F. H. Arthur, 2021. Diatomaceous earth for arthropod pest control: Back to the future. *Insects*, 12 (10): 839.
- LeOra Software, 1994. *POLO-PC: A User's Guide to Probit or Logit Analysis*. LeOra Software, Berkeley, CA., 28 pp.
- Lorini, I., H. Beckel & C. Schlemper, 2007. Chemical control of stored grain pests and the associated resistance problems in Brazil. *Journal of Stored Products Research*, 43 (3): 299-306.
- Losic, D. & Z. Korunić, 2017. "Diatomaceous Earth, a Natural Insecticide for Stored Grain Protection: Recent Progress and Perspectives, 219-247". In: *Diatom Nanotechnology: Progress and Emerging Applications* (Ed. D. Losic). Royal Society of Chemistry Publishing, Cambridge, UK, 270 pp.

- McLaughlin, A., 1994. "Laboratory trials on desiccant dust insecticides, 638-645". In: Proceedings 6th International Conference on Stored-Product Protection, (17-23 April 1994, Canberra, Australia), 1274 pp.
- Mewis, I. & C. Ulrichs, 2001. Action of amorphous diatomaceous earth against different stages of the stored product pests *Tribolium confusum*, *Tenebrio molitor*, *Sitophilus granarius* and *Plodia interpunctella*. Journal of Stored Products Research, 37 (2): 153-164.
- Minitab, LLC, 2021. Minitab 21 Statistical Software Inc. USA. (Web page: <https://www.minitab.com/en-us/products/minitab/>) (Date accessed: March 2025).
- Mutlu, Ç., A. Öğreten, C. Kaya & M. Mamay, 2019. Influence of different grain storage types on Khapra beetle, *Trogoderma granarium* Everts, 1898 (Coleoptera: Dermestidae), infestation in southeastern Anatolia (Turkey) and its resistance to malathion and deltamethrin. Turkish Journal of Entomology, 43 (2): 131-142.
- Oğreten, A., S. Eren, C. Kaya, Mutlu, T. Ayaz, A. R. Z. Gaafar & R. M. Mahmoud, 2023. Insecticidal efficacy of native raw and commercial diatomaceous earths against *Tribolium confusum* du Val (Coleoptera: Tenebrionidae) under different environmental conditions. Journal of King Saud University-Science, 35 (7): 102827.
- Öztekin, N. & Ç. Mutlu, 2020. Efficacy of three diatomaceous earth formulations against cowpea beetle, *Callosobruchus maculatus* (F.) (Coleoptera: Bruchidae), on bean. Journal of Entomology and Zoology Studies, 8 (6): 147-52.
- Phillips, T. W. & J. E. Throne, 2010. Biorational approaches to managing stored-product insects. Annual Review of Entomology, 55 (1): 375-397.
- Rees, D., 2004. Insects of Stored Products. CSIRO Publishing, Collingwood, Australia, 80 pp.
- Robinson, W. H., 2005. Handbook of Urban Insects and Arachnids: A Handbook of Urban Entomology Cambridge University Press, New York, USA, 472 pp.
- Rumbos, C. I., A. C. Dutton, N. G. Tsiropoulos & C. G. Athanassiou, 2018. Persistence and residual toxicity of two pirimiphos-methyl formulations on wheat against three stored-product pests. Journal of Stored Products Research, 76 (2018): 14-21.
- Sağlam Ö., H. Bozkurt, R. Şen, S. Henteş & A. A. Işıkber 2022b. Insecticidal efficacy of Turkish novel diatomaceous earth formulations against Cowpea weevil *Callosobruchus maculatus* (F.) (Coleoptera: Chrysomelidae: Bruchinae) on chickpea, Fresenius Environmental Bulletin, 31: 5839-5849 pp.
- Sağlam, Ö., A. A. Işıkber, H. Tunaz, M. K. Er, F. Bahadır & R. Şen, 2017. Preliminary checking of some Turkish diatomaceous earth similarities with commercial diatomaceous earths under Scanning Electron Microscope (SEM). Journal of Tekirdag Agricultural Faculty, Special Issue: 13-19.
- Sağlam, Ö., A. Bayram, A. A. Işıkber, R. Şen, H. Bozkurt & S. Henteş, 2022a. Insecticidal and repellency effects of a Turkish diatomaceous earth formulation (Detech®) on adults of three important pests of stored grain. Turkish Journal of Entomology, 46 (1): 75-88.
- Şen, R., A. A. Işıkber, H. Bozkurt & Ö. Sağlam, 2019. Effect of temperature on insecticidal efficiency of local diatomaceous earth against stored-grain insects. Turkish Journal of Entomology, 43 (4): 441-450.
- Stathers, T. E., M. Dennif & P. Golob, 2004. The efficacy and persistence of diatomaceous earths admixed with commodity against four tropical stored product beetle pests. Journal of Stored Products Research, 40 (1): 113-123.
- Subramanyam, Bh. & R. Roesli 2000. "Inert Dusts, 321-379". In: Alternatives to Pesticides in Stored-Product IPM (Eds. B. Subramanyam & D. W. Hagstrum). Kluwer Academic Publishers, Boston, MA, 447 pp.
- Subramanyam, Bh., C. L. Swanson, N. Madamanchi & S. Norwood, 1994. "Effectiveness of Insecto, a new diatomaceous earth formulation in suppressing several stored-grain insect species. 650-659". In: Proceedings of the 6th International Working Conference on Stored-Product Protection. (17-23 April 1994, Canberra, Australia) (Eds. E. Highley, E. J. Wright, H. J. Banks & B. R. Champ), CAB International, Oxford, UK, 1274 pp
- Subramanyam, Bh., R. L. Swanson, A. V. Barak & J. C. Schmidt, 1994. Effectiveness of a sorptive dust against three stored-product beetles. Journal of Economic Entomology, 87 (3): 689-694.
- Vassilakos, N. T., G. C. Athanassiou & G. N. Tsiropoulos, 2015. Influence of grain type on the efficacy of spinetoram for the control of *Rhyzopertha dominica*, *Sitophilus granarius* and *Sitophilus oryzae*. Journal Stored Products Research, 64 (2015): 1-7.

- Vayias, B. J., C. G. Athanassiou, Z. Korunić & V. Rozman, 2009. Evaluation of natural diatomaceous earth deposits from south-eastern Europe for stored-grain protection: The effect of particle size. *Pest Management Science*, 65 (10): 1118-1123.
- Wakil, W., M. Ashfaq, A. Shabbir, A. Javed & M. Sagheer, 2006. Efficacy of diatomaceous earth (Protect-it) as a protectant of stored wheat against *Rhyzopertha dominica* (F.) (Coleoptera: Bostrichidae). *Pakistan Entomologist*, 28 (2): 19-24.
- Ziaee, M., S. Moharrampour & K. Dadkhalipour, 2013. Effect of particle size of two Iranian diatomaceous earth deposits and a commercial product on *Sitophilus granarius* (Col.: Dryophthoridae). *Journal of Entomological Society of Iran*, 33 (2): 9-17.