Efficacy of filter cake and Triplex against stored- product insects on concrete surfaces and grain: Safer alternatives to protect stored grain of Ethiopian smallholder farmers

by

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B.S., Addis Ababa University, 2005

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AN ABSTRACT OF A DISSERTATION

Submitted in partial fulfillment of the requirements for the degree

DOCTOR OF PHILOSOPHY

Department of Grain Science and Industry

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Abstract

Filter cake and Triplex are powdered by-products of aluminum sulfate and soap factories, respectively. Studies were designed to determine elemental composition of these two powders and evaluate the efficacy against stored product insect species on concrete surfaces and commodities. Elemental composition of the powders was determined using conjugated scanning electron microscopy and Energy-dispersive X-ray spectroscopy. No heavy metals were found in both powders, and the dominant elements found were silicon and oxygen in the form of silicon dioxide. The efficacy of filter cake and Triplex against the maize weevil, *Sitophilus zeamais* Motschulsky; rice weevil, *Sitophilus oryzae* (Linnaeus); lesser grain borer, *Rhyzopertha dominica* Fabricius; red flour beetle, *Tribolium castaneum* (Herbst); saw-toothed grain beetle, *Oryzaephilus surinamensis* (Linnaeus); and Indian meal moth, *Plodia interpunctella* (Hübner), was determined using a range of concentrations and exposure times. On concrete surfaces ≥ 7.5 g/m^2 of filter cake produced more than 99% mortality of *S. zeamai*s and *S. oryzae* adults within 12–24 h, whereas more than three times the concentration of filter cake was required to achieve similar mortality of both species in Triplex treatments. At 3 g/m^2 of filter cake, 99% mortality *S. zeamai*s and *S. oryzae* adults was achieved within 22–27 h of exposure. The corresponding exposure time at 9 g/m^2 of Triplex was 39 h to achieve 99% mortality of both species. For both powders, lower concentrations and exposure times were required to achieve complete suppression of progeny production, percentage of insect damaged kernels, and percentage of grain weight loss compared to the concentrations and exposure times required for 00% mortality. Filter cake treated wheat at concentrations above 0.7 g/kg produced more than 99% mortality of *S. zeamai*s and *S. oryzae* adults. Similarly, filter cake concentrations above 2 g/kg on wheat produced more than 99% mortality of *R. dominica*, *T. castaneum*, and *O. surinamensis* adults. However, on maize ≥ 3 g/kg of filter cake concentration was required to achieve similar mortality of *R. dominica*, *T. castaneum*, and *O. surinamensis*. Higher concentrations of Triplex were required to achieve similar mortalities of tested species on maize and wheat. Reduction in progeny production was greater when adults were exposed to higher concentrations than lower concentrations. Complete suppression of live larvae and adult emergence of *P. interpunctella* was achieved after exposure of eggs for 21 and 42 d to ≥ 2 g/kg of filter cake treated maize and to ≥ 0.5 g/kg of filter cake treated wheat. Similarly, complete suppression of live larvae and adult emergence was achieved when eggs were exposed to ≥ 6 g/kg of Triplex treated maize and to 3g/kg of Triplex treated wheat. In general, our study consistently showed that filter cake was more efficacious compared to Triplex against all tested species on both surfaces and commodities. Filter cake and Triplex should be recommended for protecting grain stored by smallholder farmers in Ethiopia to discourage farmers from using dangerous chemical insecticides. However, field studies should be done using both powders against stored product insects in smallholder farmers' traditional storages structures in Ethiopia to determine concentrations that are practical under field conditions. The effective duration of protection offered by these powders should also be investigated.

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Dedication

This dissertation is dedicated to my ever supportive and loving wife, Ms. Abinet Mandefro Demillie and my daughter Mebatsion Tesfaye Melak, and my son Anteneh Tesfaye Melak. It is also dedicated to my dad, Merigeta Melak Tadesse, my mom Woizero Shigult Mekonnnen Admasu, my Sister Absera Melak Tadesse, brothers Abebe Melak Tadesse and Zenawi Melak Tadesse, and their families as well as my spiritual father Bishop Abuna Phillipos Ashagre. Lastly, a special dedication to my uncle Kes Mekdes Tadesse, who passed away during my elementary school, but guided me to be positive in life.

Chapter 1 - Background

1.1. Pesticide use in Ethiopia

Different types of pesticides have been used in Ethiopia since the 1960s (Mengistie, 2016). These pesticides are usually organophosphates, carbamates, and to a certain extent organochlorines. Pesticide use by smallholder farmers was frequently accompanied by misuse of pesticides resulting in poisoning of users, and causing chronic health effects, accumulation of pesticide residues in food and drinking water. There is a great concern that farm workers should be aware of the adverse effects of pesticide use if not handled properly (Mekonnen and Agonafir, 2002). The need to feed the growing population in Ethiopia, and the interest to produce exportable commodities to access the global markets led to increasing pressure to intensify agriculture resulting in increased use of chemical pesticides (Mengistie et al., 2014). To promote pesticide governance that protects the environment and human health, Ethiopia has developed a legal framework of pesticide registration and control. However, pesticides in Ethiopia are still registered, traded, and used inappropriately. The regulatory framework of pesticide use and its challenges are discussed below.

1.2. Pesticide regulatory framework

Under the current institutional arrangements, the ministry of Agriculture (MoA) and its counterparts in the agricultural bureau of regional states are major government agencies responsible for regulating, implementing, and monitoring of pesticide policies. These policies cover pesticide registration, importation, distribution, and use (Mengistie, 2016). State environmental and social institutions, and private actors could play an important role in pesticide governance to overcome failure of the state in enforcing pesticide policies. The first pesticide regulation was a single article included in the Plant Quarantine Decree No. 56 of 1971. According to this decree, the MoA was given the mandate to control the importation, production and sale of pesticides in the country. The decree was considered as a startup for the use of pesticides by farmers. However, the decree lacked the necessary details to establish an effective pesticide registration scheme (Mengistie, 2016). Before 1990 decree, ‘Pesticide Registration and Control Council of State Decree No. 20/1990’ approval, Ethiopia did not register pesticides. According to the decree, the manufacture, importation, and sale or use of unregistered pesticides was prohibited. In 1996, about 28 products were registered. The 1990 decree was still missing some of international obligations and agreements to which Ethiopia is a member. For example, definition of technical terms, pesticide registration, temporary prohibition, transport of pesticides, and penal sanctions to illegal trade of pesticides in retail shops and in open markets (Brodesser et al., 2006). In 2010, “Pesticide Registration and Control Proclamation No. 674/2010” was approved. The proclamation gave authority to the MoA to regulate all pesticides used for pest control including vectors of human and animal diseases. The proclamation adequately included many of international obligations and agreements, and issues that were not considered in the 1990 decree. For example, provision of certificates for competence and licensing, safety measures, analysis, a Pesticide Advisory Board (PAB), inspectors and some miscellaneous provisions were included (Mengistie et al., 2014).

The current structure of MoA in Ethiopia is focused on three major sectors: agricultural development, natural resource, and disaster prevention and food security (Mengistie et al., 2014). The first has most to do with pesticide management. Animal and Plant Health Regulatory Directorate (APHRD) under agricultural development sector is responsible for the development and promotion of pesticide lifecycle management system including registration and postregistration activities. Through pesticide registration, the responsible national or regional authority approves the sale and use of pesticide following the evaluation of comprehensive scientific data demonstrating that the product is effective for the intended purpose and does not pose an unacceptable risk to human or animal health or to the environment (FAO and WHO, 2014). The MoA is responsible for controlling the registration and importation of pesticides by issuing an import permit, provided the application submitted by importer contains the necessary safety data as prescribed by the MoA. Importation certificate is mandatory from MoA to import a pesticide. According to Mengistie et al. (2014), the registration process is schematically presented in Fig. 1.1.

Following the regulatory procedure, since pesticide registration started, 274 different types of pesticides were registered for agricultural and household uses, of which 44 constituted mixtures of two or more active ingredients while the rest contained a single active ingredient (Mengistie et al., 2014). Between the periods of 2013 and 2016 the number of pesticides registered increased from 276 to 376, and the number of importer organizations increased from 40 to 56 during the same period (MoA, 2016). According to USAID (2017), Ethiopian pest management and pesticide evaluation report, based on efficacy, safety to humans and non-target organisms, 28 pesticides were proposed for use against a range of pest problems on major crops of Ethiopia (Table 1.1). Among the pesticides proposed, there are nine insecticides and nematicides, nine fungicides, five herbicides, four fumigants, seed treatments and stored product insecticides, and one acaricide. Many of these are currently in use in Ethiopia. The fumigants, seed treatments and stored product treatments included aluminum-phosphide (I), imidacloprid (II-III), pirimiphose-methyle (II-III) and thiram (III). Currently there are no practical or preferable alternatives to the use of aluminum phosphide fumigants for the control of pests of stored grain in large warehouses. Due to this, USAID (2017) strongly recommended application and use of these chemicals should only be carried out by commercial fumigators in Ethiopia and under no circumstances would it be used by smallholder farmers in Ethiopia. However, many farmers purchase aluminum phosphate and use it in their traditional storages inside their homes (Dessalegn et al., 2017).

1.3. Major Challenges on the current pesticide use and enforcement

1.3.1. Pesticide Inspection and Quality Control

The regulatory requirements on the basis of article 30 of the 2010 proclamation, the ministry or a regional state organization in charge of the agricultural sector shall have the power, at working hours, without a warrant and upon presentation of their representative identity card, to carry out all the responsibilities. However, Mengistie (2016), reported from his interviews that state pesticide regulators are not carrying out their tasks in conformity with the power given in the proclamation. Lack of monitoring and surveillance after registration is one of the major problems. The pesticide market is heavily dependent on imports by local agents, representing international manufacturing/formulating companies. Mengistie et al. (2016), reported that only 12 of the 32 interviewed importers have documented records of the product quantities they imported, stored, and sold. None of the 32 importers were ever visited by MoA to inspect their stores unless they were invited to do so as a precondition for license renewal by the Ministry of Trade and Industry.

1.3.2. Lack of information and technical knowledge

The provision of useful agricultural information requires an understanding of policy issues by service providers, as well as policy makers of what providers are able to supply to support the policy making process (Blandford, 2007). Access to information for policy implementations is considered inadequate at the national and local level, and seen as a major operational challenge in Ethiopia (Mengistie et al., 2014; Mengistie, 2016). In addition, information about environmental health safety, efficacy, and safe use of pesticides is not provided by most of the importers during distribution. Mengistie et al. (2016) reported that 25 out of 32 importers did not provide this information during distribution. The report also indicated that all pesticide retailers at the district level responded that they were not familiar with the proclamation of 2010. In theory, in the decentralized system of Ethiopia, decision-making is shifted to the local level, but in practice the top-down approach is still in place. The lack of experts is a significant challenge to disseminate information on the pesticide policy with a simplistic approach (Mengistie et al., 2016). The same survey indicated that none of the retailers interviewed (*n* = 60) had received any training from the manufacturers, importers, state agencies, NGOs, or any other service providers on safe pesticide handling and storage. Due to this situation, they were not able to offer training to farmers. Nine retailers were unable to read and understand labels, but had some idea of the simple ones. Only 17 retailers were able to explain most of the labels, whereas 34 of the remaining retailers had no understanding even of the basic pesticide labels.

1.3.3. Low motivation of state actors

The motivation of state actors is critical to transfer knowledge to farmers and enhance the implementation of policy at the farm level. In the Ethiopian context, motivation refers to the dedication and willingness of the protection experts, extension supervisors, and development agents to serve farmers. Strong motivation of these actors is critical to transfer knowledge to the farmers to enhance implementation of any technology at the farmers' level. Mengistie (2016) reported that there was a common understanding among all state actors at district level that lack of motivation was manifested by inadequate support from federal and regional states, such as inadequate training, lack of clear carrier structure, work load due to shortage of extension workers compared to the size of farmers, and bureaucratic hurdles from regional states. For example, in the interview performed by Mengistie et al. (2014), 18 out of 30 development agents did not like their job because the salary was not proportional to their workload.

1.3.4. Weak interaction

In Ethiopia, each agricultural office of the district is composed of five main teams: extension, irrigation, input supply, natural resources and food security. However, the interaction between these teams is weaker and agricultural extension service activities are operating in a non-integrated manner. Mengistie (2016) reported that 28 out of the 30 interviewed development agents responded that lack of collaboration between and among relevant actors of pesticides negatively affected pesticide management, because of it was not focused on integrated service and information delivery. There is also a lack of coordination between the government and other stakeholders, especially the private sector. Lack of communication and information sharing is the major problem with extension line directorates and from the federal to kebele levels under MoA as it is the only agricultural office that is responsible at the local level for farmers on issues related to pesticides.

1.3.5. Lack of resources

Financial and human resources are core variables for determining policy implementation. Resource constraints that affect pesticide registration, distribution and use at the federal, district and most kebele levels are mostly observed in Ethiopia. For instance, at the federal level, most of the experts have at least a master degrees, but their expertise is not evenly distributed along the broad range of subjects that are relevant for evaluation of pesticide registration (Mengistie et al., 2014). Several studies indicated that there is a clear capacity gap in handling and running different responsibilities under the decentralized system in Ethiopia. Decentralization of authority to district levels has been a major government program over the last few years. However, there are many challenges, including capacity limitations to implement development activities. According to Mengistie et al. (2014), at the federal level, informants from APHRD stated that the registration process was carried out through the assessment of data provided by the importers themselves. Trial data from the country of origin are submitted to the APHRD and the values of efficacy and safety were obtained from the Codex Alimentarius or EU-MRL databases. The registration process was not supported by the independent laboratory test because MoA has no facilities to determine and control the quality of the pesticide. There is no in-depth inspection and control over inert and active ingredients, while pesticides with the same active ingredients can vary a lot in efficacy and toxicity due to differences between the inert ingredients used. Pesticides with similar names may also have been registered differently as active ingredients and mixtures of inert ingredients.

1.4. Research Justification

A wide spread availability of chemicals and drugs lead to acute poisoning resulting in medical emergencies observation worldwide (Santosh et al., 2013). Intentional and unintentional pesticide poisoning have been acknowledged as a serious problem in many agricultural communities of low- and middle-income countries (Thundiyi et al., 2008). Even though low and middle-income countries use only 20% of the world’s agrochemicals they have 99% of deaths from acute pesticide poisoning (APP) cases (Kesavachandran et al., 2009). The weak regulations and limited health care services in developing countries resulting adverse outcomes from poisoning which are more prevalent than in the developed countries (Adinew et al., 2017a). Epidemiological data on acute poisoning were extremely few and it is difficult to find primary data. This is because of unavailability of well-organized poison control centers and routine screening and confirmatory tests (Chelkeba et al., 2018). Ethiopia does not have an integrated reporting system of occupational accidents and diseases hampering the estimation of the number and prevalence of APP cases. Hospital-based studies on intentional poisoning in Ethiopia have identified organophosphate pesticide as the main means of self-poisoning (Desalew et al., 2011; Abebe, 1991).

There is also occupational poisoning during application and handling of pesticides in Ethiopia. Organophosphates, organochlorines, and carbamates are the major group of agrochemicals used in Ethiopia (Mengistie et al., 2017). Organophosphates and carbamates are potent inhibitors of acetylcholinesterase enzyme (AChE), responsible for hydrolysis of acetylcholine (ACh) which is involved in the transmission of impulses between interneuronal and neuromuscular nerve junctions. Depression of plasma or red blood cell cholinesterase activity is the most satisfactory and generally available evidence of excessive absorption of organophosphate pesticides (International Labor Organization, 1979). This situation was reported by Lakew and Mekonnen (1998) after a study of the northern Omo state farm workers exposed to the organophosphates chlorpyrifose and profenifos. The pesticide use and health problems in various activities on Birr and Ayehu, privately owned agricultural farms located in West Gojjam, Ethiopia, were studied by Ejigu and Mekonnen (2005) using liver function tests. A total of 82 farm workers, with 47 controls, from both farms volunteered for the study. Compared to the information provided on commercially available diagnosis kits used in the study, the mean values of hepatic enzymes in most farm workers at both farms were elevated. This enzyme elevation is an indication of cell damage in liver cells (International Labor Organization, 1979). A cross-sectional survey which involved 516 households by grouping them based on residence proximity to a large flower farm in Oromia region, Ethiopia, was conducted from August to September 2014 by Negatu et al. (2016a). Sixty four percent of those living near the flower farms (*n* = 257) were working in the flower farm. A total of 136 respondents had experienced two or more symptoms of acute pesticide intoxication (API) such as nervous system, respiratory and gastrointestinal symptoms. These symptoms are most frequently reported from exposure to pesticide classes of organophosphates, carbamates, and pyrethroids (Thundiyil et al., 2008). Another study by Negatu et al. (2018) in the Central Eastern part of Ethiopia, where abundant hydrological resources exist from the Rift valley Lakes and Awash River. Farms in this region produce commercial crops on which use of agrochemicals including pesticides is high due to production of different kinds of horticultural crops, namely roses, cuttings, vegetables fruit, and cotton. They surveyed a total of 256 pesticide applicators on different farm settings (large scale green houses, large scale open farms, and small scale irrigated farms). An overall prevalence of 16% acute pesticide poisoning, which manifested with neurobehavioral symptoms were reported from all the three farming systems. Fifty percent of the reported pesticides causing APP were organophosphates (predominantly profenofos) followed by organochlorines (in particular endosulfan and morpholine).

Poisoning by organophosphate and other pesticides is a common clinical problem in Ethiopia (Abebe 1991, Negatu et al., 2016a,b; Mengistie et al., 2017). According to Abebe (1991) 85 poisoning cases were diagnosed in Tikur Anbesa Hospital due to different causes from 1983 to 1985 and from 1987 to 1989. Twenty six female and 24 male patients had organophosphate poisoning, and where 74% of them were under the age of 30. Ninety four percent (*n* = 50) of the organophosphate poisoning cases were due to people committing suicide by ingesting the chemical. The medical records of 116 adult patients from January 2007 to December 2008 in Tikur Anbesa Teaching Hospital were analyzed by Desalew et al. (2011). The majority (96.5%) of cases were self-poisoning and 21.6% of them used organophosphates. Eyasu et al. (2017) analyzed a total of 592 women with acute poisoning cases from the 2010 to 2015 recorded data from St. Paul's Hospital Millennium Medical College, Zewuditu Memorial Hospital, Yekatit 12 Hospital Medical College and Ras Desta Damtew Memorial Hospital. All the hospitals are in Addis Ababa, Ethiopia. One hundred twelve cases were caused by organophosphate poisoning, and 38 cases were caused by rodenticides. The majority of them were under the age of 30 years. A short report by Mohammed et al. (2017) from Addis Ababa Burn Emergency and Trauma (AaBET) hospital over a two month period (April and May, 2016) follow up of seven patients with aluminum phosphide poisoning by investigators indicated death of five individuals.

Shibre et al. (2014) reported 14% (*n* = 919) of the suicidal attempts were caused by organophosphate poisoning after reviewing a 10 year follow up of people diagnosed with mental disorders among adults in Butajira, Ethiopia. Analysis of poisoning case records from 2012 to 2013 in Jimma University Specialized Hospital (JUSH), Ethiopia was done by Teklemariam et al. (2016). Twenty eight out of 103 cases, were caused by organophosphate poisoning with majority of the cases were under the age of 20 years.

A study on the government hospitals in northwest Ethiopia (Goder University Teaching Hospital, Metema and Debark district's Hospitals) was reported by Adinew et al. (2017a). The study reviewed 344 cases of poisoning which have complete records in the hospital's registrar offices. Of all the cases, 121 were caused by organophosphates, and 19 were caused by organochlorines. From the organophosphate poisoning cases, 70 out 121 were women. Seventy one out of 344 were recorded to have committed suicide. Adinew et al. (2017b) also reported 90 out of 234 patients recorded for organophosphate poisoning from University of Gondar Teaching hospital during September 2010 to December 2014. Out of 90 cases, 54 were women and the majority of them were under the age of 30 years. A similar study by Adinew and Asrie (2016) on acute poisoning cases in University of Gondar Teaching Hospital from 2010 to 2014 reported organophosphates were the most frequent causes of poisoning and accounted for 38.2% (89 out of 233 cases), and organochlorines comprised 2.1% (5 cases). Female patients constituted the majority (97 out of 134 cases) of suicidal poisoning. The large majority (88.42%, *n* = 233) were younger than 30 years of age.

Chelkeba et al. (2018) reviewed several studies on the prevalence of acute poisoning and reported the highest prevalence was observed below the age of 30. Individuals at this age group are the most physically, mentally, and socially active age groups, which makes them prone to increased levels of stress as the majority of them are under pressure to support themselves and their families. These make them vulnerable to commit risk-taking behaviors. The review also indicated that 78% (*n* = 9) of the studies reviewed reported the higher occurrence of acute poisoning in females than males. This could be due to the existence of social repression of females seen in due to cultural reasons and delays in seeking medical attention. In Ethiopia, the reason why females attempt suicide at a higher rate than males is hypothesized to be due the cultural malpractices towards them. Most young females are followed and controlled closely by their family compared to males (Chelkeba et al., 2018). In addition to the aforementioned reasons, farmers’ pesticide management practices are also critical causes of intentional and unintentional pesticide poisoning in Ethiopia. Farmers apply pesticides in violation of the recommendations. They use unsafe storage facilities, ignore risks and safety instructions, and do not use protective apparel when applying pesticides, and dispose containers unsafely (Negatu et al., 2016a,b; Mengistie et al., 2017). Such cases are related to violation of international and Ethiopian codes of conduct for pesticide management are of which require training and certification (FAO and WHO, 2014). A survey done by Negatu et al. (2016b) on knowledge, attitude, and practices of farmers and farm workers in Ethiopia reported that 85% (*n* = 601) of farm workers (pesticide mixers/loaders, pesticide sprayers, and application supervisors) and 100% (*n* = 275) of female re-entry farm workers (harvesters, pesticide assessors, irrigation workers, irrigation supervisors, packing and sorting workers, transport/push car workers) did not receive any pesticide-related training. In addition, 62% (*n* = 258) of farm workers did not shower after a pesticide application, and none of the small-scale farm workers used personal protective equipment and apparel. A considerable increment of direct importation of pesticides without the formal Ethiopian registration process, were also reported by several researchers (Mengistie, 2016; Mengistie et al., 2016; Negatu et al., 2016a,b; Mengistie et al., 2017; Chelkeba et al., 2018).

Therefore, safe and non-chemical alternatives should be provided to smallholder farmers in order to reduce hazards related to pesticides. New non-chemical technologies such as Purdue Improved Crop Storage (PICS) bags, GrainPro Super bags, plastic drums, and metal silos were introduced to sub-Saharan Africa to protect smallholder farmers' stored commodities (Obeng-Ofori, 2011; Murdock and Baoua, 2014; Chigoverah and Mvumi, 2016). In addition to these new technologies, some inert dusts, such as clay, sand, ground phosphate, ash, diatomaceous earths were in use for a long period of time in Africa (Headlee, 1924; Ebeling, 1971; Subramanyam and Roesli, 2000; Liška et al., 2017). Inert dusts are dry dusts that are chemically unreactive in nature (Liška et al., 2017; Bohinc et al., 2018), and are identified as alternatives to chemical pesticides (Subramanyam and Roesli, 2000). Protection of stored grain from insect pests using inert dusts such as wood ash, lime, sand, and tobacco has been practiced in Ethiopia in the past (Abraham et al., 2008).

There is a need to explore products that are safe and effective in controlling insects in smallholder farmers' storages in Ethiopia. Two such potential products are filter cake and Triplex. Filter cake is a by-product of aluminum sulfate factory (Awash Melkassa Aluminium Sulphate and Sulphuric Acid Share Company, Melkassa Awash, Ethiopia (AMASSASC)). Triplex is a by-product of Mohammed International Development Research and Organization Companies (MIDROC) soap factory (Star Soap and Detergent Industries (SSDI Private Limited Company), Addis Ababa, Ethiopia). Triplex is derived from berries of the Endod plant, *Phytolacca dodecandra* L. Studies by Girma et al. (2008a,b) suggested that filter cake and Triplex could be recommended for controlling the maize weevil, *Sitophilus zeamais* Motschulsky in maize in farmers' storages if they are safe to humans. However, there are no data on the elemental composition of these powders, especially with respect to heavy metals. There is no published information on the toxicity of filter cake and Triplex against multiple stored product insect species.

The elemental composition of filter cake and Triplex was determined as there were no data on elemental composition. The efficacy of filter cake and Triplex against the lesser grain borer, *Rhizopertha dominica* (Fabricius); *S. zeamais*; rice weevil, *Sitophilus oryzae* (Linnaeus); red flour beetle, *Tribolium castaneum* (Herbst); saw-toothed grain beetle, *Oryzaephilus surinamensis* (Linnaeus); and Indian meal moth, *Plodia interpunctella* (Hübner) on concrete surfaces, and on maize and wheat was determined because there were no data except that of Girma et al. (2008a,b) on *S. zeamais* on maize. These species are the major primary insect pests of stored grains in Ethiopia (Abraham et al., 2008; Nukenine, 2010; Befikadu, 2018). Knowing the optimum concentration and exposure time for each species on surfaces and on grain is important for the management of these species. My results are presented in the following chapters, and forms the crux of my dissertation.

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Table 1.1. List chemicals registered by MoA for use on stored commodities in Ethiopia

Trade Name	Common name	Approved use to control
Actellic 2% dust	pirimiphos-methyl	Storage pests on cereals and pulses
Alphos 56% Tab.	Aluminum phosphide 560 mg/kg	Maize weevil on maize.
Celphos	aluminum phosphide 56% tablet	Maize weevil and flour beetles
Degesch Plates/Strips	magnesium Phosphide 56%	Maize weevil on maize grain/seeds
Delicia	aluminum phosphide 56.7%	Storage pests on cereals and pulses
Deltacal 0.2DP	deltamethrin 0.2%DP	Maize weevil on stored maize
Detia Gas-Ex-T	aluminum phosphide 56.7%	Storage weevils and beetles
Ethiolathion 5% Dust	Malathion	Maize Weevil on stored maize
Fullongphos	aluminum phosphide	Maize weevil and other storage pests
Gastoxin	aluminum phosphide 57% tablet	Maize weevil and other storage pests
Helmathion 50 Ec	malathion 50% EC	Storage insect pests in storage structures
Kill-phose	aluminum phosphide	Storage insect pests
Litphos 56 TB	aluminum Phosphide 56%	Maize weevil
Mogphos 56% Tablet	aluminum phosphide	maize weevil
Helmathion 50 Ec	malathion 50% EC	Storage insect pests
Kill-phose	aluminum phosphide	Storage insect pests
Litphos 56 TB	aluminum Phosphide 56%	Maize weevil
Mogphos 56% tablet	aluminum phosphide	Maize weevil
Phostoxin	aluminum phosphide	Storage pests in warehouses
Quickphos	aluminum phosphide	Storage pests
SD-Toxin Tablet	aluminum Phosphide 56.8%	Storage insect pests
Shenphos 57% Tablet	aluminum Phosphide	Maize weevil and flour beetles
Talic 2% Dust	pirimiphos-methyl	Storage pests
Tanphos 56% TAB	aluminum-phosphide	Weevils and flour beetles

(Source: USAID, 2017)

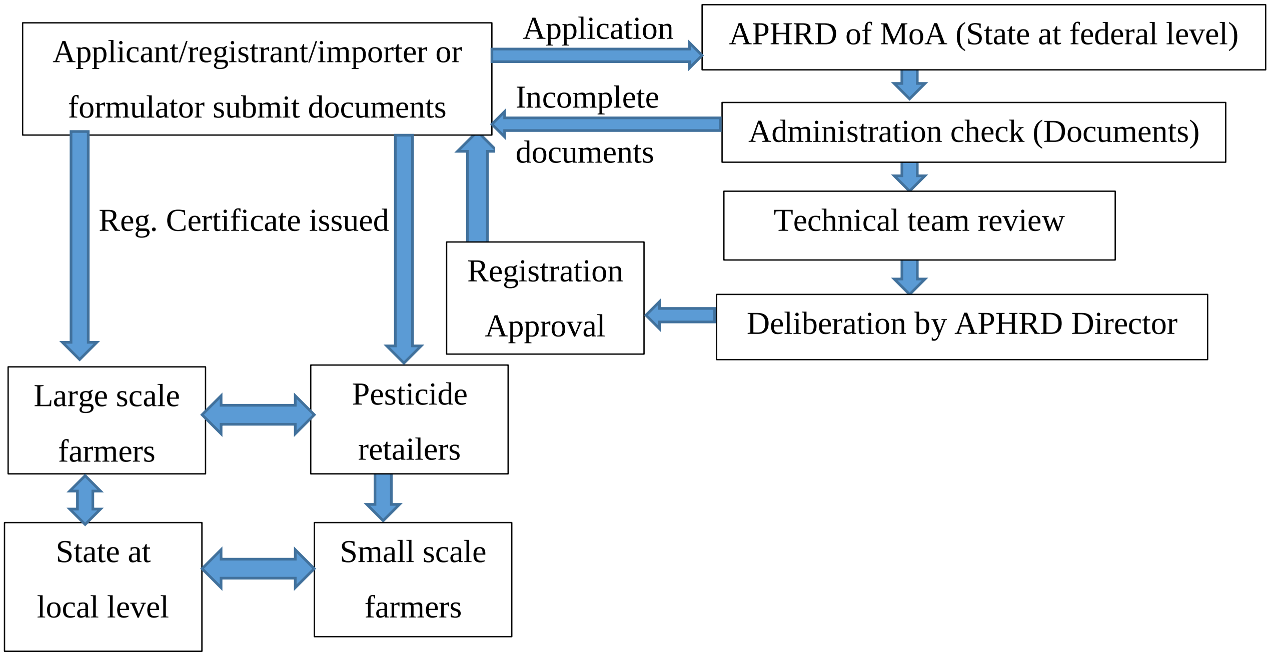

Figure 1.1. Schematic diagram of the pesticide product registration process (Modified from Mengistie et al., 2014)

Chapter 2 - Efficacy of filter cake and Triplex powders from Ethiopia applied to concrete arenas against *Sitophilus zeamais*

2.1. Abstract

Filter cake and Triplex are powdered by-products of aluminum sulfate and soap factories, respectively. This study was designed to determine the chemical composition of these two powders using the conjugated scanning electron microscopy (SEM) and Energy-dispersive X-ray spectroscopy (EDS). Another aim was to evaluate their contact toxicity to the maize weevil, *Sitophilus zeamais* Motschulsky, a common pest of stored grains in Ethiopia. Contact toxicity against *S. zeamais* was determined by exposing 10 adults for 4, 8, 12, and 24 h in 9 cm diameter concrete arenas inside Petri dishes dusted with filter cake and Triplex at each of the six concentrations (0, 2.5, 3.75, 5, 7.5 and 10 g/m^2). Each concentration and exposure time combination was replicated five times. After the intended exposure time, adults were transferred to 150-ml round plastic containers with 30 g of organic hard red winter wheat and held at 28^{o}C and 65% r.h. for 7 d to determine mortality. Adult progeny production, number of insect damaged kernels, and grain weight loss at each powder-concentration-time combination were determined after 42 d. Both powders were free of heavy metals. Silicon and oxygen were the major component of both powders in the form of silicon dioxide. The 7–d mortality was 100% after a 24 h exposure to 7.5 g/m^2 concentration of filter cake and 10 g/m^2 of both powders. Reduction in adult progeny production, number of insect damaged kernels, and grain weight loss were greater when *S. zeamais* adults were exposed to the powders for longer time periods at higher than lower concentrations. This is attributed to greater mortality at increasing concentrations and exposure times. Our results indicate that filter cake and Triplex can be used to treat concrete surfaces of warehouses and storage structures to control *S. zeamais*.

Keywords: Ethiopia; *Sitophilus zeamais*; Filter cake; Triplex; Contact toxicity

2.2. Introduction

Post-harvest losses of dry durable commodities in sub-Saharan Africa are significant, and estimated to range from 20 to 40% (Zorya et al., 2011). Grain becomes infested by insect pests during storage if the storage structure is not insect-proof. In Ethiopia farmers store grains in traditional storages, which are not insect-proof, predisposing the grain to storage losses of 20 to 30% (Tefera et al., 2011). Therefore, cheap and effective methods of reducing losses due to insect pests in smallholder farmers' storages have been explored (Abraham et al., 2008; Kemal and Mekasha, 2013). In Ethiopia, smallholder farmers purchase small amounts of unknown pesticides from local shops and self-apply the chemicals to their grains to control insect pests. A recent paper (Tilahun and Hussen, 2014) reported that farmers are improperly trained and not knowledgeable on proper and safe use of pesticides.

There is a need to explore products that are safe and effective in controlling insects in smallholder farmers' storages in Ethiopia. Two of such products are filter cake and Triplex. Filter cake is a by-product of aluminum sulfate factory (Awash Melkassa Aluminium Sulphate & Sulphuric Acid Share Company, Melkassa Awash, Ethiopia (AMASSASC)). Triplex is a by-product of Mohammed International Development Research and Organization Companies (MIDROC) soap factory (Star Soap and Detergent Industries (SSDI Private Limited Company), Addis Ababa, Ethiopia). Triplex is derived from berries of the Endod plant, *Phytolacca dodecandra* L.

Limited information is available on the effectiveness of these powders against storage insect pests when applied to stored maize (Girma et al., 2008a,b). Girma et al. (2008a) treated three maize genotypes with 1, 2.5, and 5% (w/w) of filter cake and reported 92% mortality of the maize weevil, *Sitophilus zeamais* Motschulsky, after exposure for 3 d to treated maize. Admixing

Triplex with maize at a concentration of 0.25% (w/w) was reported with no significant difference in mean percentage mortality of *S. zeamais* (93%) when compared with that of a synthetic insecticide, pirimiphos-methyl (100%), 7 months after treatment (Girma et al., 2008b). These studies by Girma et al. (2008a,b) suggest that filter cake and Triplex could be recommended for controlling *S. zeamais* in smallholder farmers' traditional storages. However, there are no data on the chemical composition of these powders, especially with respect to heavy metals. There is no published information on the toxicity of filter cake to other organisms, but there are several reports on toxicity of hexane extracts of Endod berries to mollusks (Lemma, 1970; Baalawy, 1972; Lemma et al., 1972; Parkhurst et al., 1974), and the yellow fever mosquito, *Aedes aegypti* L. (Spielman and Lemma, 1973; Yugi et al., 2014). The contact toxicity of filter cake and Triplex to *S. zeamais* or any other stored-product insect when applied to concrete surfaces is unknown. Therefore, this study was designed to determine elemental composition of these two powders and contact toxicity to adults of *S. zeamais*, an economically important stored grain insect pest in Ethiopia, under laboratory conditions (Girma et al., 2008a,b; Abraham et al., 2008; Tefera et al., 2011).

2.3. Materials and Methods

2.3.1. Determining the elemental composition of filter cake and Triplex

Elemental composition of each sample was determined using Hitachi S-3500N scanning electron microscope (SEM) for surface morphology study conjugated with Oxford energy dispersive X-ray detector for elemental analysis (Hitachi Science Systems Ltd., Tokyo, Japan, 1998) at Nanotechnology Innovation Center, Kansas State University (http://nicks.ksu.edu/electron-microscopy/). Hitachi S-3500N scanning electron microscope is a fully digital instrument that provides high resolution (3 nm) images of a sample. The image is formed by backscattered electrons (BSE), a primary electron that has been ejected from a solid by scattering through an angle greater than 90 degrees (Egerton, 2005). The backscattering coefficient (the fraction of primary electrons that escape as BSE) increases with atomic number, and BSE images can show contrast due to variations in chemical composition of a sample (Egerton, 2005). An X-ray energy dispersive spectroscopy (EDS) system is attached to provide elemental identification and quantitative compositional information of a sample. The EDS has a wide range of energy which enables it to detect all elements, with the exception of H and He, at every location sampled by the beam (Newbury and Ritchie, 2015). Ideally, each peak in the EDS spectrum represents an element present within a known region of the sample, defined by the focused probe (Egerton 2005). Six samples of each powder were mounted on scanning electron microscope stub using adhesive double-sided carbon tape. Different parts were scanned to be detected by the beam from each sample. All elements were analyzed without omitting any peak while processing the spectrum.

2.3.2. Concrete-poured Petri dishes

Rockite®, a ready-to-mix concrete product (Hartline Products Co., Inc., Cleveland, Ohio, USA), was mixed with tap water to make a slurry. The slurry was poured into 9 cm diameter and 1.5 cm high plastic Petri dishes (Fisher Scientific, Denver, Colorado, USA). Slurry was poured to cover one half of the Petri dish’s height. Slurry filled Petri dishes were allowed to dry on a laboratory bench for 24 h. Polytetrafluoroethylene (Insecta-a-Slip, Bio Quip Products, Inc., Rancho Dominguez, California, USA) was used to coat the inside walls of Petri dishes to prevent insects from crawling on sides of dishes.

2.3.3. Application of powders to concrete arenas and insect exposure

Concrete arenas of Petri dishes were treated with each powder at the following concentrations: 0 (untreated control), 2.5, 3.75, 5, 7.5, and 10 g/m^2. Insects used in this study originated from the population reared for 16 years in the Department of Grain Science and Industry, Kansas State University. A laboratory strain of *S. zeamais* was reared in an environmental growth chamber (Percival Scientific, Inc., Model I-36VL, Perry, Iowa, USA) at 28°C and 65% r.h. on organic yellow maize (Heartland Mills, Marienthal, Kansas, USA) of 13.5% moisture content (wet basis). *S. zeamais* adults were separated from maize using a 2.38 mm diameter round-holed aluminum sieve (Seedburo Equipment Company, Des Plaines, Illinois, USA). Ten unsexed adults of mixed ages were introduced to untreated concrete arenas (control) and arenas receiving each of the five concentrations of a powder. Adults were exposed to untreated and treated concrete arenas for 4, 8, 12, and 24 h, on a laboratory bench. HOBO® data loggers (Onset Computer Corp., Bourne, Massachusetts, USA) indicated the mean ± SE (*n* = 1440) of temperature and relative humidity in the laboratory during exposures were 23.5 ± 0.02˚C (range, 22.2 – 25.9˚C) and 20.7 ± 0.04% (range, 16.8 – 29.1%), respectively. Each powder-concentration-time combination was replicated five times. After introducing insects Petri dishes were covered with lids for the intended exposure time. After the intended exposure time insects were transferred carefully with a Camel's hair brush to 150ml round plastic containers holding 30 g of cleaned, organic hard red winter wheat (Heartland Mills) of ~12% moisture content (wet basis). The plastic containers had perforated lids with wire-mesh screens to facilitate air diffusion. Containers were incubated in an environmental growth chamber at 28°C and 65% r.h. After 7 d, wheat from each container was sifted using a 2.38 mm circular round-holed aluminum sieve (Seedburo Equipment Company) to separate insects from wheat. Insects that did not respond when gently prodded by a Camel's hair brush were considered dead. After mortality determination, containers with live and dead insects and wheat were held for an additional 35 d at 28°C and 65% r.h. Adult progeny produced was counted 42 d after exposure to filter cake and Triplex from each container, and the 10 starting adults were subtracted. Wheat from each container was passed three times through the Boerner® divider (Seedburo Equipment Company) to get a working subsample of ~3.8 g for determining undamaged and insect damaged kernels in replicate samples. Number of damaged kernels out of the total examined was expressed as a percentage. Grain weight loss, expressed as a percentage, was determined based on counting and weighing damaged and undamaged kernels from each replicate working sample (Adams and Schulten, 1978; Boxall, 1986). In each replicate control and treated working samples, percent weight loss was calculated using the following equation:

Weight loss (%) = $[(UN_d - DN_u) \div (U \times (N_d + N_u))] \times 100$

where, U is the weight of undamaged kernels, N_d is the number of damaged kernels, D is the weight of damaged kernels, and N_u is the number of undamaged kernels.

2.3.4. Data analysis

Atomic percent of each element from each sample obtained using SEM were used to calculate mean atomic percent ± SE. The mean ± SE mortality of *S. zeamais* on untreated (control) arenas at all exposure times ranged from 0.0 to 4.0 ± 2.4 and 0.0 to 2.0 ± 2.0 for filter cake and Triplex, respectively. Therefore, mortality data of *S. zeamais* exposed to filter cake and Triplex were corrected for responses in the control treatment (Abbott, 1925). The nonlinear models, $y^{0.5} = a + b/\ln x$ and $y^{0.5} = a + b\ln x/x^2$, were fit to corrected mortality responses (y) as a function of filter cake and Triplex concentrations (x), respectively, for each exposure time using

Table Curve 2D software (Jandel Scientific, San Rafael, California, USA). The parameters *a* and *b* were estimated by fitting equations to concentration and corrected mortality data. Each possible pair-wise comparison between exposure times for each powder was done by comparing individual models to a pooled model (Draper and Smith, 1998). Any two models being compared were considered to be significantly different ($P < 0.05$) from one another if the *F*-test showed individual models to deviate from the pooled model.

The adult progeny production data were transformed to $\log_{10}(x+1)$ scale (Bartlett, 1947), whereas percentage of insect damaged kernels and grain weight loss data were transformed to square root (Zar, 1984), to normalize heteroscedastic treatment variances. However, untransformed raw data are presented in tables and graphs. Graphs were plotted using SigmaPlot, version 12.5 (Systat Software, Inc., San Jose, California USA). Progeny production, insect damaged kernel, and grain weight loss data were subjected to three-way analysis of variance (ANOVA) to determine significant differences ($P < 0.05$) of main (powder, concentration, and time) and interactive effects (SAS Institute, 2012). Data on progeny production, insect damaged kernel, and grain weight loss at each concentration of filter cake and Triplex were subjected to one-way ANOVA to determine significant differences ($P < 0.05$) among exposure times, and means were separated by Ryan-Einot-Gabriel-Welsch multiple range test (REGWQ) (SAS Institute, 2012).

2.4. Results

2.4.1. Chemical composition of filter cake and Triplex

Oxygen, sodium, silicon, sulfur, aluminum, and potassium were found in both powders. In addition to these, calcium, iron, and zinc were found only in Triplex samples and carbon in filter cake samples. An example of identification of elements from scanning electron microscopy/energy dispersive X-ray spectrometry (SEM/EDS) is indicated in Fig. 2.1. Oxygen and silicon were major components of both powders with highest mean atomic percent in their standard form SiO_2 (silicon dioxide). The mean atomic percent ± SE of oxygen and silicon in filter cake were 42.65 ± 10.80 and 16.38 ± 5.35, respectively, and in Triplex it was 23.16 ± 2.04 and 69.06 ± 3.23, respectively. Existence of elements identified with their standard forms are given in Table 2.1.

2.4.2. Concentration and corrected mortality responses of S. zeamais at different exposure times

Mortality of *S. zeamais* adults exposed to filter cake and Triplex increased with increasing concentration and exposure time, and reached 100% at 7.5 g/m^2 for filter cake and 10 g/m^2 for both powders at 24 h (Fig. 2.2). Increase in mortality was nonlinear, and the fitted models satisfactorily described the concentration and corrected mortality data of filter cake ($r^2 = 0.79 – 0.93$) and Triplex ($r^2 = 0.81 – 0.90$) (Table 2.2). Mortality responses of *S. zeamais* exposed to filter cake and Triplex at 4 and 8 h were significantly different at $P < 0.10$, whereas those exposed to filter cake at 8 and 12 h were not significantly different ($P > 0.87$). However, all the remaining pairwise comparisons between exposure times for each powder were significant ($P < 0.05$) (Table 2.3).

2.4.3. Adult progeny production at 42 d

Mean ± SE adult progeny production of *S. zeamais* ranged from 112.0 ± 7.6 to 157.0 ± 15.7 adults per container and from 109.6 ± 8.6 to 168.2 ± 12.7 adults per container in filter cake and

Triplex control treatments of, respectively. Reduction in adult progeny production was greater when *S. zeamais* adults were exposed for longer time periods to higher than lower concentrations for both powders. The percentage reduction in progeny production of *S. zeamais* after a 4 to 24 h exposure to filter cake relative to production on untreated controls ranged from 11.1 to 100% (Fig. 2.3). The corresponding percentage reduction in progeny production relative to production on untreated controls after a 4 to 24 h exposure to Triplex ranged from 21.2 to 100% (Fig. 2.4). Adult progeny production was completely suppressed for insects exposed for 24 h to 7.5 and 10 g/m^2 of both powders.

Three-way ANOVA showed significant differences in number of adult progeny produced among powders (F = 39.95; df = 1, 192; P < 0.0001), concentrations (F = 179.47; df = 5, 192; P < 0.0001), and exposure times (F = 555.00; df = 3, 160; P < 0.0001). The interactions, powder x concentration (F = 4.09; df = 5, 192; P = 0.0015), powder x time (F = 4.23; df = 3, 192; P = 0.0063), concentration x time (F = 20.25; df = 15, 192; P < 0.0001), and powder x concentration x time (F = 4.47; df = 15, 192; P < 0.0001) were all significant.

One-way ANOVA showed significant differences in number of adult progeny produced among exposure times for each concentration of filter cake (F, range among concentrations = 4.39 – 161.84; df = 3, 16; P, range < 0.0001 – 0.0195) and Triplex (F, range among concentrations = 6.66 – 81.61; df = 3, 16; P, range < 0.0001 – 0.0040). Adults exposed to 2.5 g/m^2 of filter cake or Triplex produced significantly higher number of progeny than the rest of concentrations at each exposure time, whereas those exposed to 7.5 and 10 g/m^2 produced significantly less number of progeny. This is attributed to high mortality at these concentrations.

2.3.4. Insect damaged kernels at 42 d

Mean ± SE insect damaged kernels by *S. zeamais* ranged from 17.2 ± 1.2 to 22.4 ± 2.0% and from 13.2 ± 1.2 to 20.2 ± 1.8% in filter cake and Triplex control treatments, respectively. Percent of insect damaged kernels was greater when *S. zeamais* adults were exposed for shorter time periods to lower than higher concentrations for both powders. Percentage of reduction in insect damaged kernels by *S. zeamais* after exposure to filter cake for 4 to 24 h relative to damage in the control treatment ranged from 20.6 to 100% (Fig. 2.5). Corresponding percent reduction of insect damaged kernels after exposure to Triplex relative to that in the control treatment ranged from 0.4 to 100% (Fig. 2.6). Complete reduction in quantity of insect damaged kernels was obtained in treatments where adult insects were exposed for 24 h to 5 g/m^2 of filter cake and to 7.5 and 10 g/m^2 of both powders.

Three-way ANOVA showed significant differences in percentage of insect damaged kernels among powders (F = 5.33; df = 1, 192; P = 0.0220), concentrations (F = 96.48; df = 5, 192; P < 0.0001), and exposure times (F = 157.27; df = 3, 192; P < 0.0001). Powder x concentration (F = 4.06; df = 5, 192; P = 0.0016), and concentration x time (F = 7.07; df = 15, 192; P < 0.0001) were the only significant interactions.

One-way ANOVA showed no significant differences among exposure times for percent of insect damaged kernels data collected only for filter cake control treatments (F = 2.26; df = 3, 16; P = 0.1213). However, insect damaged kernels data collected for 2.5, 3.75, 5, 7.5 and 10 g/m^2 filter cake concentrations showed significant differences among exposure times (F, range among concentrations = 7.78 – 40.50; df = 3, 16; P, range < 0.0001 – 0.0020). Insect damaged kernels data collected from each concentration of Triplex showed significant differences among exposure times (F, range among concentrations = 3.61 – 27.75; df = 3, 16; P, range < 0.0001 –

0.0366). Adults exposed to 7.5 and 10 g/m^2 had significantly less kernel damage than the rest of concentrations at each exposure time, which can be attributed to greater mortality at these concentrations of filter cake and Triplex.

2.4.5. Grain weight loss at 42 d

Mean ± SE percentage of grain weight loss due to *S. zeamais* ranged from 7.0 ± 0.7 to 9.9 ± 1.5% and from 4.7 ± 1.4 to 8.6 ± 0.3% in filter cake and Triplex control treatments, respectively. Grain weight loss was greater when *S. zeamais* adults were exposed for shorter time periods to lower than to higher concentrations of both powders. Percent reduction in grain weight loss due to *S. zeamais* after exposure to filter cake for 4 to 24 h relative to the control treatment ranged from 26 to 100% (Fig. 2.7). Corresponding percent reduction of grain weight loss after exposure to Triplex relative to the control treatment ranged from 18.2 to 100% (Fig. 2.8). Complete reduction of grain weight loss was obtained in treatments where adults were exposed for 24 h to 5 g/m^2 of filter cake, as well as to 7.5 and 10 g/m^2 of both powders.

Three-way ANOVA showed no significant differences among powders ($F = 0.37$; df = 1, 192; $P = 0.5429$). However, significant differences were observed among concentrations ($F = 66.95$; df = 5, 192; $P < 0.0001$) and exposure times ($F = 90.21$; df = 3, 192; $P < 0.0001$). Powder x concentration ($F = 3.81$; df = 5, 192; $P = 0.0026$) and concentration x time ($F = 4.44$; df = 15, 192; $P < 0.0001$) were the only significant interactions.

One-way ANOVA showed no significant differences among exposure times for grain weight loss data collected from filter cake and Triplex control treatments (filter cake: $F = 2.01$; df = 3, 16; $P = 0.1533$; Triplex: $F = 1.97$; df = 3, 16; $P = 0.1598$). However, grain weight loss data collected from 2.5, 3.75, 5, 7.5 and 10 g/m^2 filter cake or Triplex concentrations were significantly different among exposure times (filter cake: *F*, range among concentrations = 5.84 – 17.54; df = 3, 16; *P*, range < 0.0001 – 0.0068; Triplex: *F*, range among concentrations = 5.17 – 15.98; df = 3, 16; *P*, range < 0.0001 – 0.0109). In treatments where adults were exposed to 7.5 and 10 g/m^2 of either powder had significantly less grain weight loss than the rest of concentrations at each exposure time. This is attributed to high mortality and less number of insect damaged kernels at these concentrations, especially at exposure time of 24 h.

2.5. Discussion

SEM and EDS microanalysis has given the materials community one of its most powerful microstructural characterization tools (Newbury and Ritchie, 2015). It is a widely applied elemental microanalysis method capable of identifying and quantifying all elements in the periodic table except H, He, and Li (Newbury and Ritchie, 2013). Chemical composition determined by SEM and EDS indicated that analyzed filter cake and Triplex were free of heavy metals. Oxygen and silicon in their standard form as silicon dioxide (SiO_2) were major components of both powders with highest atomic percent. The presence of silicon dioxide in filter cake might be related to the use of a diatomite as a filtering agent for sulfuric acid production in AMASSASC (Daniel, 2006). Silicon dioxide is a major constituent of sand, and occurs in many plants. Plants with high silicon dioxide content tend to be resistant to predation by herbivores (Massey et al., 2006; Keeping and Kvedaras, 2008). The silicon dioxide found in the Endod plant may serve a similar purpose. Silicon dioxide is an abrasive material and acts as a desiccant, adsorbing or abrading epicuticular lipids covering the insects' exoskeleton causing them to dry out and die (Subramanyam and Roesli, 2000). Such pesticides with low mammalian toxicity but high toxicity to insects are important for treating surfaces, cracks and crevices, or empty bins to control stored product insects (Toews and Subramanyam, 2003).

Our data demonstrated contact toxicity of filter cake and Triplex through concentration and exposure time responses of *S. zeamais* adults. The mortality of *S. zeamais* increased with increasing concentration and exposure time to filter cake and Triplex. Accumulation of powder over insect's body is directly proportional to concentration of the powder (Le Patourel et al., 1989), and insect behavior such as mobility. These factors cause adverse effects to exposed insects (Malia et al., 2016a). Since a very small application rate was used in our experiments, uneven distribution of powder was more likely to occur on the concrete arenas, and insects may have escaped contact by moving to areas in the concrete arena with little or no filter cake or Triplex. Therefore, distribution of filter cake and Triplex on concrete arenas and activity of *S. zeamais* may have affected extent and duration of contact with powders and contributed to unexplained variation in mortality responses of adult *S. zeamais* among treatments.

The mode of action of filter cake and Triplex on insects is unknown. However, since silicon dioxide is a major component of both powders, the mechanism of action could be related to abrasion or adsorption of wax on the cuticle. We hypothesize that filter cake and Triplex have a mode of action similar to that of diatomaceous earth powders (Malia et al., 2016b; Subramanyam and Roesli, 2000). In our study, we observed dead insects that were brittle, which could be due to water loss from their body. A positive correlation between oil absorption and silicon dioxide concentration was reported by Filipović et al. (2010) after testing oil absorption capacity of silica powders prepared from sodium silicate solution with different concentration of silicon dioxide. This is supported in our study where adult mortality tended to increase with increasing concentration of filter cake and Triplex. However, the proportion of oxygen and silicon as SiO_2

in both filter cake and Triplex was smaller than that in diatomaceous earth, and our result cannot be compared directly with the work done on diatomaceous earth by numerous researchers (Arthur, 2002; Athanassiou et al., 2003, 2005; Collins and Cook, 2006a,b; Girma et al., 2008a,b; Campolo et al., 2014; Doumbia et al., 2014).

Filter cake and Triplex caused significant reduction of progeny production, number of insect damaged kernels, and weight loss as the concentration and exposure time increased. These are related to greater mortality with an increase in concentration and exposure time. High adult mortality and significant reduction of adult progeny production, quantity of insect damaged kernels, and weight loss are important aspects that suggest both powders have potential for management of *S. zeamais* in farmers' storages in Ethiopia. Girma et al. (2008a,b) has shown effects of those powders against *S. zeamais* when applied to maize. Our recent study (Tesfaye and Subramanyam, 2018) on wheat showed that they are effective when applied to wheat at concentration of 700 mg/kg.

In our experiments both filter cake and Triplex powders were highly effective at 7.5 and 10 g/m^2 concentration after a 24 h of exposure. The overall results of this study showed that filter cake was more efficacious than Triplex. This could be related to its carbon content (atomic percent = 39.43 ± 12.63) in the form of calcium carbonate. Calcium carbonate at 1 and 2% (w/w) applied to maize caused 70.2 and 84.2% mortality of *S. zeamais* adults, respectively, 15 d after treatment (Silva et al., 2004). Additional work should be done using 7.5 and 10 g/m^2 application rates in empty traditional storages in Ethiopia. Our study used concrete arenas to simulate floors of warehouses and empty storage bins. However, traditional storages in Ethiopia, such as *gota* and *gotera* are made with teff straw, mud, and cow dung (Abraham et al., 2008).

Silicon dioxide containing insecticides have been shown to be safe to mammals (Subramanyam and Roesli, 2000). Filter cake and Triplex are safer alternatives to unknown pesticides used by smallholder farmers to protect their commodities from insects. A study of pesticide use, practices, and risks in Gedeo and Borena zones of Ethiopia has shown that most farmers use pesticides on their own for use on grain stored in traditional storages, and purchase pesticides from private shops, local market, and government offices. There is no regulatory oversight to prevent such purchases (Tilahun and Hussen, 2014). Our study and that of Girma et al. (2008a,b) showed that filter cake and Triplex are viable alternatives to chemical pesticides. Zorya et al. (2011) in their assessment of postharvest losses in sub-Saharan Africa also emphasized the need for using environmentally benign products than using chemical pesticides to protect stored grain from insect infestation in smallholder farmer’s storages. Moreover, their local availability and accessibility will also reduce labor and financial pressure on smallholder farmers.

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Table 2.1. Elemental composition of filter cake and Triplex obtained using conjugated scanning electron microscopy and energy-dispersive X-ray analysis.

		% Mean atomic ± SE (*n* = 6)	
Element	Standard	Filter cake	Triplex
Ca	Wallastonite, Calcium silicate ($CaSiO_3$)		1.51 ± 1.42
O	Silicon dioxide (SiO_2)	53.93 ± 10.80	69.06 ± 3.23
Na	Albite, sodium aluminum silicate ($NaAlSi_3O_8$)	0.05 ± 0.05	1.67 ± 0.67
Si	Silicon dioxide (SiO_2)	16.38 ± 5.35	23.16 ± 2.04
S	Iron sulfide (FeS_2)	0.40 ± 0.17	0.04 ± 0.04
Al	Aluminum oxide (Al_2O_3)	1.04 ± 0.39	1.51 ± 1.42
K	Potassium oxide, MAD-10 Feldspar (K_2O)	0.05 ± 0.05	0.80 ± 0.35
C	Calcium carbonate ($CaCO_3$)	39.43 ± 12.63	
Fe	Iron (Fe)		1.44 ± 0.50
Zn	Zinc (Zn)		0.01 ± 0.01

Table 2.2. Parameter estimates of nonlinear regression models fit to concentration and mean 7 d corrected mortality data of *S. zeamais* adults exposed to filter cake and Triplex.

Powder	Exposure time (h)	Mean ± SE (n = 5) for parameter:		r^2
		a	b	
Filter cake	4	11.95 ± 3.28	-8.51 ± 5.56	0.7904
	8	13.11 ± 2.43	-8.01 ± 4.00	0.8812
	12	13.45 ± 2.94	-7.52 ± 4.76	0.8224
	24	11.14 ± 0.54	-2.45 ± 0.79	0.9313
Triplex	4	8.62 ± 1.18	-34.43 ± 18.66	0.8627
	8	10.08 ± 1.04	-36.46 ± 15.43	0.8973
	12	9.94 ± 0.62	-14.18 ± 7.78	0.8138
	24	10.21 ± 0.29	-9.63 ± 3.64	0.9049

Table 2.3. Pairwise comparison of nonlinear models fit to concentration and 7 d corrected mortality data of *S. zeamais* adults exposed to filter cake and Triplex.

Powder	Exposure times compared	*F*-value	df	*P*-value
Filter cake	4 h vs 8 h	3.64	2, 6	0.0921[**]
	4 h vs 12 h	5.99	2, 6	0.0371[*]
	4 h vs 24 h	35.78	2, 6	0.0005[*]
	8 h vs 12 h	0.14	2, 6	0.8720
	8 h vs 24 h	17.67	2, 6	0.0031[*]
	12 h vs 24 h	6.27	2, 6	0.0339[*]
Triplex	4 h vs 8 h	5.07	2, 6	0.0513[**]
	4 h vs 12 h	39.17	2, 6	0.0004[*]
	4 h vs 24 h	89.77	2, 6	<0.0001[*]
	8 h vs 12 h	12.65	2, 6	0.0070[*]
	8 h vs 24 h	34.36	2, 6	0.0005[*]
	12 h vs 24 h	5.84	2, 6	0.0391[*]

[*] Significant ($P < 0.05$).

[**] Significant ($P < 0.10$).

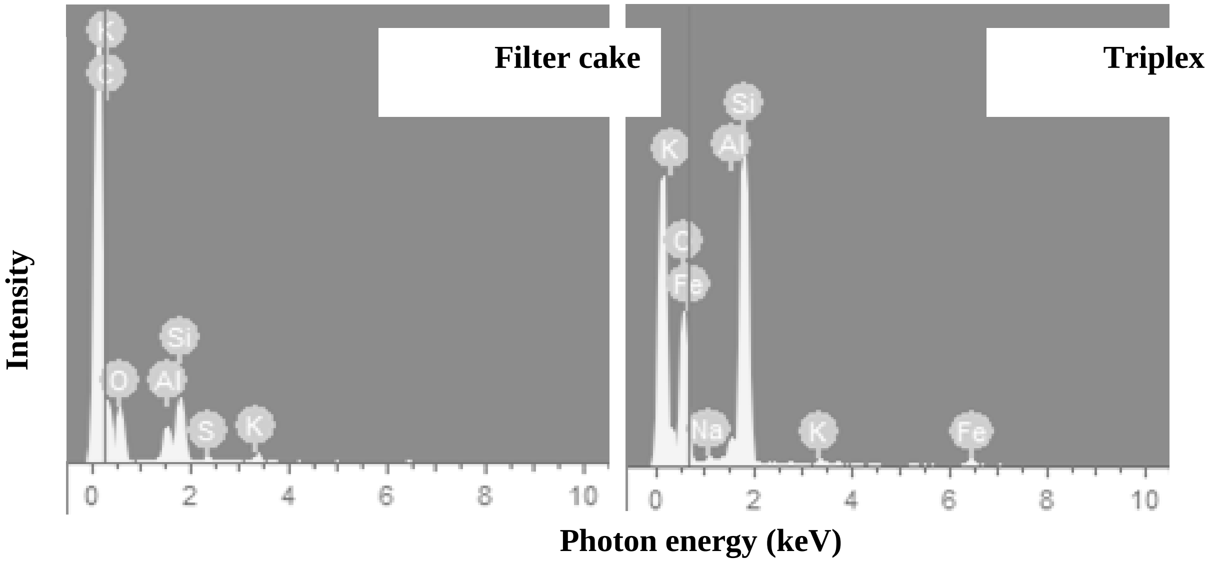

Figure 2.1. Elemental identification from spectrums produced by X-ray energy dispersive spectroscopy conjugated with scanning electron microscopy.

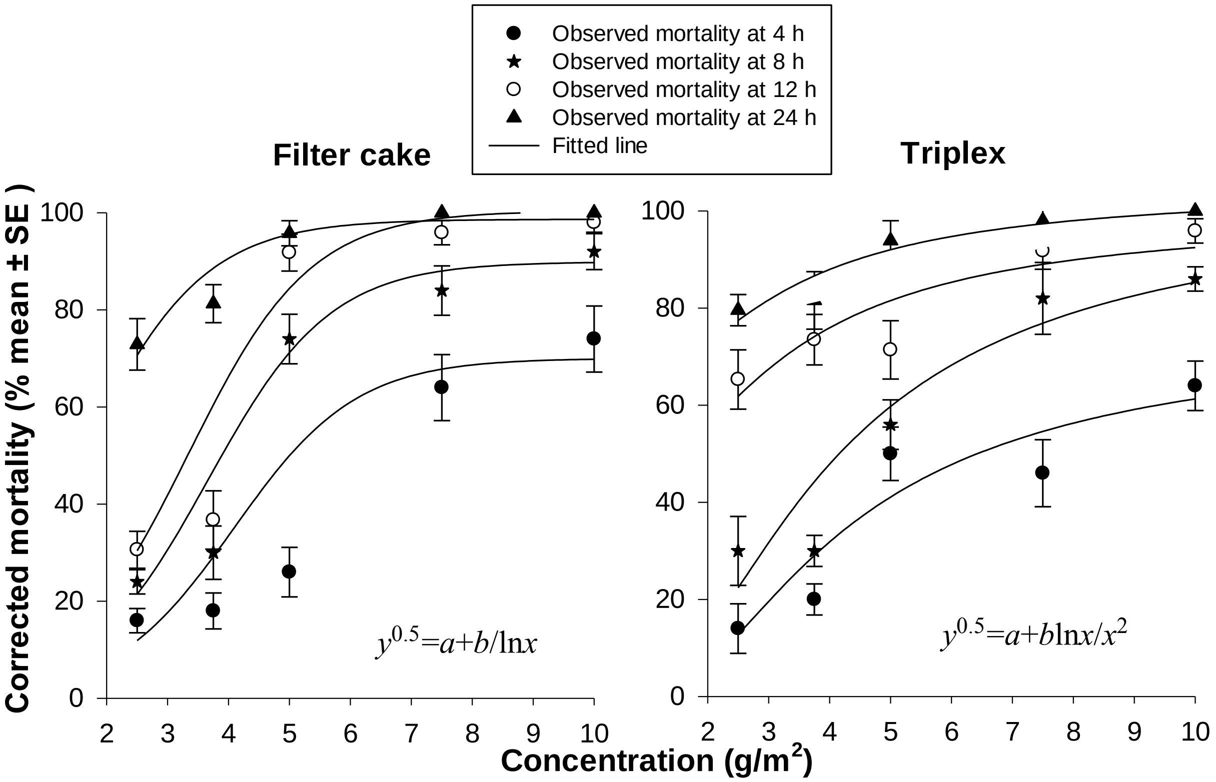

Figure 2.2. Corrected mean ± SE 7–d mortality of *S. zeamais* adults after 4, 8, 12 and 24 h of exposure to concrete arenas dusted with filter cake and Triplex concentrations of 2.5, 3.75, 5, 7.5 and 10 g/m^2.

Nonlinear models were fit to express concentration and corrected mortality data. See Table 2.2 for parameter estimate values of *a* and *b* of the fitted equations.

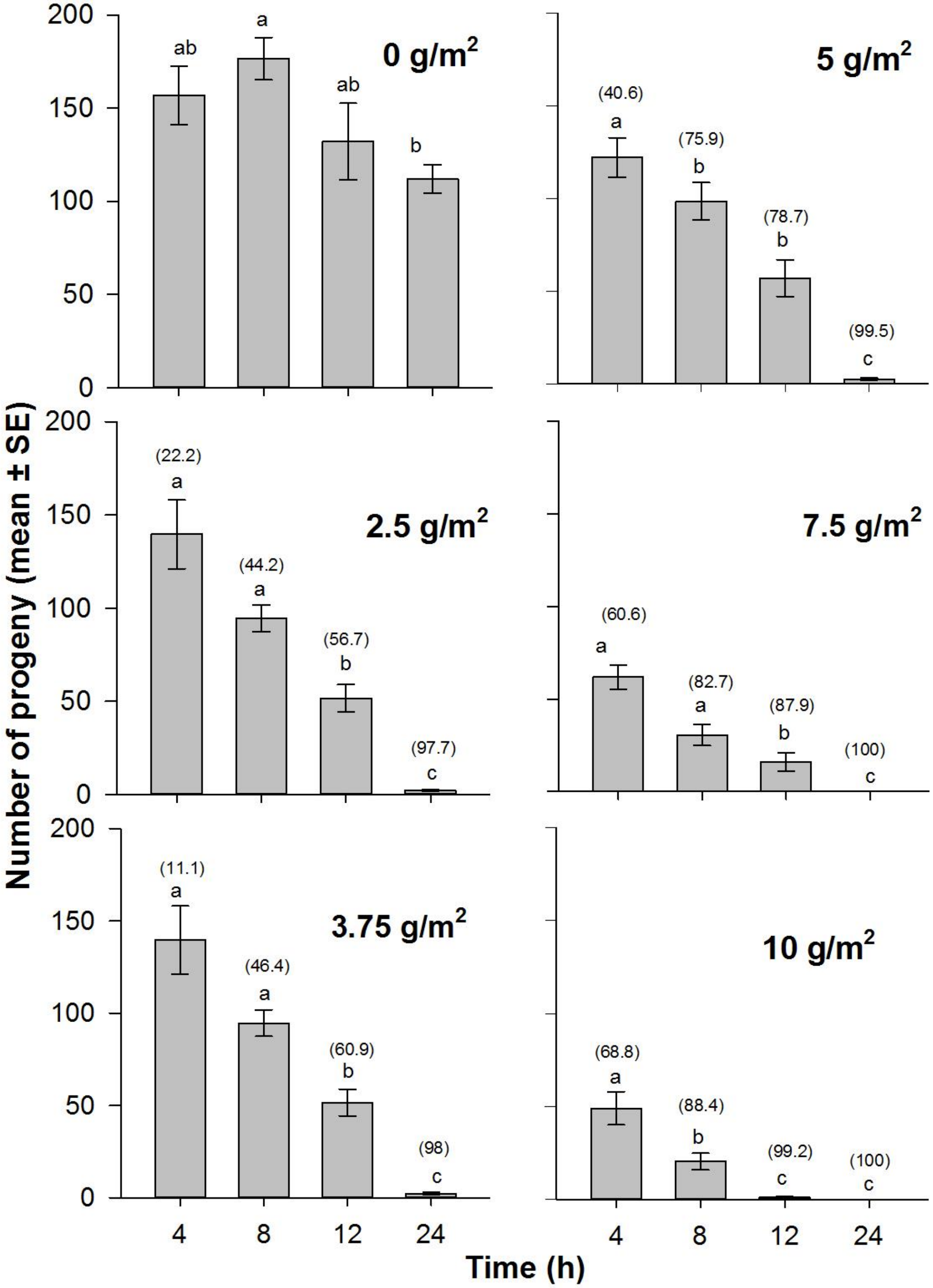

Figure 2.3. Mean ± SE number of adult progeny of *S. zeamais* produced at 42 d after a 4, 8, 12 and 24 h exposure to untreated (control) concrete arenas and concrete arenas dusted with filter cake at concentrations of 2.5, 3.75, 5, 7.5, and 10 g/m^2.

Number above each error bar is percentage reduction of adult progeny production after exposure to treated arenas relative to production on untreated arenas. Bars with different letters are significantly different (*F*, range = 4.39–161.84; df = 3, 16; *P*, range < 0.0001–0.0195; one way ANOVA with means separated by REGWQ multiple range test).

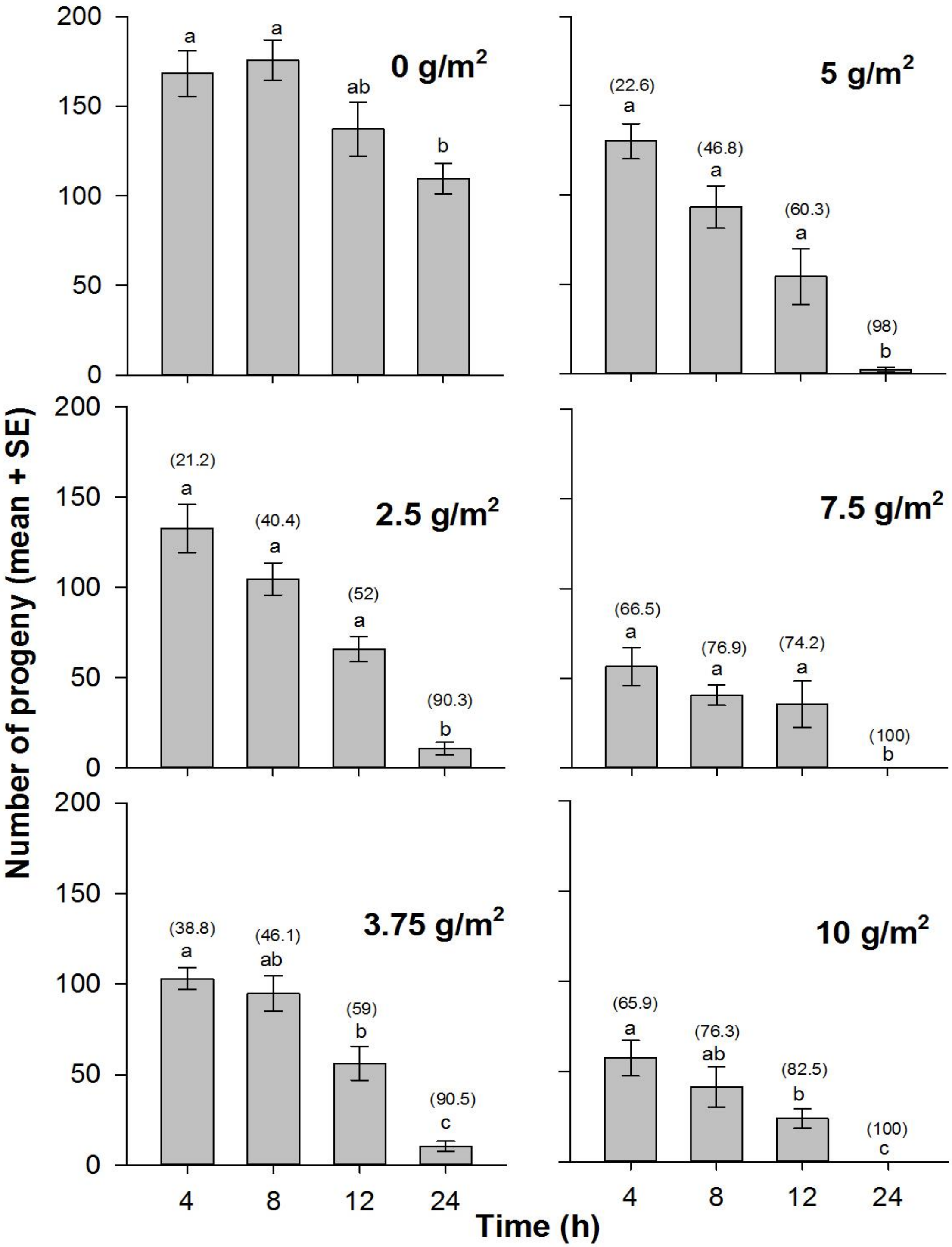

Figure 2.4. Mean ± SE number of adult progeny of *S. zeamais* produced at 42 d after a 4, 8, 12 and 24 h exposure to untreated (control) concrete arenas and concrete arenas dusted with Triplex at concentrations of 2.5, 3.75, 5, 7.5, and 10 g/m^2.

Number above each error bar is percentage reduction of adult progeny production after exposure to treated arenas relative to production on untreated arenas. Bars with different letters are significantly different (*F*, range = 6.66–81.61; df = 3, 16; *P*, range < 0.0001–0.0040; one way ANOVA with means separated by REGWQ multiple range test).

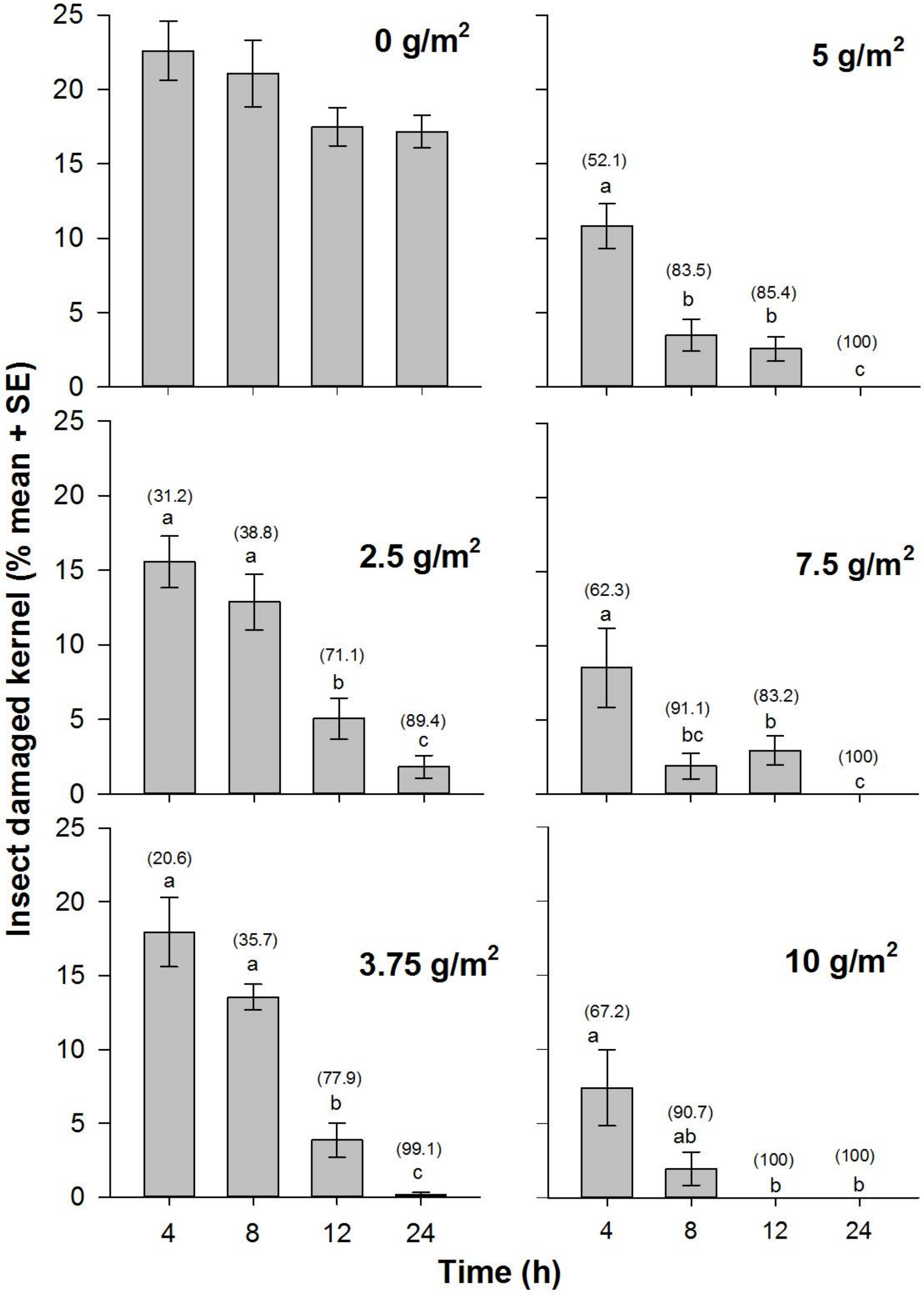

Figure 2.5. Insect damaged kernels (% mean ± SE) at 42 d caused by *S. zeamais* after exposure to 0, 2.5, 3.75, 5, 7.5, and 10 g/m^2 concentrations of filter cake for 4, 8, 12 and 24 h.

Number above each error bar is percentage reduction in number of insect damaged kernels in dust treatments relative to damaged kernels in control treatments. There was no significant difference in insect damaged kernels in 0 g/m^2 treatment among exposure times (F = 2.26; df = 3, 16; P = 0.1213; one–way ANOVA). Bars with different letters are significantly different (F, range = 10.59–40.50; df = 3, 16; P, range < 0.0001–0.0.0004; one way ANOVA with means separated by REGWQ multiple range test).

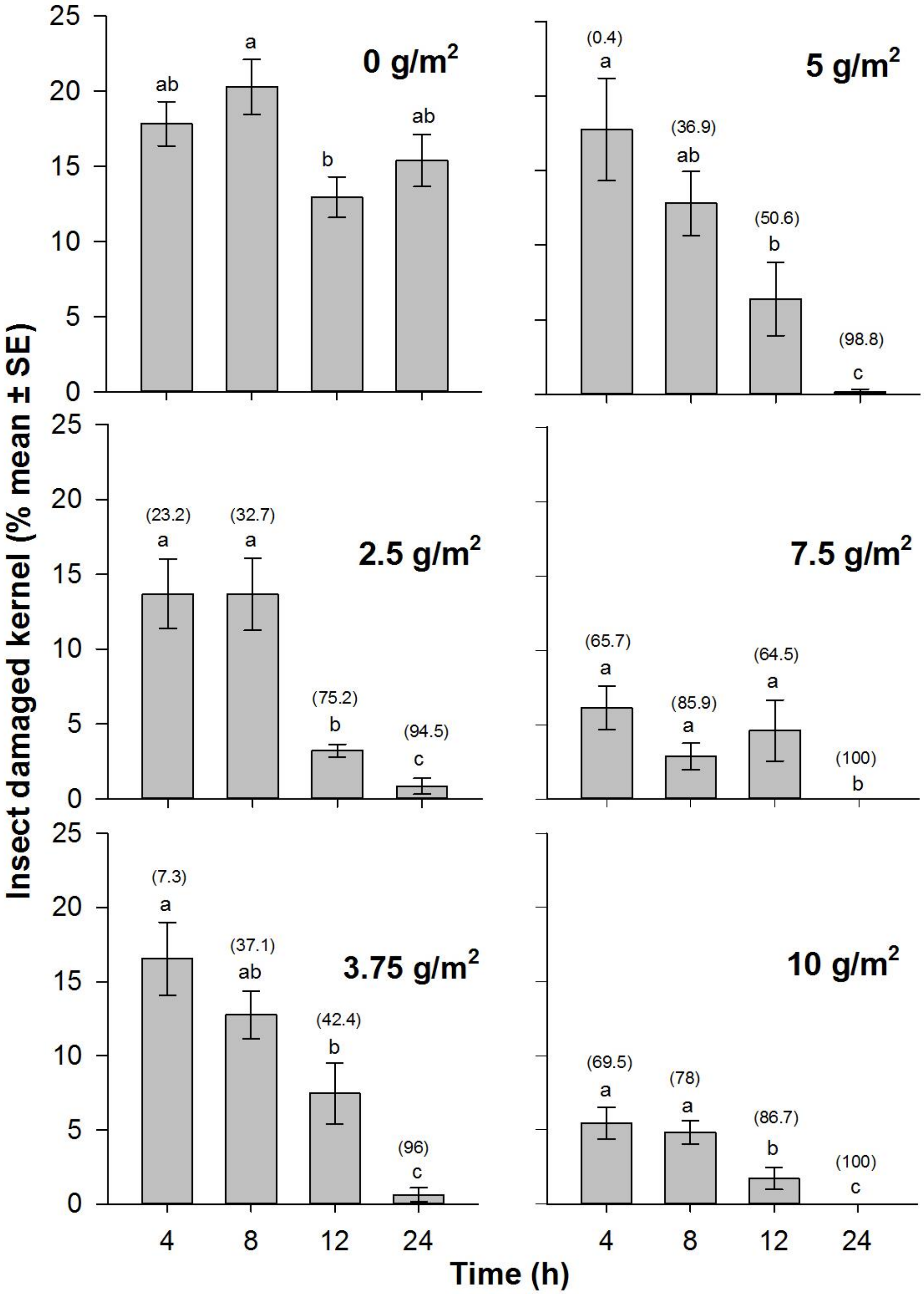

Figure 2.6. Insect damaged kernels (% mean ± SE) at 42 d caused by *S. zeamais* after exposure to 0, 2.5, 3.75, 5, 7.5, and 10 g/m^2 concentrations of Triplex for 4, 8, 12 and 24 h.

Number above each error bar is percentage reduction in number of insect damaged kernels in dust treatments relative to damaged kernels in control treatments. Bars with different letters are significantly different (*F*, range = 3.61–27.75; df = 3, 16; *P*, range < 0.0001–0.0366; one way ANOVA and means were separated by REGWQ multiple range test).

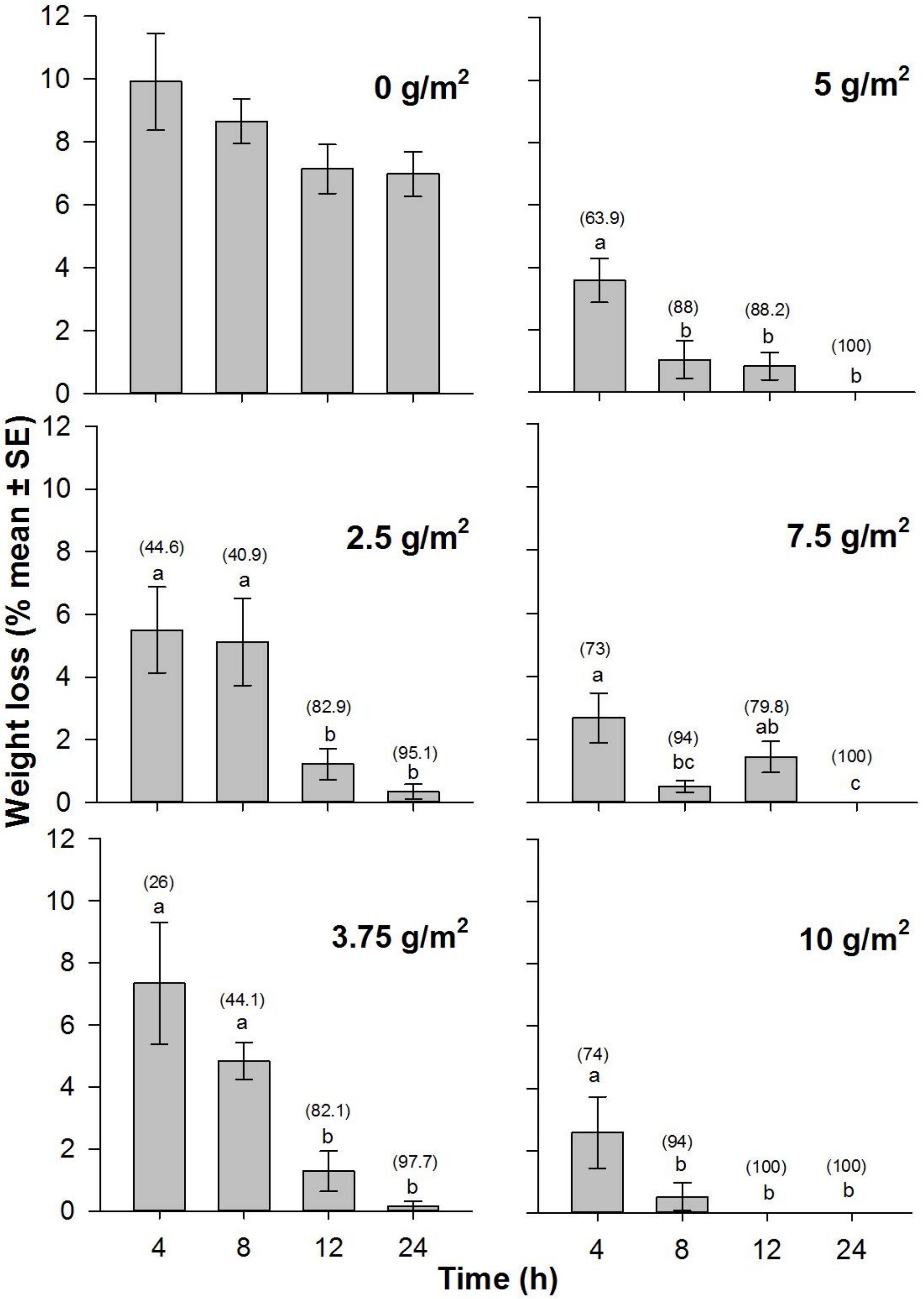

Figure 2.7. Grain weight loss (% mean ± SE) at 42 d caused by *S. zeamais* after exposure to 0, 2.5, 3.75, 5.0, 7.5, and 10 g/m^2 concentrations of filter cake for 4, 8, 12 and 24 h.

Number above each error bar is percentage reduction in weight loss in dust treatments relative to weight loss in control treatments. There was no significant difference in weight loss in 0 g/m^2 treatment among exposure times (F = 1.84; df = 3, 16; P = 0.1804; one–way ANOVA). Bars with different letters are significantly different (F, range = 3.89–17.54; df = 3, 16; P, range < 0.0001–0.1533; one–way ANOVA with means separated by REGWQ multiple range test).

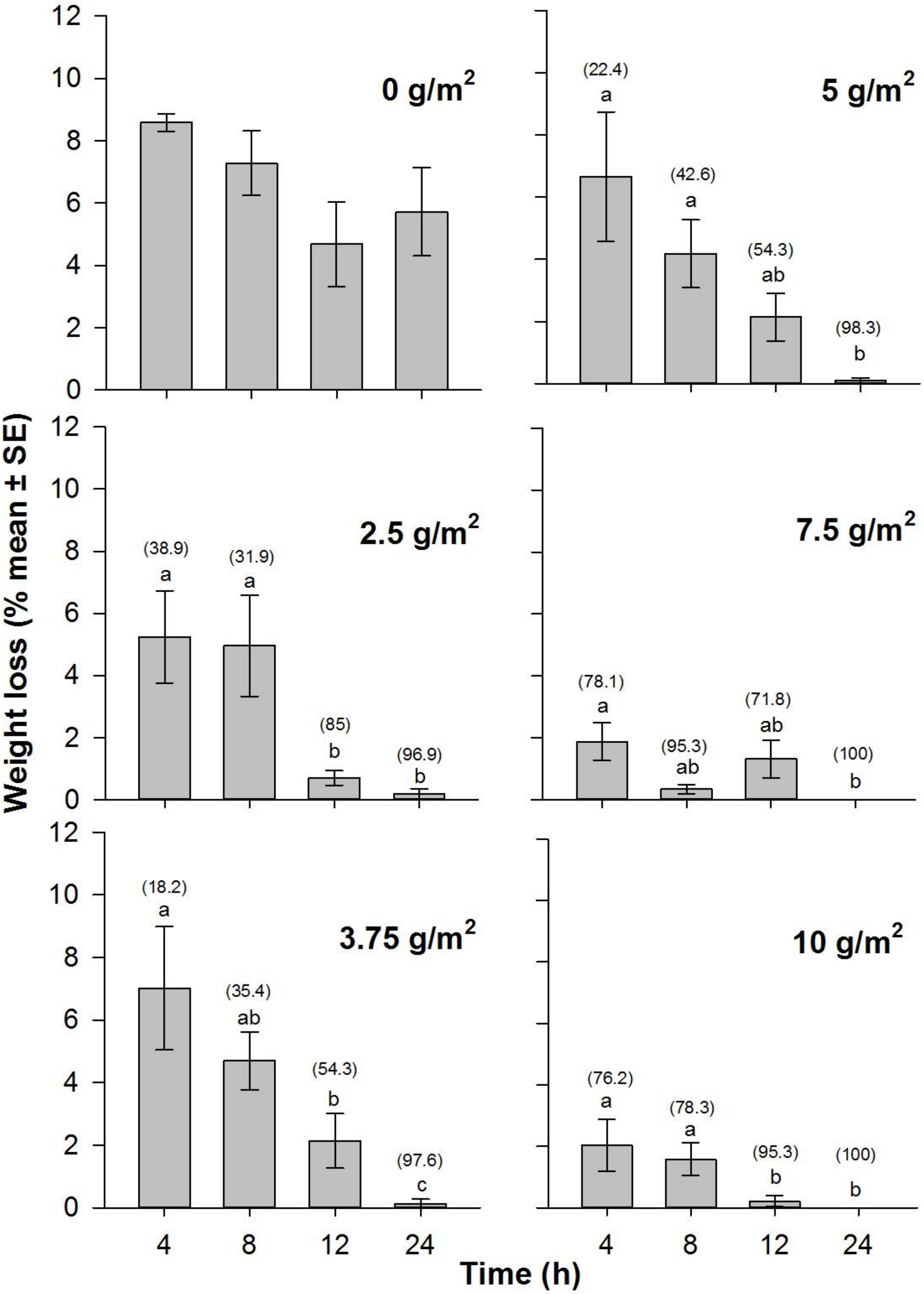

Figure 2.8. Grain weight loss (% mean ± SE) at 42 d caused by *S. zeamais* after exposure to 0, 2.5, 3.75, 5, 7.5, and 10 g/m^2 concentrations of Triplex for 4, 8, 12 and 24 h.

Number above each error bar is percentage reduction in weight loss in dust treatments relative to weight loss in control treatments. There was no significant difference in weight loss in 0 g/m^2 treatment among exposure times ($F = 1.89$; df = 3, 16; $P = 0.1713$; one–way ANOVA). Bars with different letters are significantly different (F, range = 4.00–14.88; df = 3, 16; P, range < 0.0001–0.0265; one–way ANOVA with means separated by REGWQ multiple range test).

Chapter 3 - Efficacy of filter cake and Triplex powders from Ethiopia applied to wheat against *Sitophilus zeamais* and *Sitophilus oryzae*

3.1. Abstract

The efficacy of filter cake and Triplex powders applied to wheat was evaluated in the laboratory against the maize weevil, *Sitophilus zeamais* Motschulsky and rice weevil, *Sitophilus oryzae* (L.), two of the most common insect pests associated with stored grain in Ethiopia. Efficacy of these powders was determined by exposing 20 adults of each species to 100 g of wheat treated with 0, 100, 500, 700 and 1000 mg/kg of filter cake or Triplex. Adult mortality was determined 7 and 14 d after exposure. In addition, adult progeny production, percentage of insect damaged kernels, and percentage of grain weight loss at each species-powder-concentration-time combinations were determined after 42 d. The 7 and 14 d mortality was 100% for adults of both species exposed to 1000 mg/kg of filter cake; only the 14 d mortality of *Sitophilus* species was 100% for adults exposed to 700 mg/kg. Mortality of *S. oryzae* adults was 100% when exposed for 14 d to1000 mg/kg of Triplex. Mortality of *S. zeamais* never reached 100% in any Triplex treatments. Adult progeny production of *S. zeamais* was completely suppressed at filter cake concentrations of 700 and 1000 mg/kg, whereas 1000 mg/kg was necessary to observe complete suppression of *S. oryzae* adult progeny production. Complete suppression of adult progeny production was not observed in any Triplex treatments. Complete reduction in percentage of insect damaged kernels and percentage of grain weight loss were obtained when *S. zeamais* and *S. oryzae* adults were exposed to 1000 mg/kg of filter cake;

similar reductions with *S. zeamais* occurred only at 1000 mg/kg of Triplex. In the case of *S. oryzae*, complete reduction of insect damaged kernels and grain weight loss were not achieved at any concentration of Triplex. These powders can be used in lieu of chemical insecticides for management of *Sitophilus* species.

Keywords: Filter cake; Triplex; Wheat; *Sitophilus* species; Efficacy assessment

3.2. Introduction

Food security is a major challenge for low income sub-Saharan Africa farmers (Abate et al., 2015). Factors contributing to food insecurity include low productivity, difficulty adapting to climate change, inability to handle the burden of high food or fuel prices, lack of accessibility to credit, and increased dependence on food aid (Zorya et al., 2011). Food security can be achieved by increasing food production and most importantly by reducing post-harvest losses (Obeng-Ofori, 2011; Zorya et al., 2011). Postharvest losses reduce the amount of food available for consumption by smallholder farmers sub-Saharan Africa (Sheahan and Barrett, 2017). Insect pests and toxigenic molds cause quantitative and qualitative losses to grain in storage at the farmer level (Kadjo et al., 2016). Grain losses due to insect pests in sub-Saharan Africa are very high, and the magnitude of losses varies from country to country and from region to region (Abate et al., 2015). In Ethiopia, about 80% of all grain produced is estimated to be stored at the farm or village level (Tadesse and Eticha, 2000). Grain storage losses due to insect pests were estimated to be in the range of 10 to 21% in Ethiopia (Abraham et al., 2008). The common storage structure used by most Ethiopian farmers are traditional storages with poor construction, which exposes the grain to insect pests (Tadesse and Eticha, 2000; Mezgebe et al., 2016; Hengsdijak and De Boer, 2017). These traditional storages include above-ground structures like *gota* and *gotera*, and underground pits (Tadesse and Eticha, 2000; Blum and Bekele, 2001; Dessalegn et al., 2017). Reducing storage losses will significantly improve food security in countries like Ethiopia (Godfray et al., 2010; van Gogh et al., 2017).

Chemical pesticides, regardless of their inherent hazards are used extensively in the fast changing agricultural sector of Ethiopia (Negatu et al., 2016). Ethiopia is confronted with a number of problems related to unsafe handling of pesticide distribution and use, improper training in safe use of pesticides, and inadequate infrastructure to regulate safe use of pesticides (Mengistie et al., 2017). A survey done by Negatu et al. (2016) on knowledge, attitude, and practices of farmers and farm workers in Ethiopia reported that 85% (*n* = 601) of farm workers (pesticide mixers/loaders, pesticide sprayers, and application supervisors) and 100% (*n* = 275) of female re-entry farm workers (harvesters, pesticide assessors, irrigation workers, irrigation supervisors, packing and sorting workers, transport/push car workers) did not receive any pesticide-related training. In addition, 62% (*n* = 258) of farm workers did not shower after pesticide application, and none of the small-scale farm workers used personal protective equipment and apparel. A considerable increment in chemical pesticide usage intensity, illegitimate usages of pesticides such as DDT and endosulfan on food crops, and direct importation of pesticides without the formal Ethiopian registration process, were also reported by Negatu et al. (2016). A survey of sorghum farmers by Mendesil et al. (2007) showed that 32% (*n* = 138) of farmers surveyed had access to synthetic insecticides to control insect pests of stored sorghum. The pesticides reported by 4% of farmers were pirimiphos-methyl, DDT, and Malathion; 28% of farmers could not tell which pesticides they used on their stored sorghum. A total of 70 out of 100 farmers storing maize in Ethiopia used synthetic insecticides to control stored-product insects, and 66% could not indicate which pesticides they used (Tadesse and Basedow, 2004). DDT, malathion, and pirimiphos-methyl were reported as the pesticides used on maize. In another survey, 106 farmers in Ethiopia were interviewed regarding their on-farm grain storage practices (Blum and Bekele, 2001). Contrary to other surveys, the surveyed farmers had difficulty in using pesticides suggested by extension service personnel for postharvest pest control because they are expensive, not available, or past their shelf life. Mengistie et al. (2017) reported that hazardous and unknown pesticides are common in retail stores in Ethiopia.

Retailers sold all classes of pesticides to farmers irrespective of their suitability or effectiveness. Pesticides are sold illegally by untrained people in the village markets. Additionally, in Ethiopia farmers prefer to buy small amounts of pesticides rather than the original container/package, and therefore there is no information regarding the type of pesticide and how it should be diluted and applied. In countries like Ethiopia and Ghana, unsafe handling of pesticides has resulted in ill health episodes, hospitalization, and fatalities soon after a pesticide application (Williamson et al., 2008).

There is a need to explore products that are safe and effective in controlling insects in smallholder farmer's traditional storages in Ethiopia. Two such products are filter cake and Triplex (Girma et al., 2008a,b; Tadesse and Subramanyam, 2018). Filter cake is a by-product of aluminum sulfate factory (Awash Melkassa Aluminum Sulphate & Sulphuric Acid Share Company, Melkassa Awash, Ethiopia (AMASSASC)). Triplex is a by-product of Mohammed International Development Research and Organization Companies (MIDROC) soap factory (Star Soap and Detergent Industries (SSDI Private Limited Company), Addis Ababa, Ethiopia). A study on elemental composition of both powders using energy-dispersive X-ray spectroscopy indicated that silicon and oxygen were dominant elements (Tadesse and Subramanyam, 2018). The same study showed 100% mortality when adults of maize weevil, *Sitophilus zeamais* Motschulsky were exposed to 7.5 g/m^2 of filter cake and 10 g/m^2 of both powders for 24 h on treated concrete arenas. Girma et al. (2008a) reported 92% mortality 3 d after *S. zeamais* adults were exposed to three genotypes of maize treated with 1, 2.5, and 5% (w/w) of filter cake. Similarly, no significant mean percentage mortality of *S. zeamais* (93%) was observed when adults were exposed to maize treated with 0.25% (w/w) of Triplex compared to that of a synthetic insecticide, pirimiphos-methyl (100% mortality), 7 months after treatment (Girma et al., 2008b). These studies by Girma et al. (2008a,b) and Tadesse and Subramanyam (2018) suggested that filter cake and Triplex could be potential alternatives to chemical insecticides for controlling stored-product insects. There are no data on the efficacy of filter cake and Triplex against *S. zeamais* and the rice weevil, *Sitophilus oryzae* (L.) on wheat. Therefore, this study was designed to determine the efficacy of filter cake and Triplex applied to wheat against adults of *S. zeamais* and *S. oryzae*, which are economically important stored grain pests in Ethiopia (Abraham et al., 2008; Girma et al., 2008a,b; Tilahun and Hussen, 2014).

3.3. Materials and Methods

3.3.1. Application of powders to wheat and insect exposure

Organic hard red winter wheat (Heartland Mills, Marienthal, Kansas, USA) was cleaned manually using a 2.38 mm diameter round-holed aluminum sieve (Seedburo Equipment Company, Des Plaines, Illinois, USA) to remove broken kernels and dockage. The cleaned wheat was frozen at -13°C for five days to kill any live insects present. A 100 g of cleaned, untreated organic wheat with moisture content ~12.5% (wet basis) was transferred to 0.45 L glass jars and covered with filter paper and wire-mesh screen lids to facilitate air diffusion. Filter cake and Triplex concentrations of 0 (untreated wheat), 100, 500, 700 and 1000 mg/kg were added to separate jars and each jar was manually shaken for one minute to ensure uniform distribution of powder on wheat kernels.

Laboratory strains of *S. zeamais* and *S. oryzae* were reared in an environmental growth chamber (Percival Scientific, Inc., Model I-36VL, Perry, Iowa, USA) at 28°C and 65% r.h on organic yellow maize (Heartland Mills, Marienthal, Kansas, USA) of 13.5% moisture content (wet basis) and organic hard red winter wheat with moisture content of 12.0% (wet basis), respectively. These strains have been in rearing for the last 17 years in the Department of Grain Science and Industry, Kansas State University. Twenty unsexed adults of mixed ages (3 to 4 weeks) of laboratory strains of each species were added to separate jars and kept in an environmental growth chamber at 28°C and 65 % r.h. for 7 and 14 d to determine mortality. Separate jars were used for each powder-species-concentration-day combinations with five replications. After a 7 and 14 d exposure, wheat from each container was sifted using a 2.38 mm circular round-holed aluminum sieve to separate insects from wheat. Adults that did not respond when gently prodded by a camel's hair brush were considered as dead. After 7 and 14 d mortality determination, live and dead insects and wheat were returned to the jars and held in environmental chamber at 28°C and 65% r.h. The 7 and 14 d jars were examined after 35 and 28 days, respectively, to count adult progeny produced. The adult progeny produced was counted from each jar after subtracting the original 20 added insects. Wheat from each jar was cleaned and divided five times using a Boerner® divider (Seedburo Equipment Company) to get a working subsample of ~3.2 g for determining undamaged and insect damaged kernels in replicate subsamples. Number of kernels damaged out of the total examined was expressed as a percentage. Percentage of grain weight loss was determined based on counting and weighing damaged and undamaged kernels from each replicate working subsample (Adams and Schulten, 1978; Boxall, 1986). In each replicate control and treated subsamples, percentage of grain weight loss was calculated using the following equation:

Weight loss (%) = $[(UN_d - DN_u) \div (U \times (N_d + N_u))] \times 100$, where, U is the weight of undamaged kernels, N_d is the number of damaged kernels, D is the weight of damaged kernels, and N_u is number of undamaged kernels.

3.3.2. Data analysis

The number of adults that died at 7 and 14 d for each concentration was reported as a percentage. The percentage mean ± SE mortality of *S. zeamais* at 7 and 14 d ranged from 2.0 ± 1.2 to 3.0 ± 2.0 and 1.0 ± 1.0 to 2.0 ± 1.2 in filter cake and Triplex control treatments, respectively. The corresponding percentage mean ± SE mortality of *S. oryzae* ranged from 2.0 ± 1.2 to 2.0 ± 2.0 and 3.0 ± 2.0 to 4.0 ± 1.9 in filter cake and Triplex control treatments, respectively. Therefore, mortality data of each species exposed to filter cake and Triplex treated wheat were corrected for responses in the control treatment (Abbott, 1925). A nonlinear model, $y = a + bx^3 + c/x^2$, was fit to corrected mortality responses (y) as a function of filter cake or Triplex concentrations (x), for 7 and 14 d exposure durations using TableCurve 2D software (Jandel Scientific, San Rafael, California, USA). Similarly a nonlinear model, $\ln y = a + bx$, for both adult progeny production and percentage of insect damaged kernels data, and a nonlinear model, $\ln y = a + bx^{1.5} + cx^{0.5}$, for percentage of grain weight loss data were fit by species and exposure duration. The parameters a, b, and c were estimated by fitting equations to corrected mortality, adult progeny production, percentage of insect damaged kernels, and percentage of grain weight loss data as a function of concentration. Each possible pair-wise comparison between exposure days and powders for each set of data were done by comparing individual models to a pooled model (Draper and Smith, 1998). Any two models being compared were considered to be significantly different ($P < 0.05$) from one another if the *F*-test showed individual models to deviate from the pooled model. The 7 and 14 d data were pooled for each powder and used to compare efficacy of each powder between the two species. Five out of the 16 comparisons between 7 and 14 d were significant. However, pooling 7 and 14 d data helped us to compare the efficacy between the two *Sitophilus* species. All data were analyzed using SAS software 9.4 (SAS Institute, 2012). Graphs were plotted using SigmaPlot, version 12.5 (Systat Software, Inc., San Jose, California USA).

3.4. Results

3.4.1. Concentration and mortality responses of S. zeamais and S. oryzae adults

Mortality of *S. zeamais* reached 100% when adults were exposed to 1000 mg/kg of filter cake for 7 or 14 d. However, 100% mortality of *S. zeamais* was not achieved at any concentration and exposure time for Triplex treatments. The relationship between mortality and concentration was satisfactorily described by the nonlinear model for filter cake (r^2 = 0.94 for 7 d and 0.99 for 14 d) and Triplex (r^2 = 0.98 for 7 d and 0.99 for 14 d) (Fig. 3.1). Mortality responses of *S. zeamais* exposed to filter cake for 7 and 14 d were not significantly different from one another (F = 5.39; df = 1, 4; P = 0.0810), whereas those exposed to Triplex were significantly different (F = 19.71; df = 1, 4; P = 0.0113). The mortality responses of *S. zeamais* exposed to filter cake for 14 d were significantly greater than those exposed to Triplex (F = 45.24; df = 1, 4; P = 0.0026). However, adults exposed to filter cake and Triplex for 7 d were not significantly different from one another (F = 3.80; df = 1, 4; P = 0.1230).

Mortality of *S. oryzae* was 100% when adults were exposed for 14 d to 700 and 1000 mg/kg of filter cake. Mortality was 100% only for adults exposed for 7 d to 1000 mg/kg of filter cake. Complete mortality of *S. oryzae* was achieved only after a 14 d exposure to 1000 mg/kg of Triplex. The relationship between mortality and concentration was nonlinear. The fitted models satisfactorily described the mortality and concentration data for filter cake (r^2 = 0.94 for 7 d and 0.99 for 14 d) and Triplex (r^2 = 0.97 for 7 d and 0.99 for 14 d) (Fig. 3.2). Mortality responses of *S. oryzae* exposed to filter cake for 7 and 14 d were not significantly different from one another

(*F* = 3.90; df = 1, 4; *P* = 0.1195), whereas those exposed to Triplex were significantly different (*F* = 33.11; df = 1, 4; *P* = 0.0045). The mortality of *S. oryzae* exposed to filter cake for 14 d was significantly greater than those exposed to Triplex (*F* = 31.22; df = 1, 4; *P* = 0.0050). However, adults exposed to filter cake and Triplex for 7 d were not significantly different from one another (*F* = 4.38; df = 1, 4; *P* = 0.1046).

Models fitted to 7 and 14 d pooled mortality data of *S. zeamais* (r^2 = 0.92) and *S. oryzae* (r^2 = 0.96) exposed to filter cake were not significantly different from one another (*F* = 0.06; df = 3, 10; *P* = 0.9789). Similarly, there were no significant differences between *S. zeamais* (r^2 = 0.92) and *S. oryzae* (r^2 = 0.96) exposed to Triplex (*F* = 0.28; df = 3, 10; *P* = 0.8376) (Fig. 3.3). In general, filter cake was more efficacious to *S. zeamais* and *S. oryzae* adults than Triplex.

3.4.2. Adult progeny production at 42 d

Adult progeny production was completely suppressed when *S. zeamais* adults were exposed to 700 and 1000 mg/kg of filter cake for 7 and 14 d. However, 100% suppression of progeny production was not achieved in any of the concentrations and exposure times for Triplex. Number of progeny produced decreased with increasing powder concentration. Decrease in progeny production with increased concentration was nonlinear, and fitted models satisfactorily described the progeny and concentration data of filter cake (r^2 = 0.98 for 7 d and 0.99 for 14 d) and Triplex (r^2 = 0.96 for 7 d and 0.98 for 14 d) (Fig. 3.4). Adult progeny production did not vary between exposure times and between the two powders (*F*, range = 1.36 – 5.11; df = 2, 6; *P*, range = 0.0508 – 0.3255).

Progeny production was completely suppressed when *S. oryzae* adults were exposed to 1000 mg/kg of filter cake for 7 and 14 d. However, complete suppression of progeny production was not achieved in any of the concentrations and exposure times for Triplex. Number of progeny produced decreased with increasing powder concentrations. The decrease in progeny production as a function of concentration was nonlinear. The fitted models satisfactorily described progeny production and concentration data of filter cake ($r^2 = 0.90$ for 7 d and 0.99 for 14 d) and Triplex ($r^2 = 0.97$ for 7 d and 0.90 for 14 d) (Fig. 3.5). Progeny production did not vary between the two exposure times ($F = 1.05$; df = 2, 6; $P = 0.4063$ for filter cake and $F = 0.21$; df = 2, 6; $P = 0.8126$ for Triplex), whereas progeny production was significantly reduced when adults of *S. oryzae* were exposed to filter cake than those exposed to Triplex ($F = 17.44$; df = 2, 6; $P = 0.0032$ for 7 d, and $F = 15.82$; df = 2, 6; $P = 0.0041$ for 14 d).

The nonlinear models fitted to 7 and 14 d pooled progeny production data of *S. zeamais* ($r^2 = 0.99$) and *S. oryzae* ($r^2 = 0.93$) exposed to filter cake were not significantly different from one another ($F = 0.53$; df = 2, 16; $P = 0.5991$). However, nonlinear models fitted to pooled progeny production data of *S. oryzae* ($r^2 = 0.93$) when exposed to Triplex was significantly greater ($F = 58.64$; df = 2, 16; $P < 0.0001$) than that of *S. zeamais* ($r^2 = 0.96$) (Fig. 3.6). Progeny production of both species was inversely related to the concentration of the two powders. This is attributed to greater mortality at higher concentrations.

3.4.3. Insect damaged kernels at 42 d

The percentage of insect damaged kernels was 0% when *S. zeamais* adults were exposed to 700 and 1000 mg/kg of filter cake for 7 and 1 14 d. There were no insect damaged kernels when adults were exposed to 1000 mg/kg of Triplex for 14 d. Insect damaged kernels decreased with an increase in powder concentration. The decrease in insect damaged kernels as a function of concentration was nonlinear. The fitted models satisfactorily described the relationship between insect damaged kernels and concentration data of filter cake ($r^2 = 0.99$ for both 7 and 14 d) and Triplex ($r^2 = 0.88$ for 7 and 0.97 for 14 d) (Fig. 3.7). Insect damaged kernels were significantly greater when adults of *S. zeamais* were exposed to filter cake for 7 d than those exposed for 14 d ($F = 6.98$; df = 2, 6; $P = 0.0272$), whereas insect damaged kernels did not vary between the exposure times in the case of Triplex ($F = 0.48$; df = 2, 6; $P = 0.6401$).

Insect damaged kernels were not observed when *S. oryzae* adults were exposed to 1000 mg/kg of filter cake for 7 and 14 d. However, the insect damaged kernels ranged from 2.6 – 39.0% at all Triplex concentrations and exposure times. The relationship between insect damaged kernels and concentration was nonlinear. The fitted models satisfactorily described insect damaged kernels and concentration data of filter cake ($r^2 = 0.88$ for 7 d and 0.95 for 14 d) and Triplex ($r^2 = 0.99$ for 7 d and 0.96 for 14 d) (Fig. 3.8). Insect damaged kernels were not significantly different between exposure times ($F = 0.51$; df = 2, 6; $P = 0.6259$ for filter cake, and $F = 0.16$; df = 2, 6; $P = 0.8570$ for Triplex). Insect damaged kernels were significantly greater when adults of *S. oryzae* were exposed for 7 and 14 d to Triplex than those exposed to filter cake ($F = 21.45$; df = 2, 6; $P = 0.0019$ for 7 d, and $F = 19.34$; df = 2, 6; $P = 0.0024$ for 14 d).

The models for pooled 7 and 14 d insect damaged kernels caused by *S. zeamais* ($r^2 = 0.98$) and *S. oryzae* ($r^2 = 0.90$) exposed to filter cake were not significantly different from one another ($F = 0.96$; df = 2, 16; $P = 0.4033$). However, the models for pooled 7 and 14 d insect damaged kernels caused by *S. oryzae* ($r^2 = 0.97$) exposed to Triplex was significantly greater ($F = 43.47$; df = 2, 16; $P < 0.0001$) than those caused by *S. zeamais* ($r^2 = 0.91$) (Fig. 3.9). There was an inverse relationship between the percentage of insect damaged kernels and concentration of filter cake and Triplex for both species.

3.4.4. Grain weight loss at 42 d

The percentage of grain weight loss was 0% when *S. zeamais* adults were exposed to 700 and 1000 mg/kg of filter cake for 7 and 14 d. Grain weight loss was also 0% when adults were exposed to1000 mg/kg of Triplex for 14 d. Grain weight loss decreased with increasing powder concentration. The decrease in grain weight loss as a function of concentration was nonlinear. The fitted models satisfactorily described the relationship between grain weight loss and concentration data of filter cake ($r^2 = 0.99$ for both 7 and 14 d) and Triplex ($r^2 = 0.99$ for both 7 and 14 d) (Fig. 3.10). Grain weight loss was significantly higher when adults of *S. zeamais* were exposed to filter cake ($F = 13.87$; df = 3, 4; $P = 0.0140$) and Triplex ($F = 10.35$; df = 3, 4; $P = 0.0235$) for 7 d than those exposed to 14 d. Grain weight loss was significantly greater when adults of *S. zeamais* were exposed to Triplex than those exposed to filter cake ($F = 19.28$; df = 3, 4; $P = 0.0077$ for 7 d and $F = 109.72$; df = 3, 4; $P = 0.0003$ for 14 d).

Grain weight loss was 0% when *S. oryzae* adults were exposed to 1000 mg/kg of filter cake for 7 and 14 d. However, grain weight loss ranged from 0.5 -19.6% at all Triplex concentrations and exposure times. The relationship between grain weight loss as a function of filter cake and Triplex concentration was nonlinear. The fitted models satisfactorily described the relationship between grain weight loss and concentration data of filter cake ($r^2 = 0.99$ for both 7 and 14 d) and Triplex ($r^2 = 0.99$ for 7 d and 0.95 for 14 d) (Fig. 3.11). Grain weight loss did not vary between exposure times ($F = 2.33$; df = 3, 4; $P = 0.2161$ for filter cake, and $F = 0.18$; df = 3, 4; $P = 0.9054$ for Triplex), whereas the grain weight loss was significantly greater when adults of *S. oryzae* were exposed for 7 and 14 d to Triplex than those exposed to filter cake ($F = 96.26$; df = 3, 4; $P = 0.0004$ for 7 d, and $F = 14.00$; df = 3, 4; $P = 0.0138$ for 14 d).

The models for pooled 7 and 14 d grain weight loss caused by *S. zeamais* ($r^2 = 0.98$) exposed to filter cake was significantly greater ($F = 13.70$; df = 3, 14; $P = 0.0002$) than those caused by *S. oryzae* ($r^2 = 0.99$). However, the models for pooled 7 and 14 d weight loss caused by *S. oryzae* ($r^2 = 0.95$) exposed to Triplex was significantly greater ($F = 43.47$; df = 3, 14; $P < 0.0001$) than those caused by *S. zeamais* ($r^2 = 0.99$) (Fig. 3.12). There was an inverse relationship between the percentage of grain weight loss and concentration of filter cake and Triplex for both species.

3.5. Discussion

Powdered insecticides were extensively used to protect stored grain from insect pests for a long period of time (Headlee, 1924; Ebeling, 1971; Subramanyam and Roesli, 2000). One group of these insecticides is silica- based powders, which have toxic effect on insects due to their ability to adsorb lipids from the insect cuticle leading to death by desiccation (Ebeling, 1971; Subramanyam and Roesli, 2000; Malia et al., 2016a). We hypothesize that filter cake and Triplex have similar properties to this group of insecticides because of their higher atomic percentage of silicon and oxygen content (Tadesse and Subramanyam, 2018). The proportion of silicon and oxygen as silicon dioxide in both filter cake and Triplex was smaller than that found in diatomaceous earth powders, and therefore, our result cannot be compared directly with the work done on diatomaceous earth powders by numerous researchers (Jean et al., 2015; Kljajić et al., 2010; Nukenine et al., 2010; Arthur, 2002). Complete mortality of *S. zeamais* and *S. oryzae* adults was observed at a concentration of 700 mg/kg of filter cake and at a concentration of 1000 mg/kg of both powders after a 14 d exposure. The concentrations of filter cake and Triplex used in this study are 5.6–11.7 times greater than the labeled concentrations of diatomaceous earth formulation of Protect-It^{TM} (60–180 mg/kg), recommended for 13% moisture content of grains (Subramanyam and Roesli, 2000). Girma et al. (2008a) reported 100% mortality of *S. zeamais* 15 d after exposure to maize treated with filter cake at concentrations of 10, 25 and 50 g/kg. In another study, Girma et al., (2008b) treated 50 kg of maize with 2.5 g/kg of Triplex and sampled maize at 1, 3, 5, and 7 months. They reported 93% mortality of *S. zeamais* in samples collected on month 7. Girma et al. (2008b) findings using Triplex from the same source are contrary to findings in our study, where we found filter cake to be more efficacious than Triplex. The same concentration of the two powders should be used to determine which of the powders is more efficacious against a given insect pest. Differences in toxicity depending on commodity were observed in several reports regardless of the type and concentration of insecticides used (Athanassiou et al., 2003; Athanassiou et al., 2008; Chintzoglou et al., 2008; Vassilakos et al., 2015). Therefore, our research result on wheat cannot be directly compared with the research done by Girma et al. (2008a,b) on maize.

Our data demonstrated that the mortality of both species exposed to filter cake and Triplex increased with increasing concentration and exposure time. Admixing of powder with wheat was done manually in our experiment, where uneven distribution of powder over the wheat kernel surface was more likely to occur. The insecticidal action or toxicity of such powders will depend on particle accumulation from the wheat to insect’s body and adsorption of lipid by powder particles from the insect cuticle (Le Patourel et al., 1989; Filipović et al., 2010). Accumulation of powder over insect’s body is directly proportional to concentration of the powder (Le Patourel et al., 1989), and insect behavior such as mobility (Malia et al., 2016b). Therefore, uneven distribution of filter cake and Triplex on wheat might have contributed to unexplained variation in mortality responses of both species. For both species, filter cake treatments produced greater mortality than Triplex.

The greater mortality of both species with increasing concentration of filter cake and Triplex caused significant reduction in adult progeny production, percentage of number of damaged kernels, and percentage of grain weight loss. A similar phenomenon has been reported by Tadesse and Subramanyam (2018) for both powders tested on concrete surfaces. Although adult weevils were killed by exposure to filter cake and Triplex, some oviposition might still occur, and therefore, progeny production was observed especially at the lower powder concentrations tested. A similar phenomenon was reported by Athanassiou et al. (2005) when *S. oryzae*, and the confused flour beetle, *Tribolium confusum* Jacquelin du Val, were exposed for 14 d to wheat treated with 0.25, 0.5, 1 and 1.5 g/kg concentrations of the diatomaceous earth SilicoSec® at 22ºC. Adult progeny production was recorded for both species exposed to 0.25 and 0.5 g/kg of SilicoSec®, which was attributed to less than 50% mortality of the originally exposed adult insects.

The overall results of our study showed that filter cake was consistently efficacious against *S. zeamais* and *S. oryzae* than Triplex. This could be related to its carbon content (Mean ± SE atomic percentage = 39.43 ± 12.63, *n* = 6) in the form of calcium carbonate (Tadesse and Subramanyam, 2018). Calcium carbonate at 1 and 2% (w/w) on maize was reported by Silva et al. (2004) to cause 70.2 and 84.2% mortality of *S. zeamais* adults, respectively, after a 15 d exposure. Both filter cake and Triplex powders were highly effective at 700 and 1000 mg/kg concentrations after a 14 d of exposure under controlled laboratory conditions. However, traditional storages in Ethiopia, such as *gota* and *gotera* are made with teff straw, mud, and cow dung (Abraham et al., 2008). A survey conducted in three major grain producing areas of

Ethiopia indicated that 93.3% (*n* = 300) used traditional storages, which predispose stored grains to insect pests (Gabriel and Hundie, 2006). A recent survey by Dessalegn et al. (2017) in four major grain producing states in Ethiopia (*n* = 200) estimated post-harvest losses of wheat during various stages of handling to range from 14-23%. These estimates included losses of grains in traditional storage structures due to insect pests. Therefore, including filter cake and Triplex in the integrated pest management practices in smallholder farmers' traditional storage structures will be useful to protect wheat from *S. zeamais* and S. *oryzae* infestations. After further studies on real Ethiopian field situation, filter cake and Triplex should be recommended for protecting wheat stored by smallholder farmers in Ethiopia to discourage farmers from using dangerous chemical insecticides.

3.6. References

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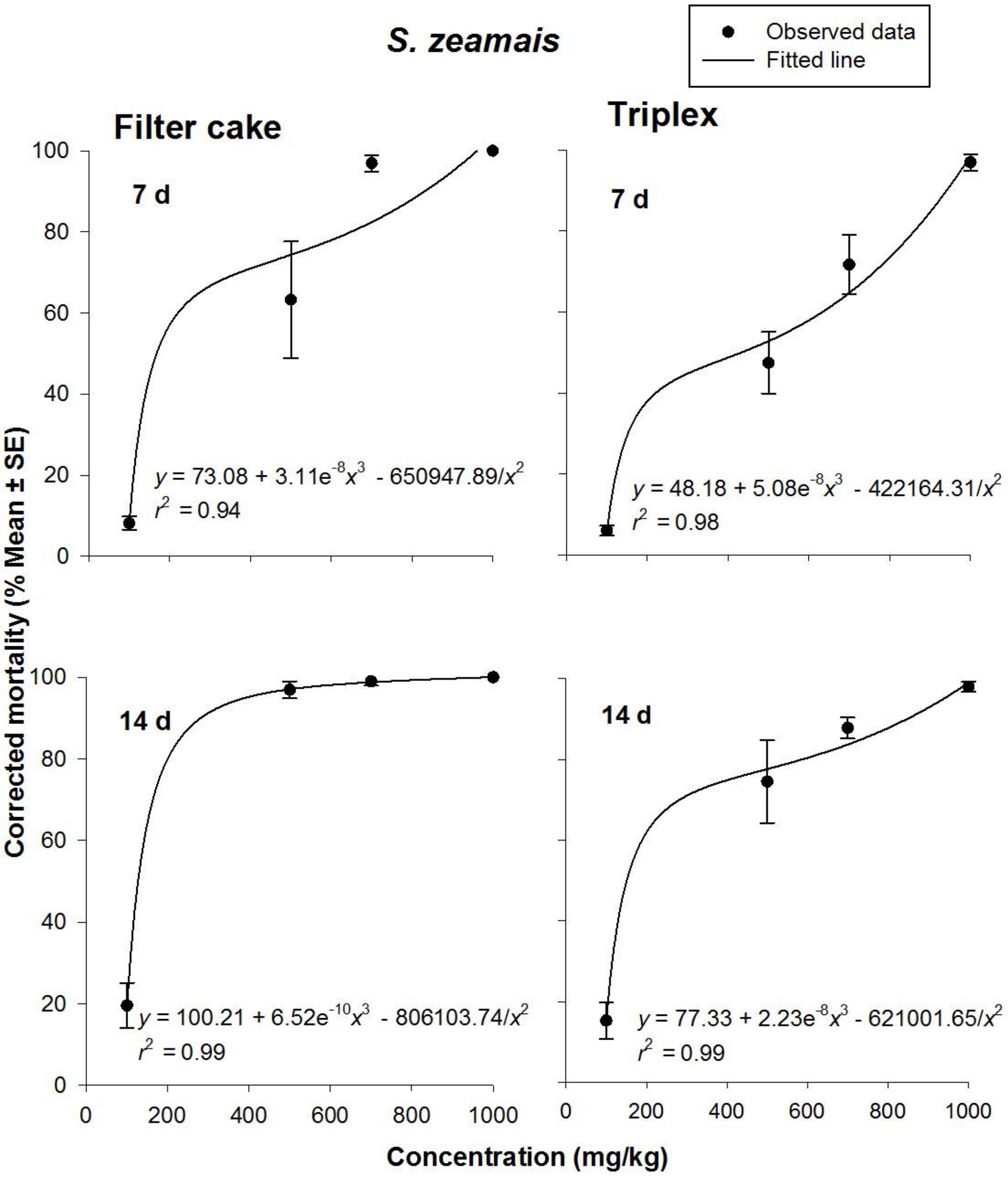

Figure 3.1. Corrected 7 and 14 d mortality data of *S. zeamais* adults after exposure to filter cake and Triplex treated wheat at concentrations of 100, 500, 700, and 1000 mg/kg.

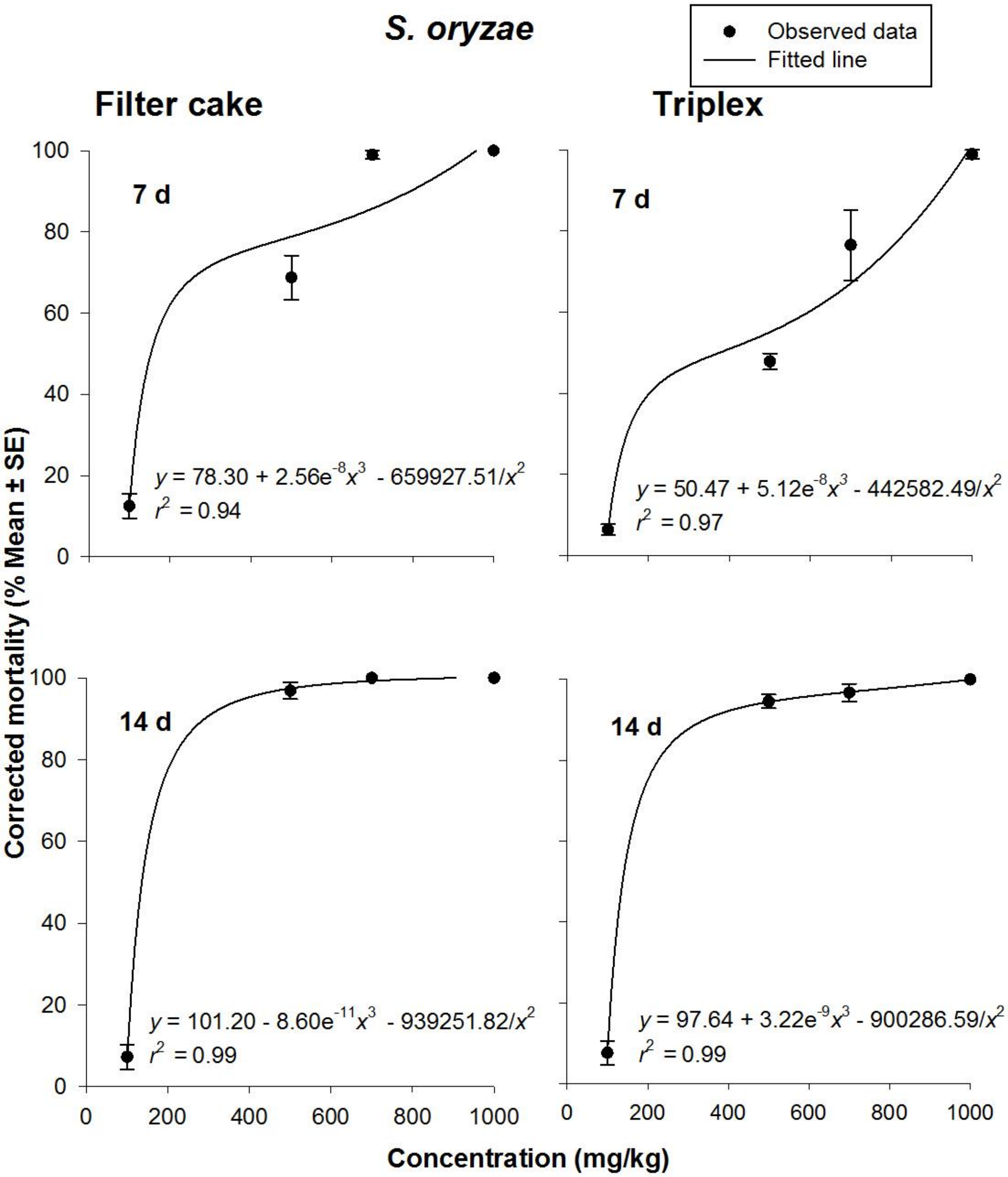

Figure 3.2. Corrected 7 and 14 d mortality data of *S. oryzae* adults after exposure to filter cake and Triplex treated wheat at concentrations of 100, 500, 700, and 1000 mg/kg.

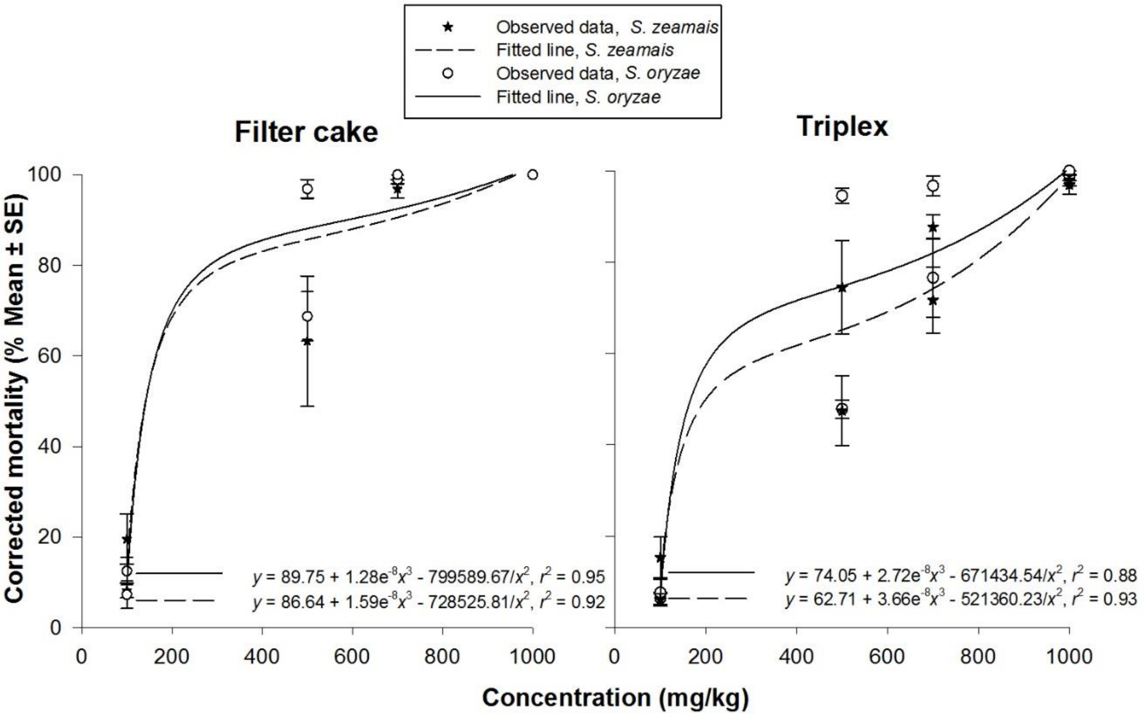

Figure 3.3. Corrected 7 and 14 d pooled mortality data of *S. zeamais* and *S. oryzae* adults after exposure to filter cake and Triplex treated wheat at concentrations of 100, 500, 700, and 1000 mg/kg.

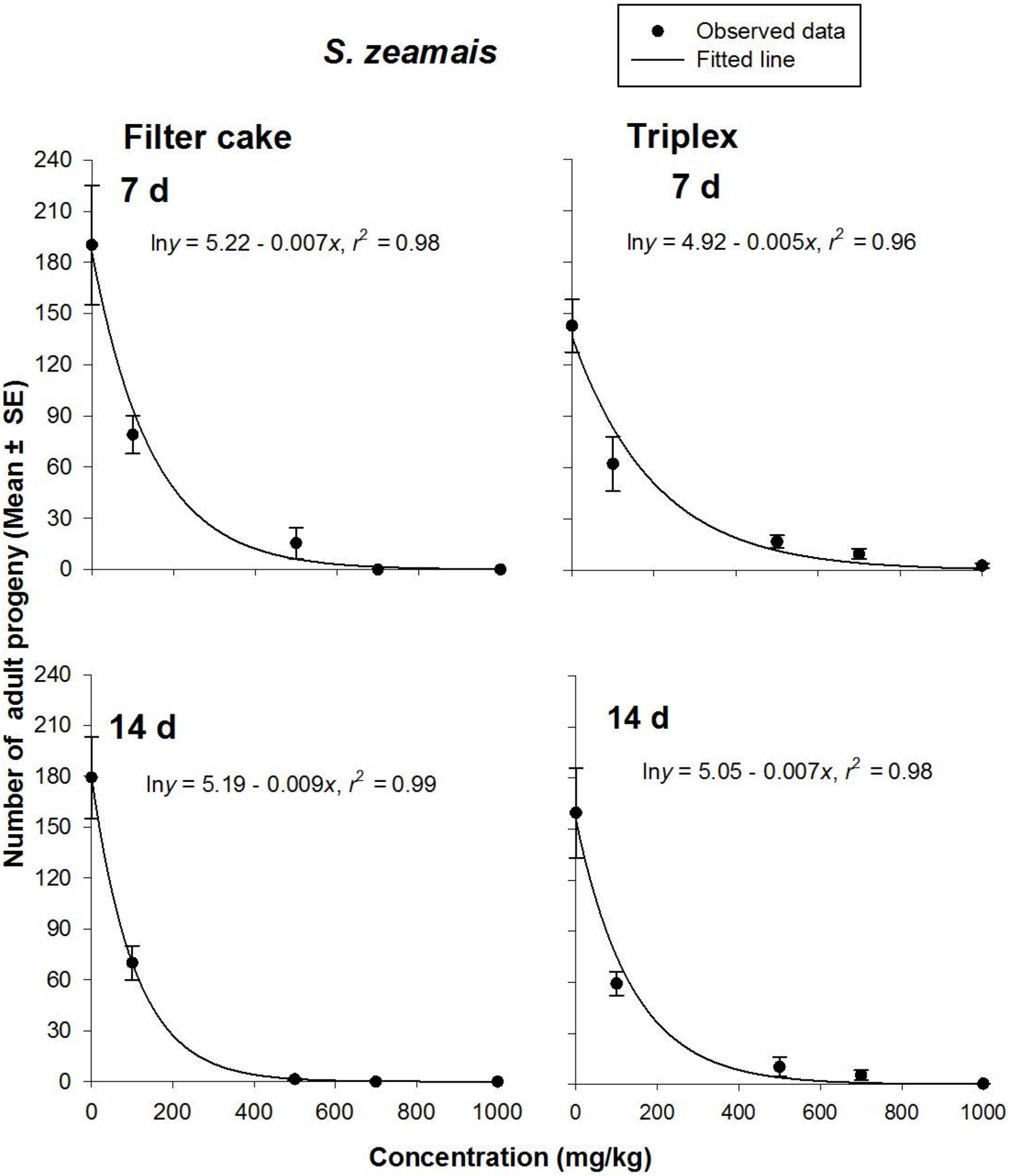

Figure 3.4. Number of adult progeny of *S. zeamais* produced at 42 d after a 7 and 14 d exposure to filter cake and Triplex treated wheat at concentrations of 0, 100, 500, 700 and 1000 mg/kg.

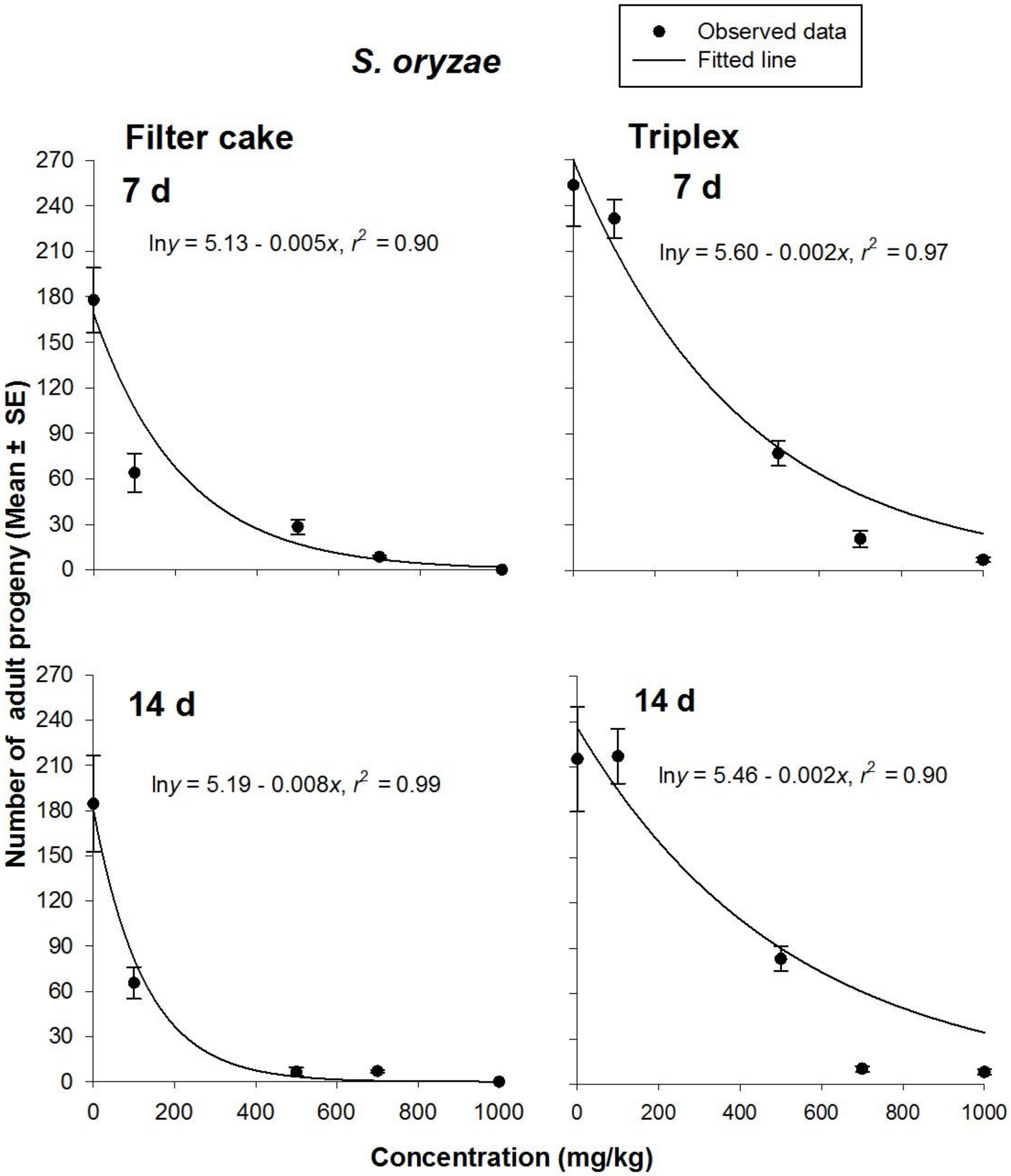

Figure 3.5. Number of adult progeny of *S. oryzae* produced at 42 d after a 7 and 14 d exposure to filter cake and Triplex treated wheat at concentrations of 0, 100, 500, 700 and 1000 mg/kg.

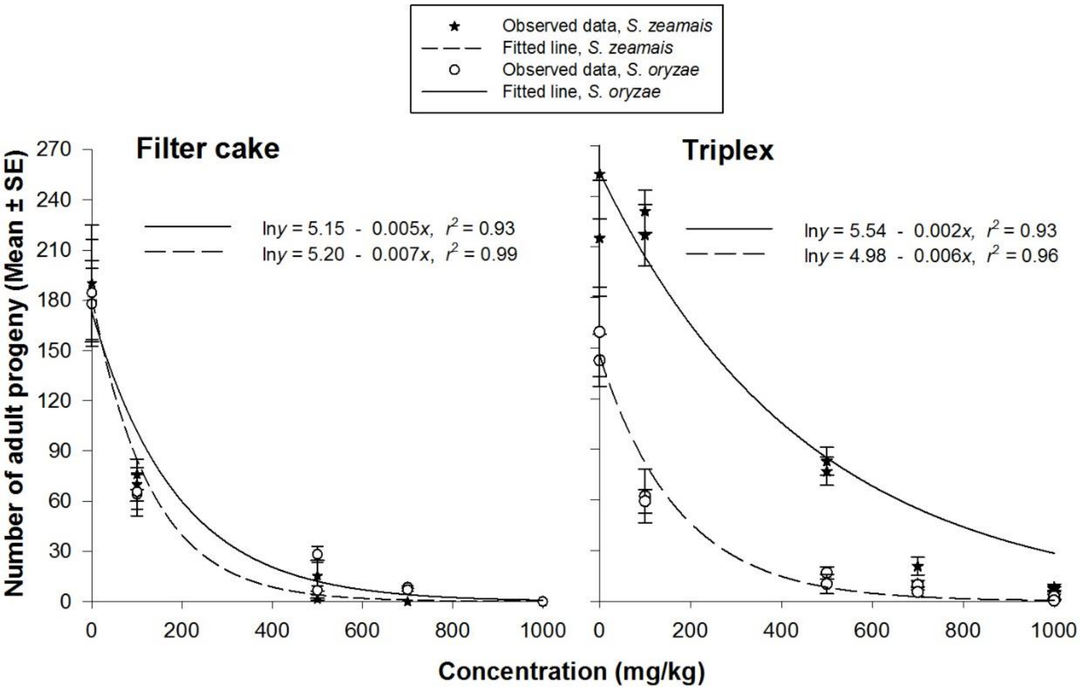

Figure 3.6. Pooled 7 and 14 d adult progeny production data of *S. zeamais* and *S. oryzae* at 42 d after exposure to filter cake and Triplex treated wheat at concentrations of 0, 100, 500, 700 and 1000 mg/kg.

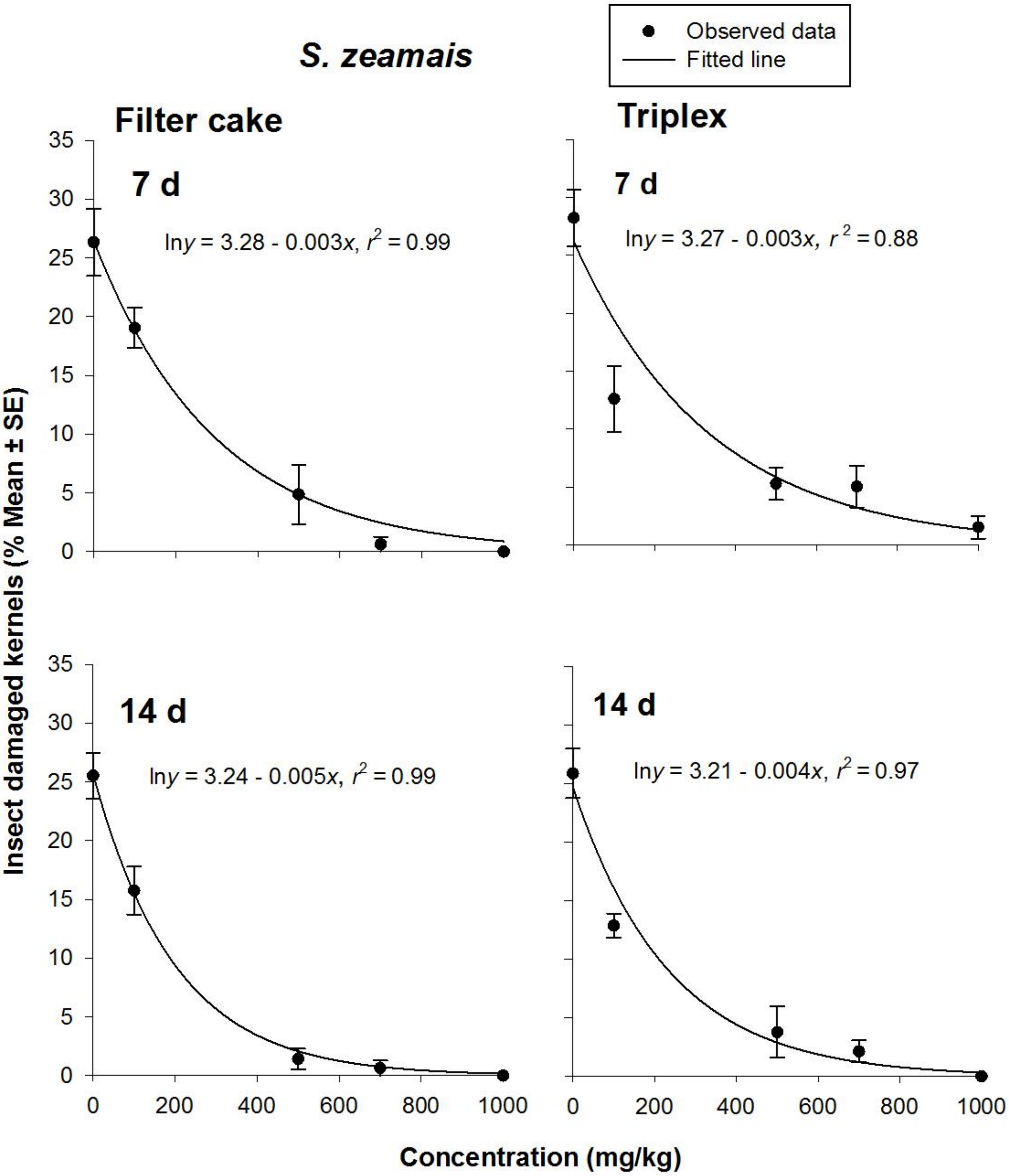

Figure 3.7. Percentage of kernels damaged by *S. zeamais* at 42 d after a 7 and 14 d exposure to filter cake and Triplex treated wheat at concentrations of 0, 100, 500, 700 and 1000 mg/kg.

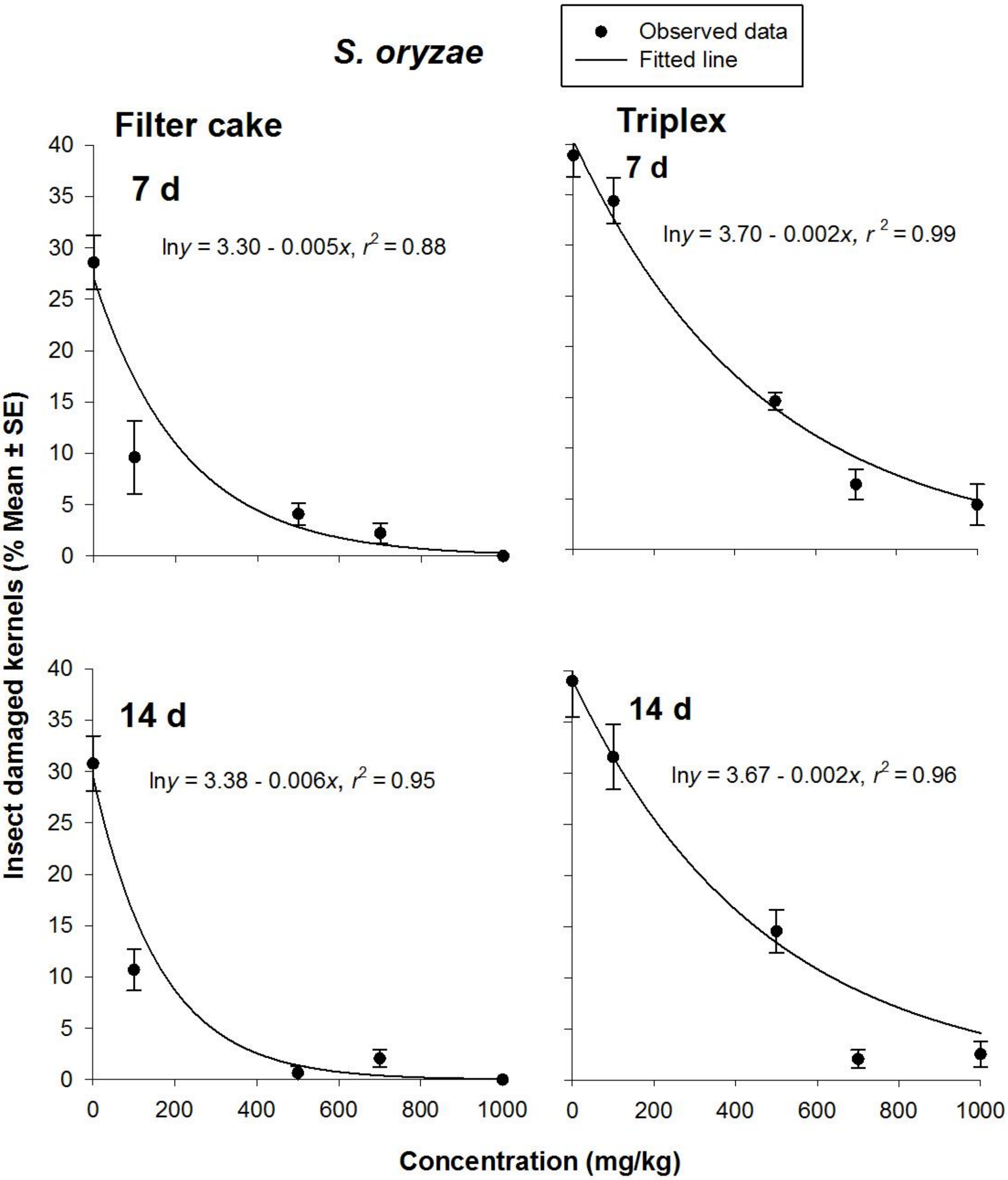

Figure 3.8. Percentage of kernels damaged by *S. oryzae* at 42 d after a 7 and 14 d exposure to filter cake and Triplex treated wheat at concentrations of 0, 100, 500, 700 and 1000 mg/kg.

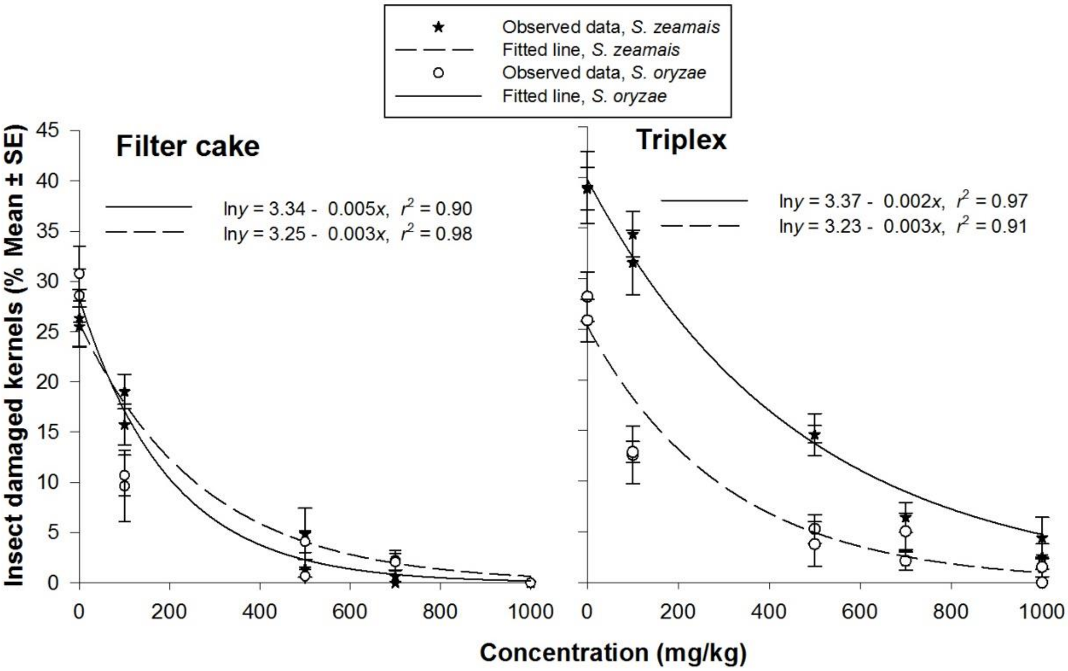

Figure 3.9. Pooled 7 and 14 d data for percentage of kernels damaged by *S. zeamais* and *S. oryzae* at 42 d after exposure to filter cake and Triplex treated wheat at concentrations of 0, 100, 500, 700 and 1000 mg/kg.

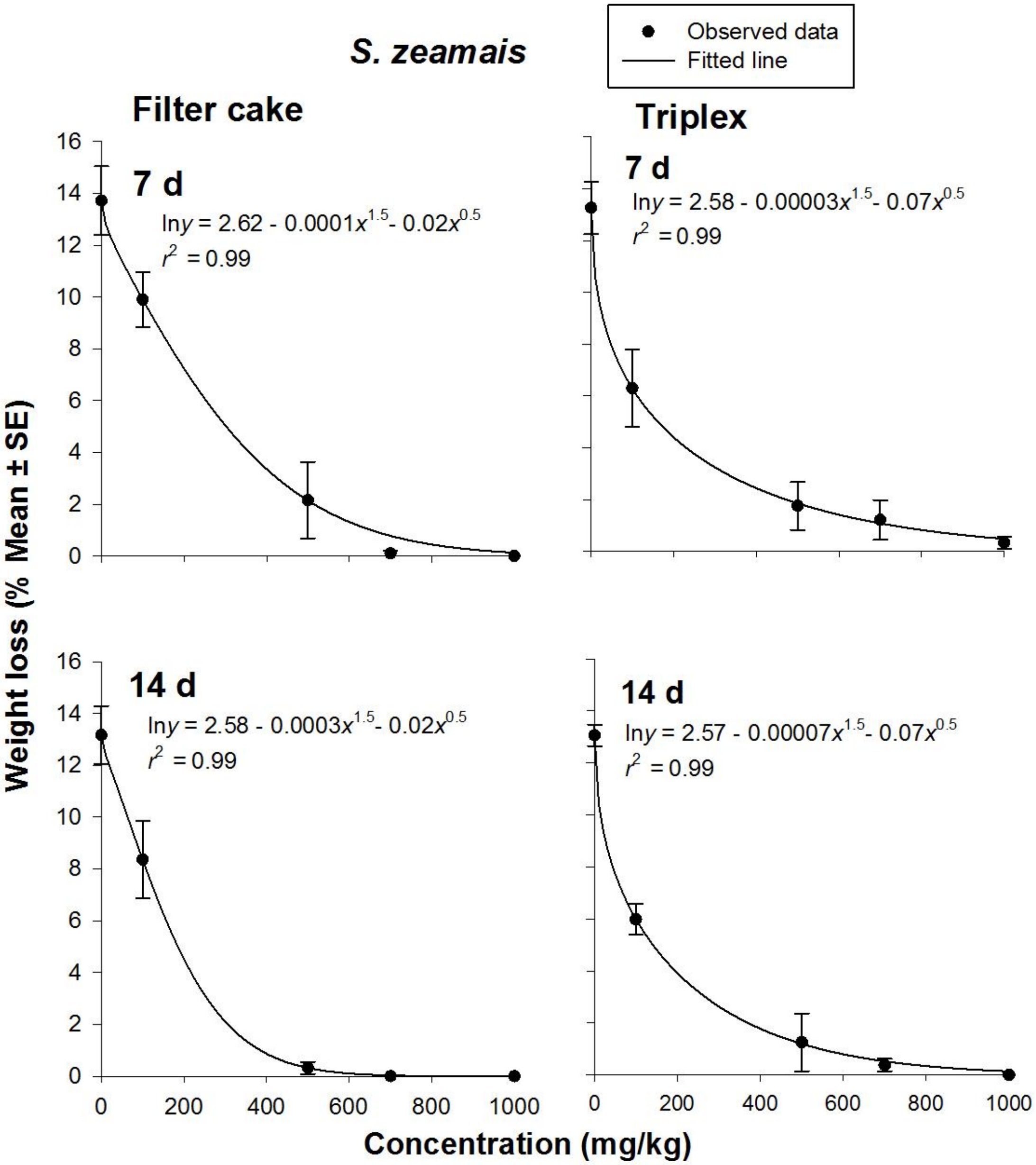

Figure 3.10. Percentage of grain weight loss at 42 d caused by *S. zeamais* after a 7 and 14 d exposure to filter cake and Triplex treated wheat at concentrations of 0, 100, 500, 700 and 1000 mg/kg.

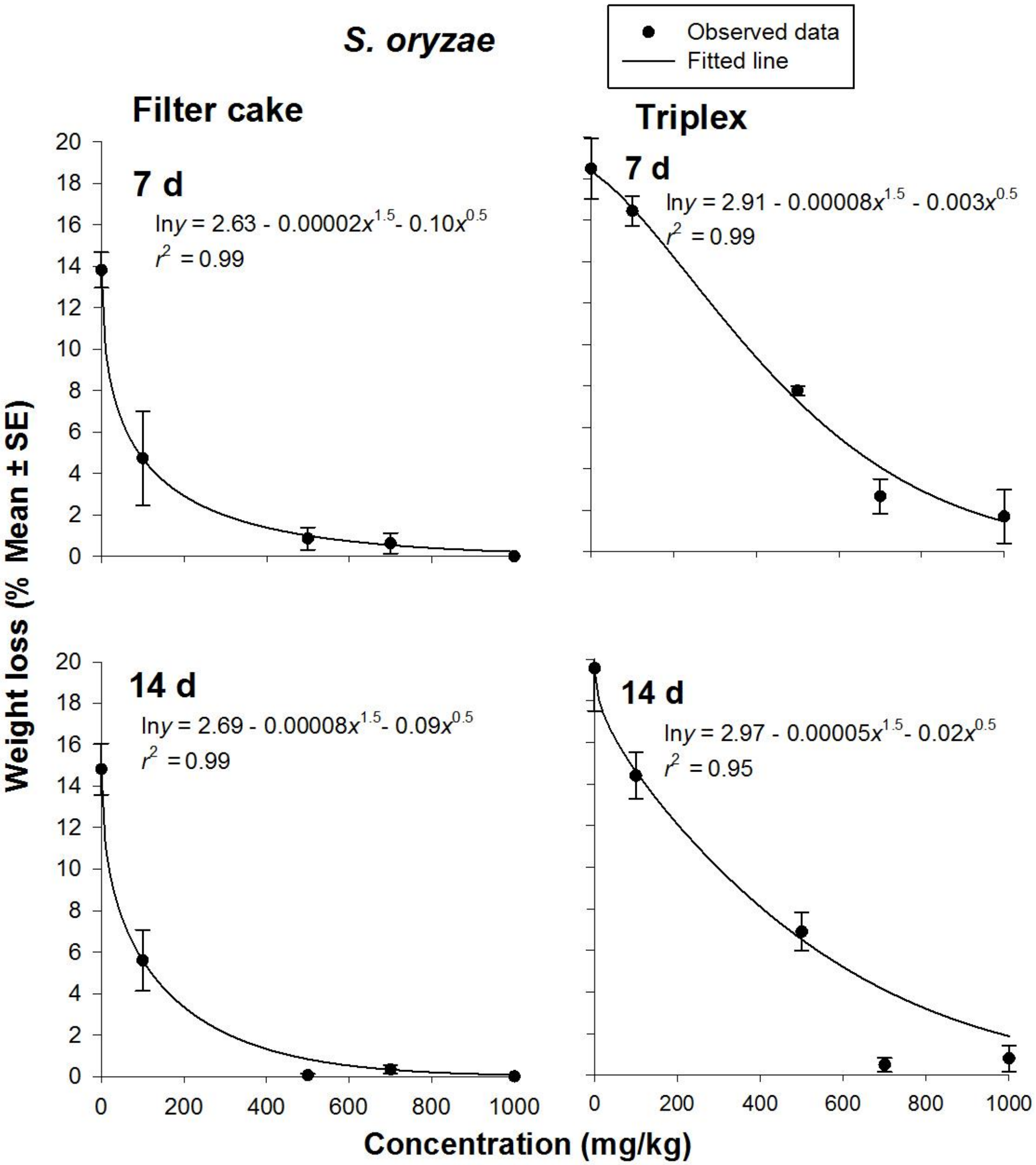

Figure 3.11. Percentage of grain weight loss at 42 d caused by *S. oryzae* after a 7 and 14 d exposure to filter cake and Triplex treated wheat at concentrations of 0, 100, 500, 700 and 1000 mg/kg.

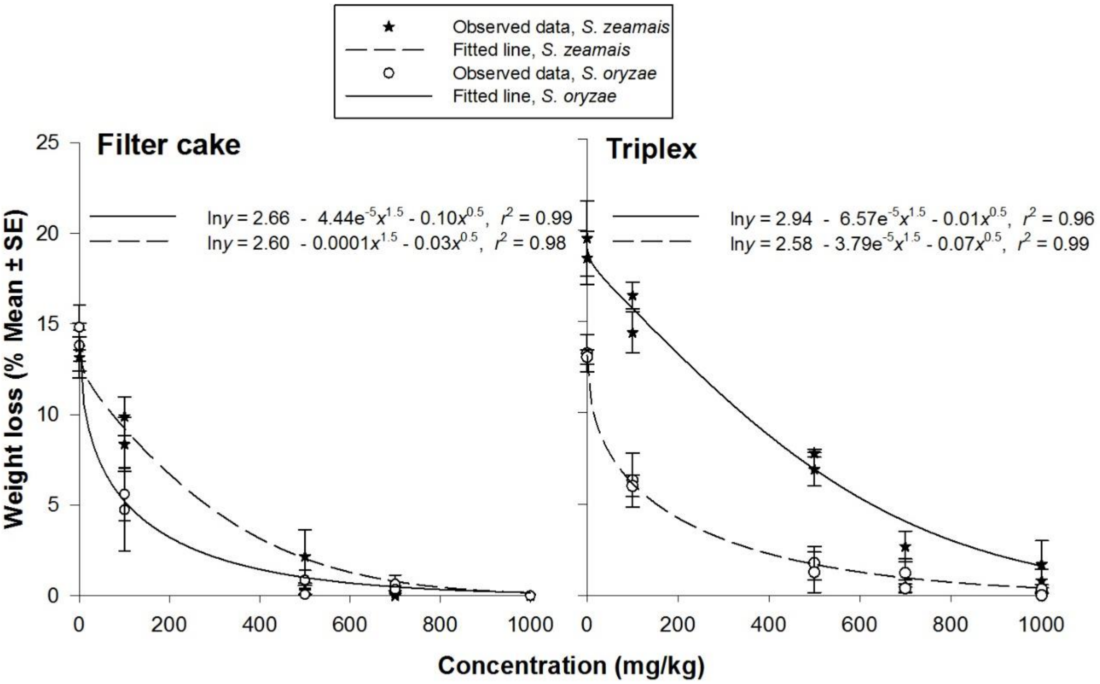

Figure 3.12. Pooled 7 and 14 d data for percentage of grain weight loss caused by *S. zeamais* and *S. oryzae* at 42 d after exposure to filter cake and Triplex treated wheat at concentrations of 0, 100, 500, 700 and 1000 mg/kg.

Chapter 4 - Contact Toxicity of filter cake and Triplex powders from Ethiopia against adults of *Sitophilus zeamais* Motschulsky (Coleoptera: Curculionidae)

4.1. Abstract

Filter cake and Triplex are powdered by-products of aluminum sulfate and soap factories in Ethiopia, respectively. This study was aimed at determining contact toxicity of filter cake and Triplex powders against maize weevil, *Sitophilus zeamais* Motschulsky. Lethal concentrations for 99% mortality (LC_{99}) against *S. zeamais* were determined by exposing adults for 12 h to filter cake (0.5–8 g/m^2) and Triplex (1–9 g/m^2) in concrete arenas. Lethal times for 99% mortality (LT_{99}) were determined by exposing adults over time (1–24 h) in concrete arenas to 3 g/m^2 of filter cake and 9 g/m^2 of Triplex. Exposed adults were transferred to containers with 30 g of organic wheat and held at 28°C and 65% RH for 14 d to determine mortality. LC_{99} values for *S. zeamais* adults were 7.54 and 23.46 g/m^2 when exposed to filter cake and Triplex, respectively. The corresponding LT_{99} values were 21.92 and 39.62 h when exposed to filter cake and Triplex, respectively. Effective concentrations and times for the 99% reduction of progeny production were determined from percentage reduction in adult progeny relative to production in control treatments after 42 d. EC_{99} values for progeny reduction were 2.48 and 18.59 g/m^2 for filter cake and Triplex treatments, respectively. The corresponding ET_{99} values for progeny reduction were 17.49 and 22.31 h for filter cake and Triplex, respectively. *S. zeamais* exposed to filter cake produced lower percentage insect damaged kernels and weight loss than Triplex. Filter cake was more efficacious against *S. zeamais* than Triplex.

Keywords: Ethiopia, Filter cake; Triplex; *Sitophilus zeamais*; Efficacy assessment

4.2. Introduction

Chemical pesticides are used extensively in the Ethiopian agricultural sector (Negatu et al., 2016). A number of challenges related to unsafe handling, importation, distribution and use of pesticides in Ethiopia were reported by Negatu et al. (2016) and Mengistie et al. (2017). Information about environmental health and personnel safety, efficacy, and safe use of pesticides were not provided by most of the importers (Mengistie, 2016). The pesticide market is heavily dependent on imports by local agents representing international manufacturing and formulating companies, and is dominated by immediate profit motives (Mengistie et al., 2015). This led to human and environmental health problems (Loha et al., 2018). Pesticide regulators are not carrying out their tasks in conformity with the power given in the pesticide registration and control proclamation number 647/210 (Mengistie et al., 2015, Mengistie, 2016, Mengistie et al., 2017). Lack of information and resources, weak interaction and lack of motivation of state actors, and lack of technical knowledge in safe handling of pesticides have caused improper and misuse of pesticides by end users (Mengistie et al., 2017, Loha et al., 2018). Many cases of intentional and unintentional pesticide poisoning have been reported in Ethiopia (Desalew et al., 2011, Abebe, 1991). Intentional poisonings were a result of people committing suicides. In Ethiopia, unsafe handling of pesticides has resulted in ill health episodes, hospitalizations, and fatalities soon after a pesticide application (G/Mariam and Gelaw, 2016, Williamson et al., 2008).

In a meta-analytical study, Affognon et al. (2015) reviewed 213 documents in sub-Saharan Africa regarding postharvest losses and reported that 139 of the documents estimated the losses either as weight of edible mass or volume of commodity discarded due to damage or spoilage. They indicated that 80.4% of estimated losses were related to storage. In the same study, 28 out of 213 documents reported quality losses in terms of price discounts, nutritional value loss, or volume of downgraded produce. In Ethiopia postharvest losses are a major issue in smallholder farmers' traditional storage structures (Sheahan and Barrett, 2017). Insect pests and toxigenic molds cause quantitative and qualitative losses to grain in storage at the farmer level in Ethiopia, and the magnitude of losses are in the range of 20 to 30% (Tefera et al., 2011). In Ethiopia, smallholder farmers use synthetic pesticides without proper training on handling, application, and disposal to reduce grain postharvest losses (Mengistie et al., 2017). A number of chemical pesticides such as organophosphates and carbamates are used by Ethiopian smallholder farmers to protect their grain in storage (Abraham et al., 2008, Girma et al., 2008a,b, Hengsdijk and De Boer, 2017, Mengistie et al., 2017, Dessalegn et al., 2017, Loha et al., 2018). Dessalegn et al. (2017) reported that farmers use pesticides like phosphine, a toxic fumigant, without personal protective equipment. Pesticide retailers in Ethiopia sell many expired pesticides to smallholder farmers with low financial status, irrespective of their effectiveness (Mengistie et al., 2016). They also indicated that unknown chemicals and unlabeled pesticides were sold illegally by unauthorized and untrained persons at local village markets.

There is a need to explore safe and non-chemical alternatives in controlling insects in stored commodities in Ethiopia. New non-chemical technologies such as Purdue Improved Crop Storage (PICS) bags, GrainPro Super bags, plastic drums, and metal silos were introduced to sub-Saharan Africa to protect smallholder farmers' stored commodities (Obeng-Ofori, 2011, Murdock and Baoua, 2014, Chigoverah and Mvumi, 2016). In addition to these technologies, in Ethiopia two other inert dusts, filter cake and Triplex, were found to be potential alternatives to chemical pesticides (Girma et al., 2008a,b; Tadesse and Subramanyam, 2018 a,b). Filter cake is a by-product of aluminum sulfate factory (Awash Melkassa Aluminium Sulphate & Sulphuric

Acid Share Company, Melkassa Awash, Ethiopia), and Triplex is a by-product of Mohammed International Development Research and Organization Companies soap factory (Star Soap and Detergent Industries Private Limited Company, Addis Ababa, Ethiopia) (Girma et al., 2008a,b, Tadesse and Subramanyam, 2018a,b). An energy-dispersive X-ray spectroscopy elemental study of the two powders indicated that silicon and oxygen were the dominant elements in the form of silicon dioxide (Tadesse and Subramanyam, 2018a). The same study reported 100% mortality when adults of the maize weevil, *Sitophilus zeamais* Motschulsky, were exposed for 24 h to 7.5 and 10 g/m^2 of filter cake and Triplex treated concrete arenas, respectively. Girma et al. (2008a) reported 92% mortality in 3 d after *S. zeamais* adults were exposed to maize treated with 1, 2.5, and 5% (w/w) of filter cake. There was 100% mortality of the rice weevil, *Sitophilus oryzae* (Linnaeus) and *S. zeamais* adults after a 14-d exposure to wheat treated with 700 and 1000 mg/kg of filter cake (Tadesse and Subramanyam, 2018b). The same study reported 100% mortality when adults of *S. oryzae* were exposed for 14 d to 1000 mg/kg of Triplex treated wheat. Unlike the previous study by Tadesse and Subramanyam (2018a), which used four concentrations of filter cake and Triplex (2.5, 3.75, 5 and 10 g/m^2), the present study uses a range of filter cake and Triplex concentrations and various exposure times. This study was designed to determine concentrations and times for the 99% mortality of *S. zeamais* adults, and effective concentrations and times for 99% reduction of adult progeny production. In addition, the study also determined percentage of insect damaged kernels and percentage of grain weight losses to show which of the two products was efficacious against *S. zeamais*.

4.3. Materials and Methods

4.3.1. Concrete-poured Petri Dishes

Rockite®, a ready-to-mix concrete product (Hartline Products Co., Inc., Cleveland, OH), was mixed with tap water to make a slurry. The slurry was poured into 9 cm diameter and 1.5 cm high plastic Petri dishes (Fisher Scientific, Denver, CO). Slurry was poured to cover one half of the Petri dish's height. Slurry filled Petri dishes were allowed to dry on a laboratory bench for 24 h. Polytetrafluoroethylene (Insect-a-Slip, Bio Quip Products, Inc., Rancho Dominguez, CA) was used to coat the inside walls of Petri dishes to prevent insects from crawling on the sides of dishes.

4.3.2. Application of Powders to Concrete Arenas and Insect Exposure

A brass frame, stainless steel wire cloth sieve, with US Standard 80 mesh size (Seedburo Equipment Company, Des Plaines, IL), with 177 µm openings, was used to sift filter cake and Triplex powders prior to use in tests. In the first experiment, concrete arenas were treated with filter cake and Triplex powders at the following concentrations: 0 (untreated control), 0.5, 1, 2, 3, 4, 5, 6, 7 and 8 g/m^2 of filter cake or 0, 1, 2, 3, 4, 5, 6, 7, 8, and 9 g/m^2 of Triplex. A laboratory strain of *S. zeamais* was reared in an environmental growth chamber (Percival Scientific, Inc., Model I-36VL, Perry, IA) at 28°C and 65% r.h. on organic hard red winter wheat (Heartland Mills, Marienthal, KS) of 12.5% moisture content (wet basis) in the Department of Grain Science and Industry, Kansas State University. Adults of *S. zeamais* were separated from wheat using a 2.38 mm diameter round-holed aluminum sieve (Seedburo Equipment Company). Ten unsexed, three-week old adults, were exposed for 12 h to untreated concrete arenas (control) and arenas receiving each of the 9 concentrations of filter cake and Triplex with three replications.

After insect introduction, the Petri dishes were held at 28°C and 65% r.h. In the second experiment, 10 adults extracted from cultures were separated as explained above and exposed to 3 g/m^2 of filter cake and 9 g/m^2 of Triplex in separate concrete arenas for 1, 2, 4, 6, 8, 10, 12, 14, 16, 18, 20, 22, and 24 h with three replications. All dishes were held at 28°C and 65% r.h. After exposure at intended concentration or time, adults of *S. zeamais* were transferred carefully with a Camel's hair brush to 150 ml round plastic containers holding 30 g of cleaned, organic hard red winter wheat of ~12.5% moisture content (wet basis). The plastic containers had perforated lids with wire-mesh screens to facilitate air diffusion. Containers were held at 28°C and 65% RH for 14 d. After 14 d wheat from each container was sifted using a 2.38 mm circular round-holed aluminum sieve to separate adults from wheat. Adults that did not respond when gently prodded by a Camel's hair brush were considered dead.

In the third experiment, progeny production was determined 42 d after *S. zeamais* adults were exposed to a new set of untreated and concrete arenas treated with same concentrations and exposure times as outlined in experiments 1 and 2. Adult progeny produced was counted from each container by subtracting the 10 originally added adults. Wheat from each container was passed three times through the Boerner® divider (Seedburo Equipment Company) to get a working subsample of ~3.8 g from each replicate for determining undamaged and insect damaged kernels. Number of insect damaged kernels out of the total examined was expressed as a percentage. Percentage of grain weight loss was determined based on counting and weighing damaged and undamaged kernels from each replicate working subsample (Adams and Schulten, 1978, Boxall, 1986). In each replicate control and treated working samples, percentage of grain weight loss was calculated using the following equation:

Weight loss (%) = $[(UN_d - DN_u) \div (U \times (N_d + N_u))] \times 100$

where, U is the weight of undamaged kernels, N_d is the number of damaged kernels, D is the weight of damaged kernels, and N_u is the number of undamaged kernels (Adams and Schulten, 1978, Boxall, 1986).

4.3.3. Data Analysis

The mean ± SE mortality of *S. zeamais* on untreated (control) arenas at all exposure times ranged from 0 to 0.33 ± 0.33%. Therefore, mortality data of *S. zeamais* exposed to filter cake and Triplex were corrected for responses in the control treatment (Abbott, 1925). Probit analysis was used to generate lethal concentrations (LCs) and times (LTs) for 99% mortality of *S. zeamais* when exposed to filter cake and Triplex. Similarly, probit analysis was used to generate effective concentrations (ECs) and effective times (ETs) for 99% reduction of adult progeny production when exposed to filter cake and Triplex. The percentage reduction in adult progeny production relative to that in the control treatment was fit to the probit model. The LC_{99}, LT_{99}, EC_{99}, and ET_{99} values of filter cake were compared to the corresponding values of Triplex using ratio tests (Robertson et al. 2007). Concentrations that produced either 0 or 100% mortality of reduction of progeny production were not used for probit analysis. Differences between any two LC_{99}, LT_{99}, EC_{99}, or ET_{99} values between the two powders were considered to be significantly different ($P < 0.05$) if the 95% confidence interval (CI) for the ratio did not include 1 (Robertson et al. 2007). The nonlinear model, $y = a + bx + cx^{1.5} + dx^{0.5}$, was fit to percentage of insect damaged kernels and percentage of grain weight loss as a function of concentration of filter cake and Triplex using the TableCurve 2D software (Jandel Scientific, San Rafael, California, USA). Similarly, a nonlinear model, $\ln y = a + bx$, was fit to percentage of insect damaged kernels and percentage of grain weight loss as a function of exposure time for both powders. The parameters *a*, *b*, *c*, and *d* were estimated by fitting equations to percentage of insect damaged kernels and percentage of grain weight loss data as a function of concentration and exposure time. Each possible pair-wise comparison between powders for each set of data was done by comparing individual models to a pooled model (Draper and Smith, 1998). Any two models being compared were considered to be significantly different ($P < 0.05$) from one another if the F-test showed individual models to deviate from the pooled model. Graphs were plotted using SigmaPlot, version 12.5 software (Systat Software, Inc., San Jose, California USA). All data were analyzed using SAS software (SAS Institute, 2012).

4.4. Results

4.4.1. Responses of S. zeamais Adults to the Powders

The mean ± SE percentage corrected mortality of *S. zeamais* adults exposed to 0.5, 1, 2, 3, 4, 5, 6, 7 and 8 g/m^2 concentrations of filter cake ranged from 41.6 ± 6.9 to 100%. At 1, 2, 3, 4, 5, 6, 7, 8 and 9 g/m^2 concentrations of Triplex, adult mortality ranged from 21 ± 9.1 to 96.6 ± 3.4%. The χ^2 value for goodness-of-fit were not significant ($P > 0.05$), indicating good fit of probit model to the data. The lethal concentrations for 99% mortality of *S. zeamais* adults exposed to filter cake and Triplex were 7.54 and 23.46 g/m^2, respectively (Table 4.1). Triplex had significantly higher LC_{99} value than filter cake (LC_{99} ratio (95% CI) = 3.11 (1.39–6.94)), indicating that filter cake was more efficacious than Triplex. The mean ± SE mortality of *S. zeamais* adults after exposure to 3 g/m^2 of filter cake and 9 g/m^2 of Triplex for 1, 2, 4, 6, 8, 10, 12, 14, 16, 18, 20, 22, and 24 h, ranged from 21 ± 6.9 to 100% and 24.4 ± 3.4 to 96.6 ± 3.4%, respectively. The corresponding lethal times for 99% mortality after exposure to 3 g/m^2 of filter cake and 9 g/m^2 of Triplex were 21.92 and 39.62 h, respectively (Table 4.2), confirming that filter cake was more efficacious than Triplex. Triplex took a significantly longer time to produce 99% adult mortality than filter cake (LT_{99} ratio (95% CI) = 1.81 (1.12–2.92)).

4.4.2. Adult Progeny Production at 42 d

The mean ± SE number of *S. zeamais* adult progeny produced at 42 days after exposure to 0.5, 1, and 2 g/m^2 concentrations of filter cake for 12 h ranged from 0 to 135.0 ± 27.8 with the highest number of progeny being produced at lower concentrations. *S. zeamais* adults tested at filter cake concentrations of 3–8 g/m^2 produced zero progeny because of 100% mortality of adults. The corresponding mean ± SE number of adult progeny produced by *S. zeamais* at 42 days after exposure to 1, 2, 3, 4, 5, 6, 7, 8 and 9 g/m^2 of Triplex for 12 h ranged from 1.0 ± 0.6 to 123.7 ± 34.8, with the highest number of adult progeny being produced at lower concentrations. The χ^2 value for goodness-of fit of probit model to percentage adult progeny reduction data collected 42 d after exposure to filter cake was not significant ($P > 0.05$), indicating good fit of model to data. However, the χ^2 value for goodness-of fit of the model to percentage adult progeny reduction data collected 42 d after exposure to Triplex was significant ($P < 0.05$), indicating poor fit of probit model to the data. Despite poor fit of model to data, the variances and covariances were adjusted by the heterogeneity factor (χ^2 values divided by the df), and a critical value from the *t* distribution is used to compute the 95% confidence limits (Subramanyam et al. 2014). The effective concentration to decrease 99% of adult progeny production for *S. zeamais* 42 d after exposure to filter cake and Triplex was2.48 and 18.58 g/m^2, respectively (Table 4.3). Triplex had significantly higher EC_{99} value than filter cake (EC_{99} ratio (95% CI) = 7.49 (4.10–13.68)).

The mean ± SE number of adult progeny produced by *S. zeamais* adults at 42 d after exposure to 3 g/m^2 of filter cake or 9 g/m^2 of Triplex for 1, 2, 4 , 6, 8, 10, 12, 14, 16, 18, 20, 22, 24 h ranged from 0 to 111.3 ± 7.6 and 24.4 ± 3.4 to 96.6 ± 3.4, respectively. *S. zeamais* adults exposed to 3 g/m^2 of filter cake for 14–24 h produced no progeny. The χ^2 values for goodness-of fit of the model to adult progeny reduction data collected at 42 d after exposure to 3 g/m^2 of filter cake and 9 g/m^2 of Triplex were significant ($P < 0.05$), indicating poor fit of probit model to data. The effective exposure times required to reduce 99% of adult progeny production of *S. zeamais* 42 d after exposure to 3 g/m^2 of filter cake and 9 g/m^2 of Triplex were 17.49 and 22.31 h for filter cake and Triplex, respectively (Table 4.4). There was no significant difference between the ET_{99} values of filter cake and Triplex (ET_{99} ratio (95% CI) = 1.28 (0.81–2.01)).

4.4.3. Insect Damaged Kernels at 42 d

The percentage of insect damaged kernels was 0% at 42 d after *S. zeamais* adults were exposed to 3, 4, 5, 6, 7, and 8 g/m^2 of filter cake for 12 h. However, complete control of insect damaged kernels was not achieved in any of the Triplex treatments. Insect damaged kernels decreased with an increase in powder concentration. The decrease in insect damaged kernels as a function of concentration was nonlinear. The fitted models satisfactorily described the relationship between insect damaged kernels and concentration data of filter cake ($r^2 = 0.99$) and Triplex ($r^2 = 0.88$) (Fig. 4.1). Insect damaged kernels were significantly greater when adults of *S. zeamais* were exposed to Triplex compared to filter cake ($F = 14.70$; df = 4, 12; $P = 0.0001$; one–way ANOVA).

Insect damaged kernels were not observed when *S. zeamais* adults were exposed for 12 to 24 h to 3 g/m^2 of filter cake, and 18 to 24 h to 9 g/m^2 of the Triplex. Insect damaged kernels decreased with an increase in exposure time. The decrease in insect damaged kernels as a function of exposure time was nonlinear. The fitted models satisfactorily described the relationship between insect damaged kernels and exposure time data of filter cake ($r^2 = 0.97$) and Triplex ($r^2 = 0.92$) (Fig. 4.2). Insect damaged kernels were significantly greater when adults of *S. zeamais* were exposed to Triplex compared to filter cake ($F = 4.19$; df = 2, 24; $P = 0.0275$; one–way ANOVA).

4.4.4. Grain Weight Loss at 42 d

Percentage of grain weight loss was 0% at 42 d after *S. zeamais* adults were exposed for 12 h to 3, 4, 5, 6, 7, and 8 g/m^2 of filter cake. However, complete reduction of grain weight loss was not achieved in any of the Triplex treatments. Grain weight loss decreased with an increase in powder concentration. The decrease in grain weight loss as a function of powder concentration was nonlinear. The fitted models satisfactorily described the relationship between percentage of grain weight loss and concentration data of filter cake ($r^2 = 0.98$) and Triplex ($r^2 = 0.87$) (Fig. 4.3). Grain weight loss was significantly greater when adults of *S. zeamais* were exposed to Triplex compared to filter cake ($F = 14.13$; df = 4, 12; $P = 0.0001$; one–way ANOVA).

Grain weight loss was 0% at 42 d after *S. zeamais* adults were exposed for 12 to 24 h to 3 g/m^2 of filter cake and for 18 to 24 h to 9 g/m^2 of Triplex. Grain weight loss decreased with an increase in exposure time. The decrease in grain weight loss as a function of exposure time was nonlinear. The fitted models satisfactorily described the relationship between grain weight loss and exposure time data of filter cake ($r^2 = 0.96$) and Triplex ($r^2 = 0.91$) (Fig. 4.4). Grain weight loss was significantly greater when adults of *S. zeamais* were exposed to Triplex compared to filter cake ($F = 4.27$; df = 2, 24; $P = 0.0259$; one–way ANOVA).

4.5. Discussion

Silica-based insecticides have been used to protect stored commodities from insect pests over a long period of time (Headlee, 1924; Ebling, 1971; Subramanyam and Roesli, 2000; Arthur, 2002; Athanassiou et al., 2003, 2005; Collins and Cook, 2006a,b; Girma et al., 2008a,b; Campolo et al., 2014; Doumbia et al., 2014; Tadesse and Subramanyam, 2018a,b). These products have large proportion of silicon dioxide, which has greater capacity to adsorb epicuticular lipid from the insect's integument (Ebling, 1971; Subramanyam and Roseli, 2000; Malia et al., 2016a,b), causing death by desiccation. Silicon and oxygen are the major components of filter cake and Triplex (Tadesse and Subramanyam, 2018a) in the form of silicon dioxide, and the mode of action may be similar to silica-based inert dusts. In our experiment, dead insects we observed were dry and brittle.

Our data demonstrated contact toxicity of filter cake and Triplex through concentration and exposure time responses of *S. zeamais* adults. The mortality of *S. zeamais* increased with increasing concentration of filter cake and Triplex. The accumulation of an inert powder over an insect's body is directly proportional to the powder concentration (Le Patourel et al., 1989), and insect behavior such as mobility (Malia et al., 2016a). Filipović et al. (2010) tested oil adsorption capacity of silica powders prepared from sodium silicate solution with different concentrations of silicon dioxide. They reported a positive correlation between oil absorption and silicon dioxide concentration. This is supported in our study, where adult mortality tended to increase with increasing concentration of filter cake and Triplex. The same phenomenon was reported by Tadesse and Subramanyam (2018a,b) in tests with filter cake and Triplex against *S. zeamais* in tests with concrete arenas, and against *S. zeamais* and *S. oryzae* in tests with wheat.

Only the χ^2 values for goodness-of fit to determine EC_{99} for Triplex and ET_{99} for both powders were significant, which indicated a poor fit of probit models to data. These heterogeneous responses could be due to the fact that unsexed adults of 1-3 weeks old were used in the experiment. The level of adult activity could have resulted in differential pick-up (Le Patourel et al., 1989; Malia et al., 2016a) of filter cake or Triplex powders, and may have contributed to the heterogeneity observed. Heterogeneous responses of insects were reported by several researchers when exposed to different temperatures (Mahroof et al., 2003), spinosad (Subramanyam et al., 2014), chlorpyrifos-methyl plus deltamethrin (Seghal et al., 2013), and chlorine dioxide (E et al., 2017).

The LC_{99} and EC_{99} values of filter cake were significantly less than that of Triplex. The LT_{99} and ET_{99} values of Triplex were higher than filter cake. This was confirmed by the ratio test results which showed significantly higher toxicity of filter cake to *S. zeamais* than Triplex. A 7.5 g/m^2 concentration of filter cake was effective in killing 99% of *S. zeamais* adults in 12 h and about 22 h were required to achieve the same mortality when adults were exposed to 3 g/m^2 of filter cake. A concentration of 7.5 g/m^2 of filter cake was reported by Tadesse and Subramanyam (2018a) to kill 100% of *S. zeamais* adults after a 24–h exposure. However, more than three times the concentration of filter cake was required to achieve 99% mortality of *S. zeamais* adults after a 12 h exposure to Triplex. This could be related to the carbon content (atomic percent = 39.43 ± 12.63) of filter cake in the form of calcium carbonate, in addition to the silicon dioxide content (Tadesse and Subramanyam, 2018a). Adults of *S. zeamais* exposed on calcium carbonate treated maize at 1 and 2% (w/w) concentrations for 15 d produced 70.2 and 84.2% mortality, respectively (Silva et al., 2004).

Filter cake was effective in 99% suppression of progeny production at concentrations six times less than that of Triplex. There were zero insect damaged kernels and grain weight losses when adults were exposed for 12 h to 3 g/m^2 of filter cake. However, complete suppression of insect damaged kernels and grain weight loss were not achieved when adults of *S. zeamais* were exposed for 12 h to 9 g/m^2 of Triplex. Tadesse and Subramanyam (2018b) reported significantly higher mortality, and lower progeny production, insect damaged kernels, and grain weight loss when *S. zeamais* and *S. oryzae* were exposed to 100, 500, 700 and 1000 mg/kg concentrations of filter cake or Triplex for 7 and 14 d on wheat. These results indicated that filter cake was consistently more efficacious against *S. zeamais* and *S. oryzae* than Triplex. The present study at various concentrations and exposure times consistently showed that filter cake was more efficacious than Triplex against *S. zeamais* adults. Smallholder farmers in Ethiopia can use filter cake and Triplex rather than chemical pesticides in their storage structures to control *S. zeamais*. However, field study in Ethiopia needs to be conducted in smallholder farmers' traditional storages, which include above-ground structures like *gota* and *gotera*, and underground pits (Tadesse and Eticha, 2000; Blum and Bekele, 2001; Dessalegn et al., 2017). The next step is to get these powders registered in Ethiopia to be used by smallholder farmer's storages and warehouses owned or leased by non-governmental organizations (https://dlca.logcluster.org/plugins/servlet/mobile#content/view/2525925), and the United States Agency for International Development (USAID, 2016) as potential alternatives to chemical pesticides.

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Table 4.1. Probit regression estimates and concentrations required for 99% mortality for *S. zeamais* adults based on mortality assessments made 14 d after exposure to concrete arenas treated with various concentrations of filter cake and Triplex for 12 h.

Powder	*N*[a]	Mean ± SE		LC_{99} (95% CI) (g/m^2)	χ^2 (df)	*P*-value
		Intercept	Slope			
Filter cake	180	0.46 ± 0.13	2.13 ± 0.36	7.54 (4.65 – 18.60)	11.01 (16)*	0.8087
Triplex	270	-1.03 ± 0.20	2.45 ± 0.31	23.46 (15.90– 44.05)	18.88 (25)*	0.8027

[a] *N* = Total number of adults used to generate the probit regression estimates

*χ^2 values for goodness-of-fit were not significant ($P > 0.05$), indicating good fit of probit model to data.

Table 4.2. Probit regression estimates and times required for 99% mortality for *S. zeamais* adults based on mortality assessment made 14 d after exposure to concrete arenas treated with 3 g/m^2 of filter cake and 9 g/m^2 of Triplex for various time periods.

Powder	*N*[a]	Mean ± SE		LT_{99} (95% CI) (h)	χ^2 (df)	*P*-value
		Intercept	Slope			
Filter cake	240	-2.12 ± 0.32	2.72 ± 0.35	21.92 (16.75 – 33.56)	23.49 (22)*	0.3748
Triplex	390	-1.61 ± 0.24	2.00 ± 0.22	39.62 (29.87 – 60.30)	23.28 (37)*	0.9616

[a] *N* = Total number of adults used to generate the probit regression estimates.

[*] χ^2 value for goodness-of-fit were not significant ($P > 0.05$), indicating good fit of probit model to data.

Table 4.3. Probit regression estimates and concentrations required for 99% reduction of *S. zeamais* adult progeny production at 42 d after exposure to concrete arenas treated with various concentrations of filter cake and Triplex for 12 h.

Powder	*N*[a]	Mean ± SE		EC_{99} (95% CI) (g/m^2)	χ^2 (df)	*P*-value
		Intercept	Slope			
Filter cake	120	1.47 ± 0.06	2.18 ± 0.25	2.48 (1.96 − 3.54)	10.30 (10)[b]	0.4147
Triplex	270	-1.17 ± 0.28	2.76 ± 0.45	18.59 (11.91− 43.60)	470.42 (25)[c]	<0.0001

[a] *N* = Total number of adults used to generate the probit regression estimates.

[b] χ^2 value for goodness-of-fit was not significant ($P > 0.05$), indicating good fit of probit model to data.

[c] χ^2 value for goodness-of-fit was significant ($P < 0.05$), indicating poor fit of probit model to data.

Table 4.4. Probit regression estimates and times required for 99% reduction of *S. zeamais* adult progeny production 42 d after exposure to concrete arenas treated with 3 g/m^2 of filter cake and 9 g/m^2 of Triplex for various time periods.

Powder	*N*[a]	Mean ± SE		ET_{99} (95% CI)	χ^2 (df)[b]	*P*-value
		Intercept	Slope	(h)		
Filter cake	210	-0.82 ± 0.19	1.89 ± 0.25	17.49 (12.50 – 30.42)	132.51 (19)	<0.0001
Triplex	390	-1.08 ± 0.16	1.93 ± 0.16	22.31 (18.23 – 29.12)	200.52 (37)	<0.0001

[a] *N* = Total number of adults used to generate the probit regression estimates

[b] χ^2 values for goodness-of-fit of model to data were significant ($P < 0.0001$), indicating poor fit of probit model to data.

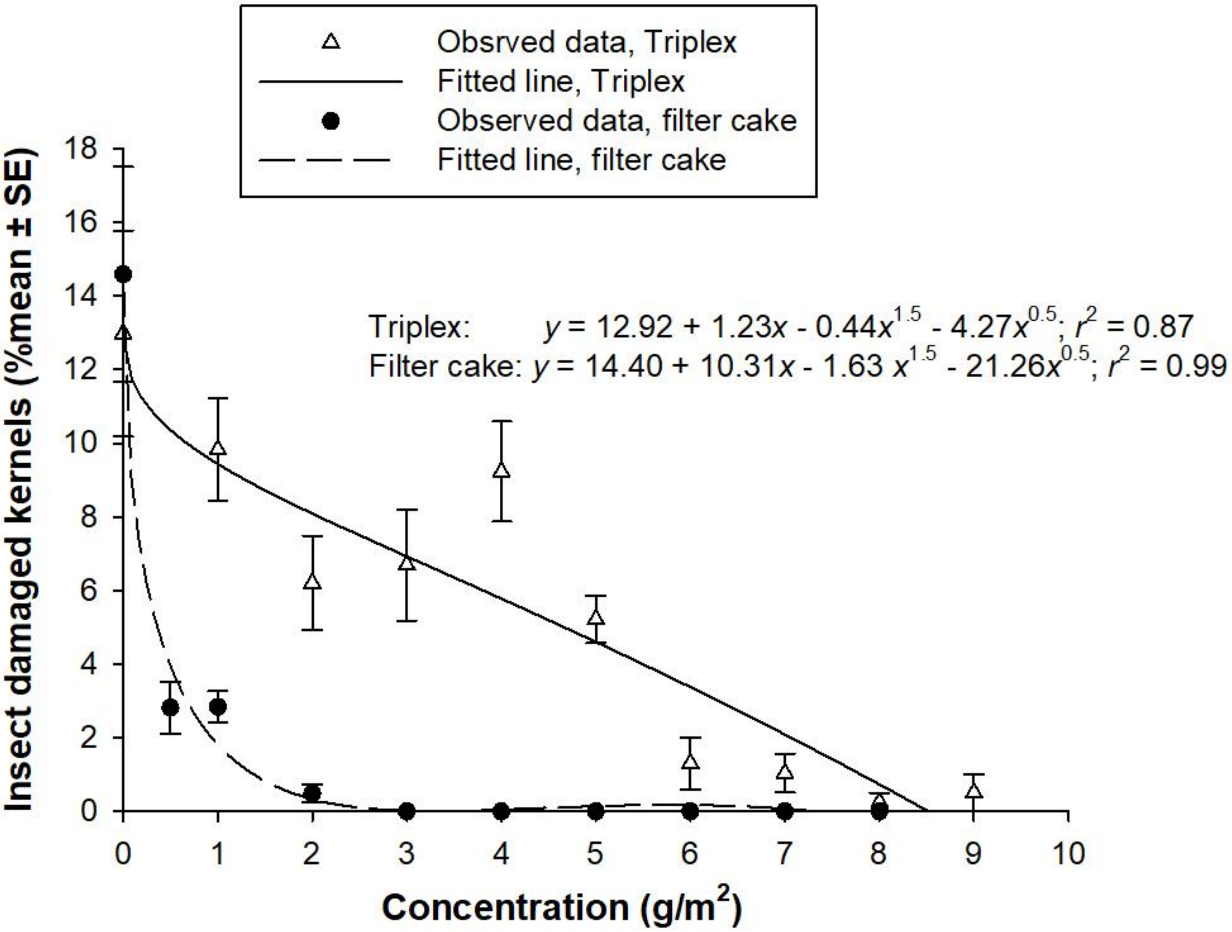

Figure 4.1. Percentage of kernels damaged by *S. zeamais* at 42 d after a 12 h exposure to various concentrations of filter cake and Triplex treated concrete arenas.

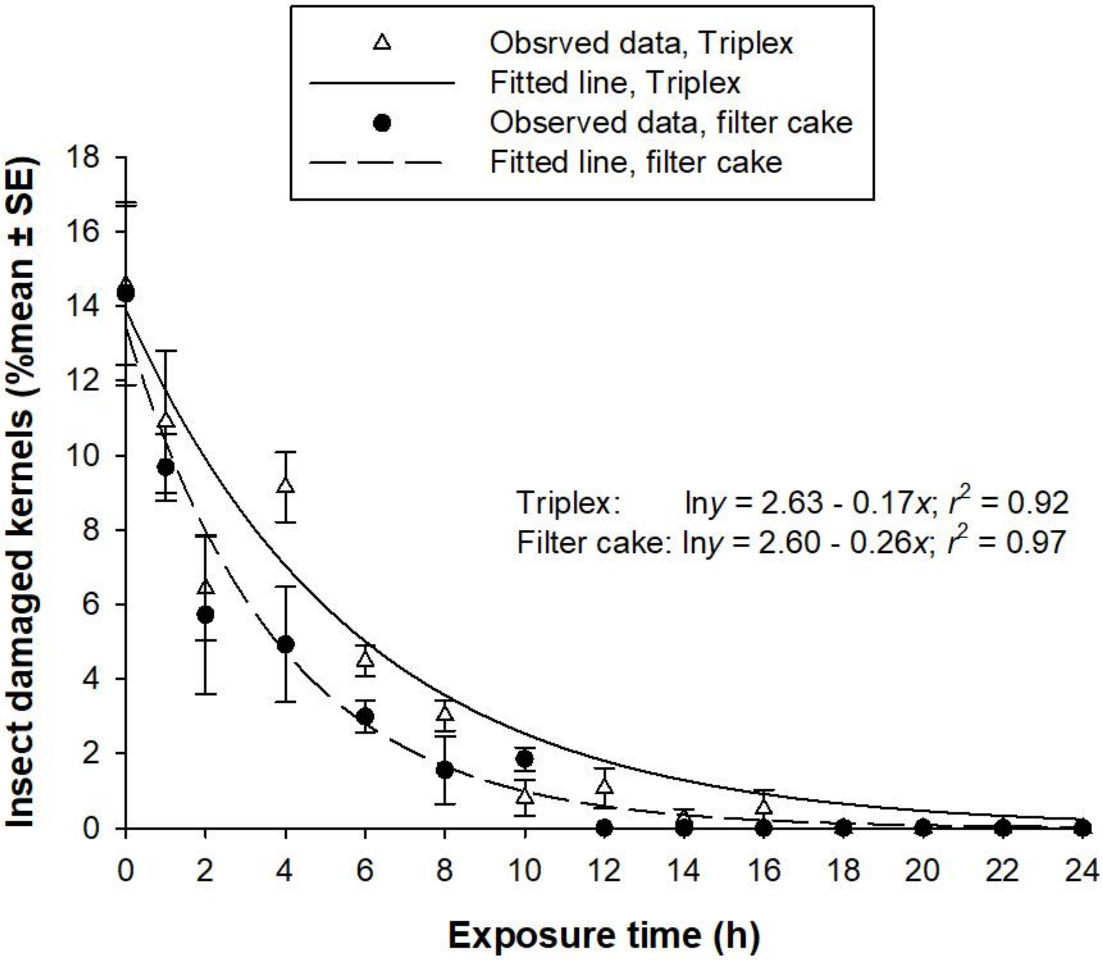

Figure 4.2. Percentage of kernels damaged by *S. zeamais* at 42 d after exposure to concrete arenas treated at 3 g/m^2 of filter cake and 9 g/m^2 of Triplex for various time periods.

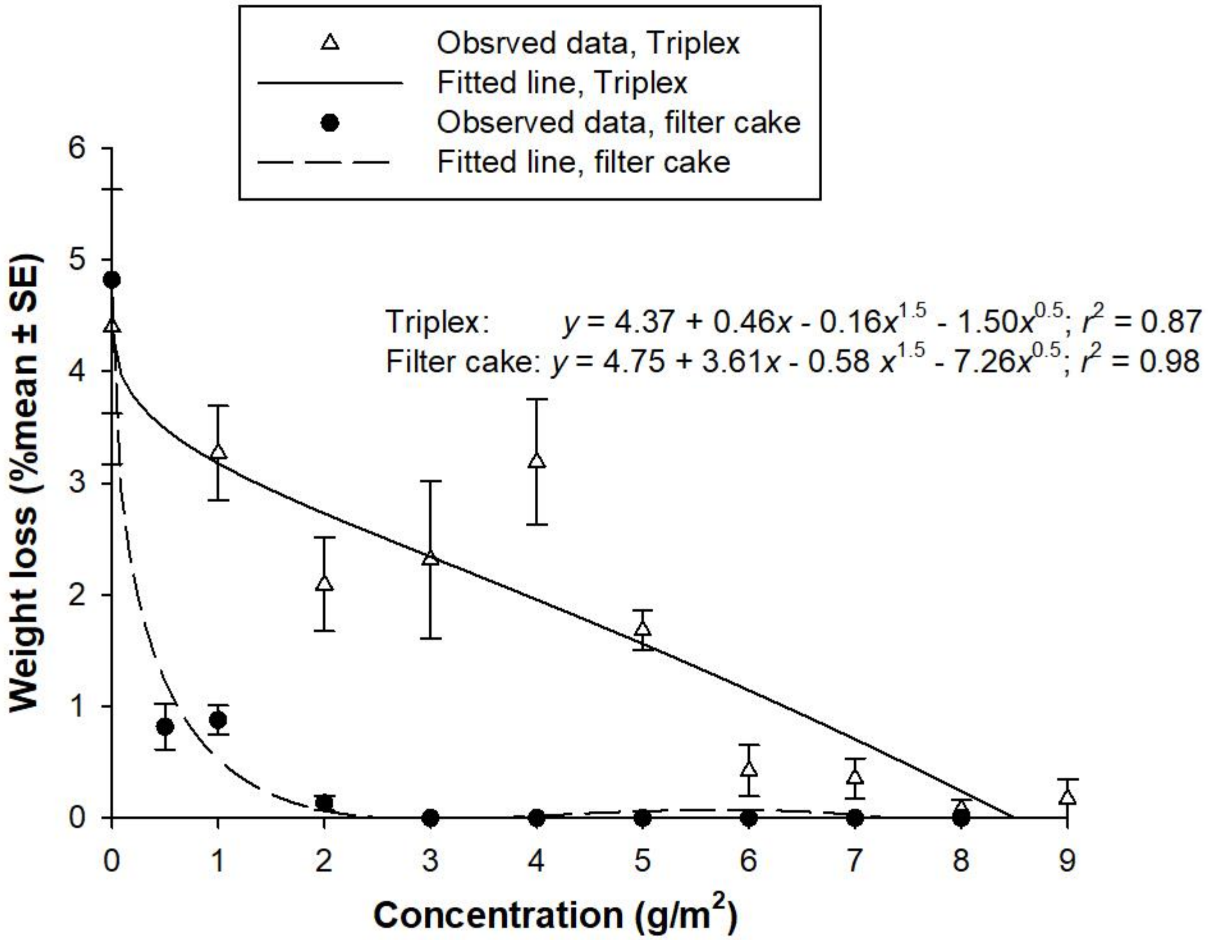

Figure 4.3. Percentage of grain weight loss at 42 d caused by *S. zeamais* after a 12 h exposure to various concentrations of filter cake and Triplex treated concrete arenas.

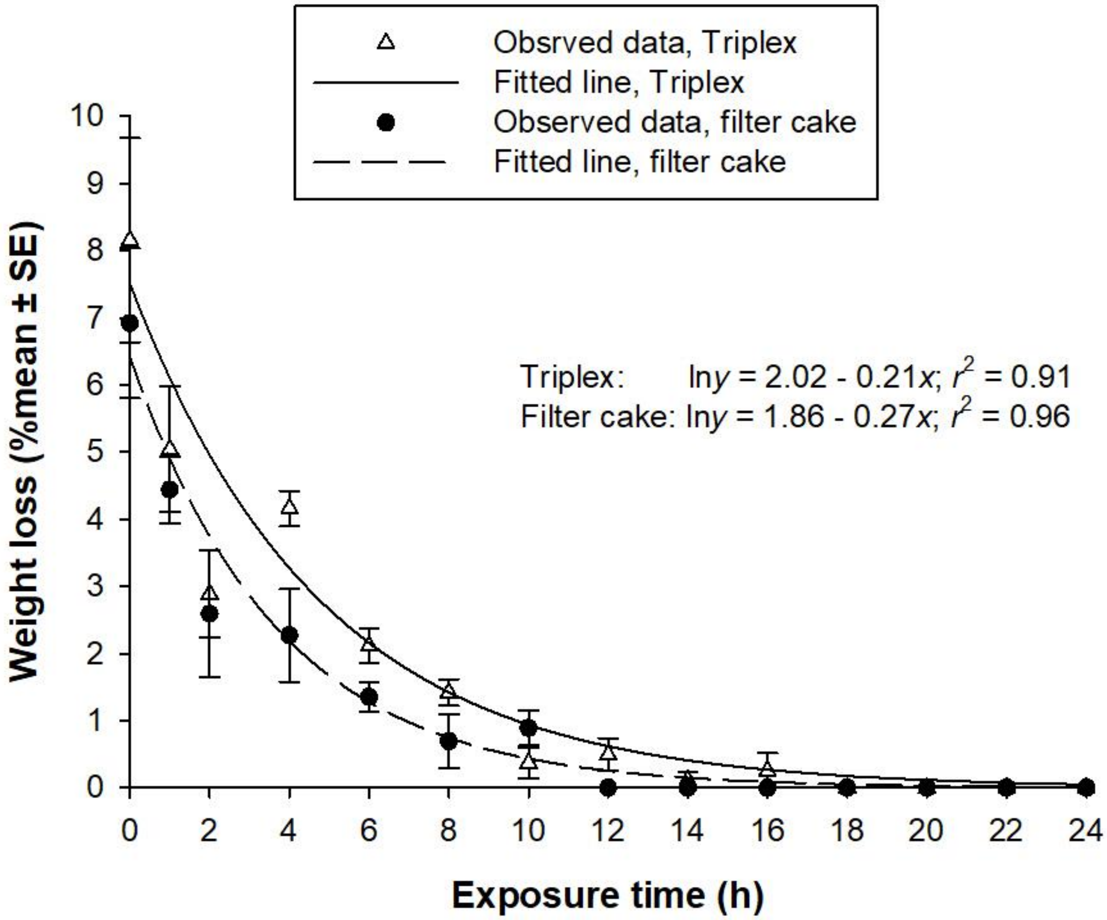

Figure 4.4. Percentage of grain weight loss at 42 d caused by *S. zeamais* after exposure to concrete arenas treated at 3 g/m^2 of filter cake and 9 g/m^2 of Triplex for various time periods.

Chapter 5 - Contact Toxicity of filter cake and Triplex powders from Ethiopia against *Sitophilus oryzae* (Coleoptera: Curculionidae)

5.1. Abstract

Contact toxicity of filter cake and Triplex powders from Ethiopia were evaluated against the rice weevil, *Sitophilus oryzae* (Linnaeus)—a common insect pest in Ethiopia. The lethal concentrations for 50 and 99% mortality (LC_{50} and LC_{99}) of filter cake and Triplex against *S. oryzae* were determined by exposing 10 adults for 12 h to concrete arenas inside Petri dishes treated with filter cake concentrations of 0.5-8 g/m^2 and Triplex concentrations of 1-9 g/m^2. Lethal times for 50 and 99% mortality (LT_{50} and LT_{99}) were determined by exposing adults for 1-24 h to 3 g/m^2 of filter cake and 9 g/m^2 of Triplex. Adults were transferred to containers with 30 g of wheat and held at 28°C and 65% r.h. for 14 d to determine mortality. Effective concentrations (EC_{50} and EC_{99}) and times (ET_{50} and ET_{99}) for 50 and 99% reduction of adult progeny production were determined from reduction in adult progeny production relative to production in control treatments at 42 d after exposure to filter cake and Triplex. LC_{50} and LC_{99} values for *S. oryzae* adults were 0.70 and 8.49 g/m^2, respectively, when exposed to filter cake and 2.27 and 21.38 g/m^2, respectively, when exposed to Triplex. The corresponding LT_{50} and LT_{99} values were 3.13 and 27.21 h, respectively, for filter cake and 4.72 and 38.60 h, respectively, for Triplex. EC_{50} and EC_{99} values for progeny reduction were 0.57 and 7.95 g/m^2, respectively, for filter cake and 2.77 and 18.82 g/m^2, respectively, for Triplex. The corresponding ET_{50} and ET_{99} values were 2.57 and 17.73 h, respectively for filter cake and 3.39 and 24.74 h, respectively, for Triplex. *S. oryzae* exposed to filter cake produced less number of insect damaged kernels and grain weight loss than those exposed to Triplex. Filter cake was more efficacious against *S. oryzae* than Triplex.

Keywords: Filter cake; Triplex; *Sitophilus oryzae*; Contact toxicity

5.2. Introduction

The postharvest losses are a major issue contributing to food insecurity in sub-Saharan Africa (Sheahan and Barrett, 2017). Insect pests and toxigenic molds cause quantitative and qualitative losses to grain in storage at the farmer level (Kadjo et al., 2016). Zorya et al. (2011) reported that each year, significant volumes of food grains were lost after harvest in sub-Saharan Africa and losses were estimated at $4 billion. The report indicated that this magnitude of food grain losses exceeded the value of total food aid received by sub-Saharan African countries between 2000 and 2010.

More than 80% of all grain produced is estimated be stored at the farm or village level in Ethiopia (Tadesse and Eticha, 2000). Farmers in Ethiopia have complained of the difficulty in keeping maize in stores without insect pest infestation due to ineffectiveness of insecticides approved for grain treatment (Williamson et al., 2008). The authors also indicated that despite the use of pesticides in stores, Ethiopian farmers reported that most storages in villages had become severely infested with weevils. A more drastic tactic for dealing with increased pest pressure is to abandon production or not use chemical pesticides to protect grain in storage (Mengistie et al., 2015, 2017; Hengsdijk and De Boer, 2017). Smallholder farmers cited pest-specific problems as the reason for abandoning production and storage of chickpea, noug, flax, field pea, and horse beans in some areas of Ethiopia (Williamson et al., 2008).

Most of the chemical pesticides used by Ethiopia smallholder farmers are organophophates, carbamates, and to some extent organochlorines (Mengistie et al., 2016). Pesticide usage by smallholder farmers in Ethiopia was frequently accompanied by poisoning of users and caused chronic health effects (Mekonnen and Agonafir, 2002). Use of unsafe storage facilities, improper training on safe use of pesticides, and inadequate infrastructure to regulate safe use of pesticides are major challenges in managing insects in the postharvest system in Ethiopia (Mengistie et al., 2017). In Ethiopia, unsafe handling of pesticides has resulted in ill health episodes, hospitalizations, and fatalities soon after a pesticide application (G/Mariam and Gelaw, 2016; Williamson et al., 2008).

Exploring products that are safe to humans and effective against stored-product insects in smallholder farmer's traditional storages in Ethiopia is necessary. Filter cake and Triplex are two such products found in Ethiopia as by-products of aluminum sulfate and soap factories, respectively (Girma et al., 2008a,b; Tadesse and Subramanyam, 2018a,b). Tadesse and Subramanyam (2018a) reported that silicon and oxygen were the dominant elements of filter cake and Triplex using energy-dispersive X-ray spectroscopy for analysis of elemental composition. Mortality of the maize weevil, *Sitophilus zeamais* Motschulsky, was 100% when adults were exposed for 24 h to 7.5 g/m^2 of filter cake on concrete arenas (Tadesse and Subramanyam, 2018a). Lethal concentrations for 99% mortality of *S. zeamais* adults were 7.54 and 23.46 g/m^2 when adults were exposed for 12 h to filter cake and Triplex treated concrete arenas, respectively (Tadesse et al., 2018). The corresponding lethal times for the 99% mortality (LT_{99}) were 21.92 and 39.62 h when *S. zeamais* adults were exposed to 3 g/m^2 of filter cake and 9 g/m^2 of Triplex, respectively. However, the toxicity of filter cake and Triplex against the rice weevil, *Sitophilus oryzae* (Linnaeus) on surfaces is unknown. Contact toxicity studies of filter cake and Triplex against *S. oryzae* will help in recommending these products to farmers as safer and effective alternatives to synthetic pesticides. This study was designed to determine the lethal concentrations and times for 50 and 99% mortality and effective concentrations and times for the 50 and 99% reduction of adult progeny production of filter cake and Triplex against *S. oryzae*, an economically important stored grain pest in Ethiopia (Abraham et al., 2008; Girma et al., 2008a,b; Tilahun and Hussen, 2014). In addition, the study examined percentage of insect damaged kernels and grain weight loss caused by *S. oryzae* at 42 d after exposure to filter cake and Triplex.

5.3. Materials and Methods

5.3.1. Concrete-poured Petri dishes

A concrete powder product, Rockite® (Hartline Products Co., Inc., Cleveland, Ohio, USA) was mixed with tap water to make a slurry. A 9 cm diameter and 1.5 cm high plastic Petri dishes (Fisher Scientific, Denver, Colorado, USA) were used to make concrete arenas using the slurry. Petri dishes were filled with slurry to a height of 0.75 cm, and were allowed to dry on a laboratory bench for 24 h. Polytetrafluoroethylene (Insect-a-Slip, Bio Quip Products, Inc., Rancho Dominguez, California, USA) was used to coat the inside walls of Petri dishes to prevent insects from crawling on sides of dishes.

5.3.2. Application of powders to concrete arenas and insect exposure

A brass US standard sieve #80 (mesh size of 177 µm) (Seedburo Equipment Company, Des Plaines, Illinois, USA) was used to sift filter cake and Triplex powders. Powders that passed through the sieve were used to treat concrete arenas. Concrete arenas inside Petri dishes were treated with the following concentrations: 0 (untreated control), 0.5, 1, 2, 3, 4, 5, 6, 7 and 8 g/m^2 of filter cake or 0, 1, 2, 3, 4, 5, 6, 7, 8, and 9 g/m^2 of Triplex. A laboratory strain of *S. oryzae* was reared in an environmental growth chamber (Percival Scientific, Inc., Model I-36VL, Perry, Iowa, USA) at 28 °C and 65% r.h. on organic hard red winter wheat (Heartland Mills, Marienthal, Kansas, USA) of 12.5% moisture content (wet basis) in the Department of Grain

Science and Industry, Kansas State University. The strain has been in rearing for 17 years. Adults of *S. oryzae* were separated from wheat using a 2.38 mm diameter round-holed aluminum sieve (Seedburo Equipment Company).

In the first set of experiments, 10 unsexed adults of 1-3 weeks old were exposed for 12 h to untreated concrete arenas (control) and arenas receiving each of the 9 concentrations of filter cake and Triplex with three replications to determine lethal concentrations producing 50 and 99% mortality (LC_{50} and LC_{99}). In the second set of experiments, 10 adults from the same culture were exposed to 3 g/m^2 of filter cake and 9 g/m^2 of Triplex separately for 1, 2, 4, 6, 8, 10, 12, 14, 16, 18, 20, 22 and 24 h with three replications to determine the lethal times producing 50 and 99% mortality (LT_{50} and LT_{99}). After each intended exposure time, adults were transferred carefully with the Camel's hair brush into 150 ml round plastic containers holding 30 g of cleaned, wheat of ~12.5% moisture content (wet basis). The plastic containers had perforated lids with wire-mesh screens to facilitate air diffusion. Containers were incubated at 28 °C and 65% r.h. for 14 d to determine mortality. A round-holed aluminum sieve with 2.38 mm was used to separate *S. oryzae* adults from wheat in each container. Adults that did not respond when gently prodded with a Camel's hair brush were considered dead.

In the third set of experiments, adults of *S. oryzae* were exposed at the same concentrations and exposure times as described above to determine adult progeny production, percentage of insect damaged kernels, and percentage of grain weight loss at 42 d after exposure to filter cake and Triplex. Adult progeny produced was counted from each container and the initially added 10 adults were subtracted. Percentage reduction in adult progeny production was calculated in filter cake and Triplex treatments relative to progeny production in the control treatments, to determine effective concentrations and times for 50 and 99% reduction in adult progeny production (EC_{50}, EC_{99}, ET_{50}, and ET_{99}).

Undamaged and insect damaged kernels were determined after wheat from each container was passed three times through the Boerner® divider (Seedburo Equipment Company) to get a working subsample of ~3.8 g. Number of insect damaged kernels out of the total examined was expressed as a percentage. Percentage of grain weight loss was determined based on counting and weighing damaged and undamaged kernels from each replicate working subsample (Adams and Schulten, 1978; Boxall, 1986). In each replicate control and treated working subsample, percentage of weight loss was calculated using the following equation:

Weight loss (%) = $[(UN_d - DN_u) \div (U \times (N_d + N_u))] \times 100$

where, U is the weight of undamaged kernels, N_d is the number of damaged kernels, D is the weight of damaged kernels, and N_u is the number of undamaged kernels (Adams and Schulten 1978; Boxall, 1986).

5.3.3. Data analysis

There was no mortality of *S. oryzae* adults in untreated (control) arenas at all concentrations and exposure times. Probit regression estimates and lethal concentrations and times producing 50 and 99% mortality and 50 and 99% reduction in adult progeny production were generated using probit analysis (SAS institute, 2012). Concentrations that produced either 0 or 100% mortality or reduction of adult progeny production were not used for probit analysis. The LC_{50}, LC_{99}, LT_{50}, LT_{99}, EC_{50}, EC_{99}, ET_{50}, and ET_{99} values of filter cake were compared with the corresponding values of Triplex using ratio tests (Robertson et al. 2007). Differences between any two lethal or effective values were considered to be significantly different ($P < 0.05$) if the 95% CI for the ratio did not include 1 (Robertson et al., 2007). A nonlinear model, $\ln y = a + bx$, was fit to percentage of insect damaged kernels and percentage of grain weight loss data as a function of concentration using TableCurve 2D software (Jandel Scientific, San Rafael, California, USA). The same nonlinear model, $\ln y = a + bx$, was fit to percentage of insect damaged kernels and percentage of grain weight loss data as a function of exposure time. The parameters a and b were estimated by fitting equations to percentage of insect damaged kernels and percentage of grain weight loss data as a function of concentration or exposure time. Each possible pair-wise comparison between the two powders for each set of data was done by comparing individual models to a pooled model (Draper and Smith, 1998). Any two models being compared were considered to be significantly different ($P < 0.05$) from one another if the F-test showed individual models to deviate from the pooled model. Graphs were plotted using SigmaPlot, version 12.5 software (Systat Software, Inc., San Jose, California USA).

5.4. Results

5.4.1. Mortality responses of S. oryzae to powders

The mean ± SE percentage mortality of *S. oryzae* adults ranged from 40.00 ± 5.77 to 100% when they were exposed for 12 h to 0.5, 1, 2, 3, 4, 5, 6, 7 and 8 g/m^2 of filter cake. The corresponding mortality ranged from 26.67 ± 8.82 to 96.67 ± 3.33% when adults were exposed for 12 h to 1, 2, 3, 4, 5, 6, 7, 8 and 9 g/m^2 of Triplex. The χ^2 values for goodness-of-fit were not significant ($P > 0.05$) indicating good fit of probit model to data. The LC_{50} and LC_{99} values were 0.70 and 8.49 g/m^2, respectively, when *S. oryzae* adults were exposed for 12 h to filter cake. The corresponding LC_{50} and LC_{99} values were 2.27 and 21.38, respectively, when adults were exposed for 12 h to Triplex (Table 5.1). Triplex had significantly greater LC_{50} and LC_{99} values than filter cake (LC_{50} ratio (95% CI) = 3.24 (2.18 – 4.82); LC_{99} ratio (95% CI) = 2.52 (1.13 – 5.60)) indicating filter cake was more toxic to *S. oryzae* adults compared to Triplex. The mean ± SE percentage mortality of *S. oryzae* adults ranged from 33.33 ± 6.67 to 100% and 20.0 ± 5.77 to 96.67 ± 3.33% after exposure to 3 g/m^2 of filter cake and 9 g/m^2 of Triplex, respectively, for 1, 2, 4, 6, 8, 10, 12, 14, 16, 18, 20, 22 and 24 h. The χ^2 values for goodness-of-fit were not significant ($P > 0.05$) indicating good fit of the probit model to data. The LT_{50} and LT_{99} values were 3.13 and 27.21 h, respectively, after exposure to 3 g/m^2 of filter cake, and the values were 4.72 and 38.60 h, respectively, after exposure to 9 g/m^2 of Triplex (Table 5.2). Triplex had significantly greater LT_{50} value than filter cake (LC_{50} ratio (95% CI) = 1.51 (1.03 – 2.20)). However, there was no significant difference in LT_{99} values of filter cake and Triplex (LT_{99} ratio (95% CI) = 1.42 (0.80 – 2.51)).

5.4.2. Adult progeny production at 42 d

The mean ± SE number of *S. oryzae* adult progeny produced after a 12 h exposure to 0, 0.5, 1, 2, 3, 4, 5, 6, 7 and 8 g/m^2 of filter cake ranged from 0 to 59.33 ± 6.36. It ranged from 1.00 ± 0.56 to 66.00 ± 3.05 when adults were exposed for 12 h to 0, 1, 2, 3, 4, 5, 6, 7, 8 and 9 g/m^2 of Triplex. Adult progeny production was 0% when adults were exposed for 12 h to 4-8 g/m^2 of filter cake concentrations. However, 100% reduction in progeny production was not achieved when adults were exposed for 12 h to any of Triplex concentrations. The χ^2 values for goodness-of-fit were significant ($P < 0.05$) indicating poor fit of probit model to data. Despite poor fit of model to data, the variances and covariances were adjusted by the heterogeneity factor (χ^2 values divided by the df), and a critical value from the t distribution is used to compute the 95% confidence limits (Subramanyam et al., 2014). The EC_{50} and EC_{99} values were 0.57 and 7.95

g/m^2, respectively, when *S. oryzae* adults were exposed for 12 h to filter cake. The corresponding EC_{50} and EC_{99} values were 2.77 and 18.82 g/m^2, respectively when adults were exposed for 12 h to Triplex (Table 5.3). Triplex had significantly higher EC_{99} value than filter cake (EC_{50} ratio (95% CI) = 4.85 (3.40 – 6.93); EC_{99} ratio (95% CI) = 2.37 (1.02 – 5.48)). The mean ± SE number of *S. oryzae* adult progeny produced at 42 d after exposure to 3 g/m^2 of filter cake, and 9 g/m^2 of Triplex for 1, 2, 4, 6, 8, 10, 12, 14, 16, 18, 20, 22 and 24 h ranged from 0 to 76.33 ± 6.74 and 0.33 ± 0.33 to 84.66 ± 11.46, respectively. There was no progeny production when adults were exposed for 14-24 h to 3 g/m^2 of filter cake. However, 100% reduction in progeny production was not achieved when adults were exposed for 1-24 h to 9 g/m^2 of Triplex. The χ^2 values for goodness-of-fit were significant ($P < 0.05$) indicating poor fit of probit model to data. The ET_{50} and ET_{99} values were 2.57 and 17.73 h, respectively, when *S. oryzae* adults were exposed to 3 g/m^2 of filter cake, and the values were 3.39 and 24.74 h, respectively, when adults were exposed to 9 g/m^2 of Triplex (Table 5.4). There were no significant differences in effective exposure times required for 50 and 99% reduction in adult progeny production between filter cake and Triplex treatments (ET_{50} ratio (95% CI) = 1.31 (0.97 – 1.78); ET_{99} ratio (95% CI) = 1.40 (0.97 – 2.02)).

5.4.3. Insect damaged kernels at 42 d

Insect damaged kernels were not observed when adults of *S. oryzae* were exposed for 12 h to 4-8 g/m^2 of filter cake and 9 g/m^2 of Triplex. The mean ± SE percentage of insect damaged kernels ranged from 0 to 10.57 ± 1.22 and 0 to 12.81 ± 3.15 for all the concentrations of filter cake and Triplex, respectively. There was a decrease in insect damaged kernels with an increase in powder concentration. The decrease in insect damaged kernels at increasing concentrations was nonlinear, and the fitted models satisfactorily described the relationship between insect damaged kernels and concentration data of filter cake ($r^2 = 0.99$) and Triplex ($r^2 = 0.85$) (Fig. 5.1). Insect damaged kernels were significantly lower ($P < 0.05$) when *S. oryzae* adults were exposed for 12 h to filter cake when compared with Triplex ($F = 62.98$; df = 2, 16; $P < 0.0001$; one–way ANOVA).

There were no insect damaged kernels when *S. oryzae* adults were exposed for 14-24 h to 3 g/m^2 of filter cake and for 24 h to 9 g/m^2 of Triplex. The mean ± SE percentage of insect damaged kernels ranged from 0 to 9.51 ± 1.13 and 0 to 13.31 ± 0.92 at all exposure times at 3 g/m^2 of filter cake and to 9 g/m^2 of Triplex treatments, respectively. There was a decrease in insect damaged kernels with an increase in exposure time. The decrease in insect damaged kernels with an increase in exposure time was nonlinear, and the fitted models satisfactorily described the relationship between insect damaged kernels and exposure time data of filter cake ($r^2 = 0.95$) and Triplex ($r^2 = 0.94$) (Fig. 5.2). Insect damaged kernels were significantly lower ($P < 0.05$) when *S. oryzae* adults were exposed to 3 g/m^2 of filter cake when compared to 9 g/m^2 of Triplex ($F = 29.82$; df = 2, 24; $P < 0.0001$; one–way ANOVA).

5.4.4. Grain weight loss at 42 d

There was no grain weight loss after exposure of *S. oryzae* adults for 12 h to 4-8 g/m^2 of filter cake and to 9 g/m^2 of Triplex. The mean ± SE percentage of grain weight loss ranged from 0 to 5.26 ± 0.78 and 0 to 7.29 ± 2.15 after a 12 h exposure to all the concentrations of filter cake and Triplex, respectively. There was a decrease in percentage of grain weight loss with an increase in powder concentrations. The decrease in grain weight loss with an increase in powder concentrations was nonlinear, and the fitted models satisfactorily described the relationship between percentage of grain weight loss and concentration data of filter cake ($r^2 = 0.98$) and Triplex ($r^2 = 0.75$) (Fig. 5.3). Grain weight losses were significantly lower ($P < 0.05$) after a 12 exposure of *S. oryzae* adults to filter cake when compared with Triplex ($F = 47.54$; df = 2, 16; $P < 0.0001$; one–way ANOVA).

There was no grain weight loss when *S. oryzae* adults were exposed for 14-24 h to 3 g/m^2 of filter cake, and for 18-24 h to 9 g/m^2 of Triplex. The mean ± SE percentage of grain weight loss ranged from 0 to 5.43 ± 0.84 and 0 to 6.78 ± 1.90 at all exposure times of filter cake and of Triplex, respectively. There was a decrease in grain weight loss with an increase in exposure time. The decrease in grain weight losses with an increase exposure time was nonlinear, and the fitted models satisfactorily described the relationship between percentage of grain weight loss and exposure time data of filter cake ($r^2 = 0.88$) and Triplex ($r^2 = 0.77$) (Fig. 5.4). Grain weight loss was significantly lower ($P < 0.05$) when *S. oryzae* adults were exposed to 3 g/m^2 of filter cake compared to 9 g/m^2 of Triplex ($F = 36.15$; df = 2, 24; $P < 0.0001$; one–way ANOVA).

5.5. Discussion

Unlike synthetic pesticides, inert dusts that are abrasive cause mortality of insects through their physical properties like adsorption of wax from the insect's epicuticle leading to loss of water from their body, and eventually causing death by desiccation (Ebeling 1971; Subramanyam and Roseli, 2000; Tadesse et al., 2008; Malia et al. 2016a,b). Protection of stored grain from insect pests using inert dusts such as wood ash, lime, and sand, has been practiced in Ethiopia (Tadesse et al., 2008). Silica-based powders are one of the inert dust groups which are used to protect stored grains from insect pests (Subramanyam and Roseli, 2000). Tadesse and Subramanyam (2018a) reported that filter cake and Triplex have higher atomic percentage of silicon and oxygen in in the form of silicon dioxide, and the mode of action may be similar to other silica-based dusts. In our experiment, we observed dry and brittle dead insects, and therefore we hypothesized that the mode of action of filter cake and Triplex may be similar to silica-based dusts.

The free movement of insects over inert dust treated surfaces could increase the accumulation of powders over the insect's body leading to death by adsorption of the epicuticular wax (Le Patourel et al. 1989; Malia et al. 2016a). Filipović et al. (2010) reported an increase in oil absorption capacity with increasing silicon dioxide concentration in their study of oil absorption of silica powders with various concentrations of silicon dioxide. Our data demonstrated similar phenomenon because adult mortality tended to increase with an increase in concentration of filter cake and Triplex. Increased mortality of *S. zeamais* adults with increasing concentration of filter cake and Triplex on concrete arenas was reported by Tadesse and Subramanyam (2018a) and Tadesse et al. (2018). Tadesse and Subramanyam (2018b) also reported an increase in adult mortality with an increase in filter cake and Triplex concentration after *S. zeamais* and *S. oryzae* adults were exposed for 7 and 14 d to filter cake and Triplex treated wheat.

Heterogeneous responses in adult progeny production of *S. oryzae* were observed in our study. This indicated poor fit of the probit model to percentage reduction in adult progeny production data. We used unsexed adults of 1-3 weeks old in our experiment. The heterogeneity could be related on adult sex and age. We had used very small amount of filter cake and Triplex to treat concrete arenas, and there could be uneven distribution of particles on concrete surfaces in Petri dishes. Some insects might have escaped from contacting with powder particles by moving to areas with little or no particles (Le Patourel et al., 1989; Malia et al., 2016a,b). This could have contributed to the unexplained heterogeneity. Physiological differences among individuals to dehydration by filter cake and Triplex particles also may have contributed to the observed heterogeneity (Malia et al., 2016b). Heterogeneity responses of stored-product insects were reported by several researchers when exposed to different pesticides (Seghal et al. 2013; Subramanyam et al. 2014; E et al. 2017; Tadesse et al., 2018).

The lethal concentrations for the 50 and 99% mortality were significantly less when *S. oryzae* adults were exposed for 12 h to filter cake compared to Triplex. This was confirmed by the ratio test of the corresponding LC_{50} and LC_{99} values which showed significantly higher toxicity of filter cake to *S. oryzae* adults than Triplex. A 0.70 and 8.49 g/m^2 of filter cake were effective to achieve 50 and 99% mortality of *S. oryzae* adults, respectively, within 12 h. Similarly, 3.13 and 27.21 h were required to kill 50 and 99% of *S. oryzae* adults, respectively, when exposed to 3 g/m^2 of filter cake. However, more than three times the concentration of filter cake was required to produce similar mortality of *S. oryzae* adults at the same exposure times when adults were exposed to Triplex. A similar phenomenon was observed when *S. zeamais* adults were exposed to filter cake and Triplex using the same experimental set up (Tadesse et al., 2018). Tadesse and Subramanyam (2018a) reported 100% mortality of *S. zeamais* adults after a 24 h exposure to 7.5 g/m^2 of filter cake. Unlike Triplex, filter cake contains higher carbon (atomic percent = 39.43 ± 12.63) in the form of calcium carbonate (Tadesse and Subramanyam, 2018a) which could have contributed to its higher efficacy. A 98% mortality of *S. oryzae* adults was reported at 14 d after exposure to wheat treated with 600 ppm of calcium carbonate containing inert dusts (Liška et al., 2017). Silva et al. (2004) reported 70.2 and 84.2% mortality of *S. zeamais* adults at 15 d after exposure to maize treated with 1 and 2% (w/w) calcium carbonate, respectively.

Adult progeny was not produced at 42 d after exposure for 14-24 h to 3 g/m^2 of filter cake. A 0.57 and 7.95 g/m^2 of filter cake was effective in suppressing 50 and 99% of adult progeny production after a 12-h exposure. However, more than two times the concentration of filter cake was required to achieve 50 and 99% reduction of progeny production after a 12 h exposure to Triplex. Tadesse et al. (2018) reported a similar phenomenon in tests with filter cake and Triplex against *S. zeamais*.

There were no insect damaged kernels and grain weight loss when *S. oryzae* adults were exposed for 12 h to 4-8 g/m^2 of filter cake and to 9 g/m^2 of Triplex. In tests with filter cake and Triplex against *S. zeamais* on concrete arenas using only four exposure times, Tadesse and Subramanyam (2018a) reported 100% suppression of insect damaged kernels and grain weight loss when adults were exposed for 24 h to 5 g/m^2 of filter cake and 7.5 g/m^2 of Triplex. Tadesse et al. (2018) reported 100% suppression of insect damaged kernels and grain weight loss when *S. zeamais* adults were exposed for 12 h to 3 g/m^2 of filter cake and for 18 h to 9 g/m^2 of Triplex. A significantly higher mortality, and lower adult progeny production, insect damaged kernels, and grain weight loss were reported by Tadesse and Subramanyam (2018b) when *S. zeamais* and *S. oryzae* were exposed for 14 d to wheat treated with 100, 500, 700, and 1000 mg/kg filter cake and Triplex. Our study consistently showed that filter cake was more efficacious compared to Triplex against *S. zeamais* and *S. oryzae* adults. Smallholder farmers in Ethiopia can use filter cake and Triplex rather than chemical pesticides in their storage structures to control *S. oryzae*. Field studies of filter cake and Triplex against *S. oryzae* in smallholder farmers' traditional storages structures in Ethiopia such as above-ground structures like *gota* and *gotera*, and underground pits (Tadesse and Eticha, 2000; Blum and Bekele, 2001; Dessalegn et al., 2017) are warranted.

5.6. References

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Table 5.1. Probit regression estimates and concentrations required for 50 and 99% mortality for *S. oryzae* adults based on mortality assessments made 14 d after exposure for 12 h to concrete arenas treated with various concentrations of filter cake and Triplex.

Powder	N^a	Mean ± SE		LC (95% CL) (g/m^2)		χ^2(df)	*P*-value
		Intercept	Slope	LC_{50}	LC_{99}		
Filter cake	180	0.33 ± 0.13	2.15 ± 0.34	0.70 (0.45 − 0.93)	8.49 (5.24 − 20.45)	8.57(16)*	0.9300
Triplex	270	-0.85 ± 0.19	2.38 ± 0.31	2.27 (1.77 − 2.72)	21.38 (14.49 − 40.28)	20.31 (25)*	0.7302

[a] *N* = Total number of adults used to generate the probit regression estimates

$^*\chi^2$ values for goodness-of-fit were not significant (*P* > 0.05), indicating good fit of probit model to data.

Table 5.2. Probit regression estimates and times required for 50 and 99% mortality for *S. oryzae* adults based on mortality assessment made 14 d after exposure to concrete arenas treated with 3 g/m^2 of filter cake and 9 g/m^2 of Triplex for various time periods.

Powder	N[a]	Mean ± SE		LT (95% CL) (h)		χ^2(df)	P-value
		Intercept	Slope	LT$_{50}$	LT$_{99}$		
Filter cake	240	-1.37 ± 0.25	2.02 ± 0.29	3.13 (2.16 – 3.99)	27.21 (18.83 – 50.94)	17.57 (22)*	0.7311
Triplex	390	-1.76 ± 0.25	2.07 ± 0.23	4.72 (3.54– 5.79)	38.60 (29.53 – 57.23)	20.54 (37)*	0.9870

[a] N = Total number of adults used to generate the probit regression estimates.

[*] χ^2 value for goodness-of-fit were not significant ($P > 0.05$), indicating good fit of probit model to data.

Table 5.3. Probit regression estimates and concentrations required for 50 and 99% reduction of *S. oryzae* adult progeny production at 42 d after exposure for 12 h to concrete arenas treated with various concentrations of filter cake and Triplex.

Powder	N^a	Mean ± SE		EC (95% CL) (g/m^2)		χ^2(df)^b	*P*-value
		Intercept	Slope	EC_{50}	EC_{99}		
Filter cake	120	0.49 ± 0.10	2.03 ± 0.36	0.57 (0.34 − 0.76)	7.95 (4.31 − 31.56)	60.48 (10)	<0.0001
Triplex	270	-1.24 ± 0.19	2.80 ± 0.30	2.77 (2.33 − 3.19)	18.82 (13.79 − 30.12)	199.97 (25)	<0.0001

[a] *N* = Total number of adults used to generate the probit regression estimates

[b] χ^2 values for goodness-of-fit of model to data were significant ($P < 0.0001$), indicating poor fit of probit model to data.

Table 5.4. Probit regression estimates and times required for 50 and 99% reduction of *S. zeamais* adult progeny production 42 d after exposure to concrete arenas treated with 3 g/m^2 of filter cake and 9 g/m^2 of Triplex for various time periods.

Powder	N[a]	Mean ± SE		ET (95% CL) (h)		χ^2(df)[b]	P-value
		Intercept	Slope	ET$_{50}$	ET$_{99}$		
Filter cake	210	-1.29 ± 0.18	2.26 ± 0.24	2.57 (1.98 – 3.11)	17.73 (13.72 – 25.87)	97.12 (19)	<0.0001
Triplex	390	-1.53 ± 0.20	2.19 ± 0.20	3.39 (2.61 – 4.12)	24.74 (20.23 – 32.48)	257.13 (37)	<0.0001

[a] N = Total number of adults used to generate the probit regression estimates

[b] χ^2 values for goodness-of-fit of model to data were significant ($P < 0.0001$), indicating poor fit of probit model to data.

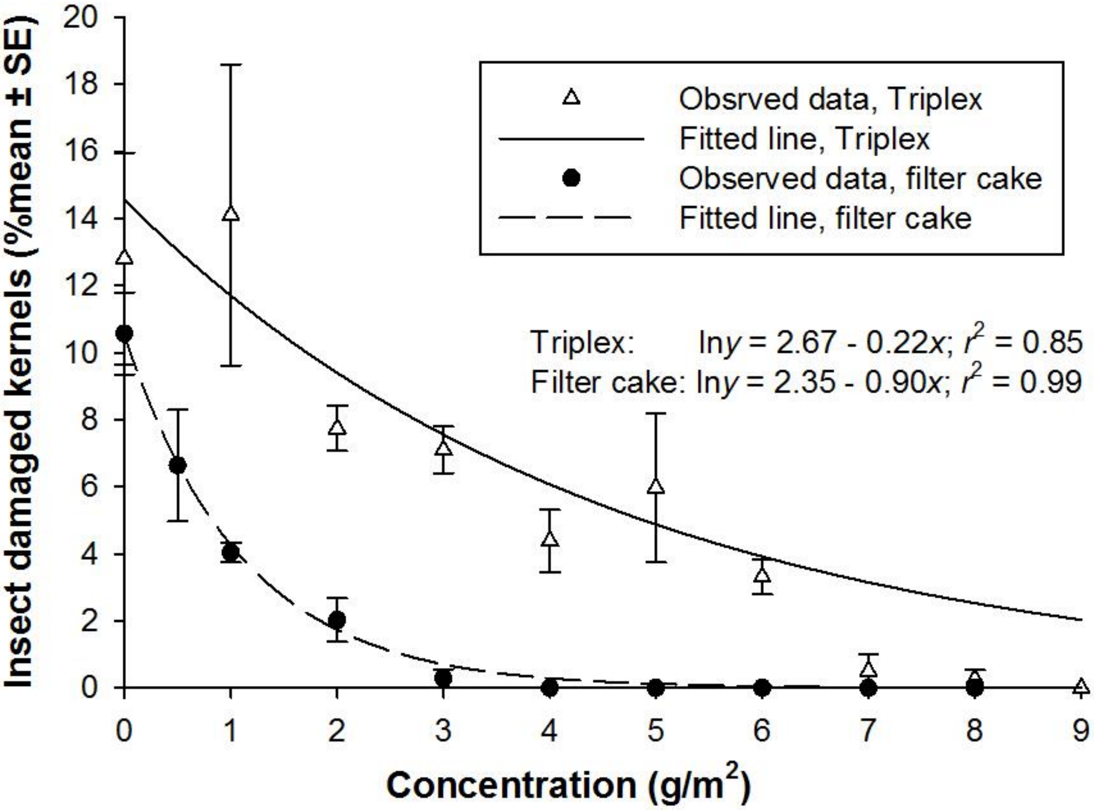

Figure 5.1. Percentage of kernels damaged by *S. oryzae* at 42 d after a 12 h exposure to various concentrations of filter cake and Triplex treated concrete arenas.

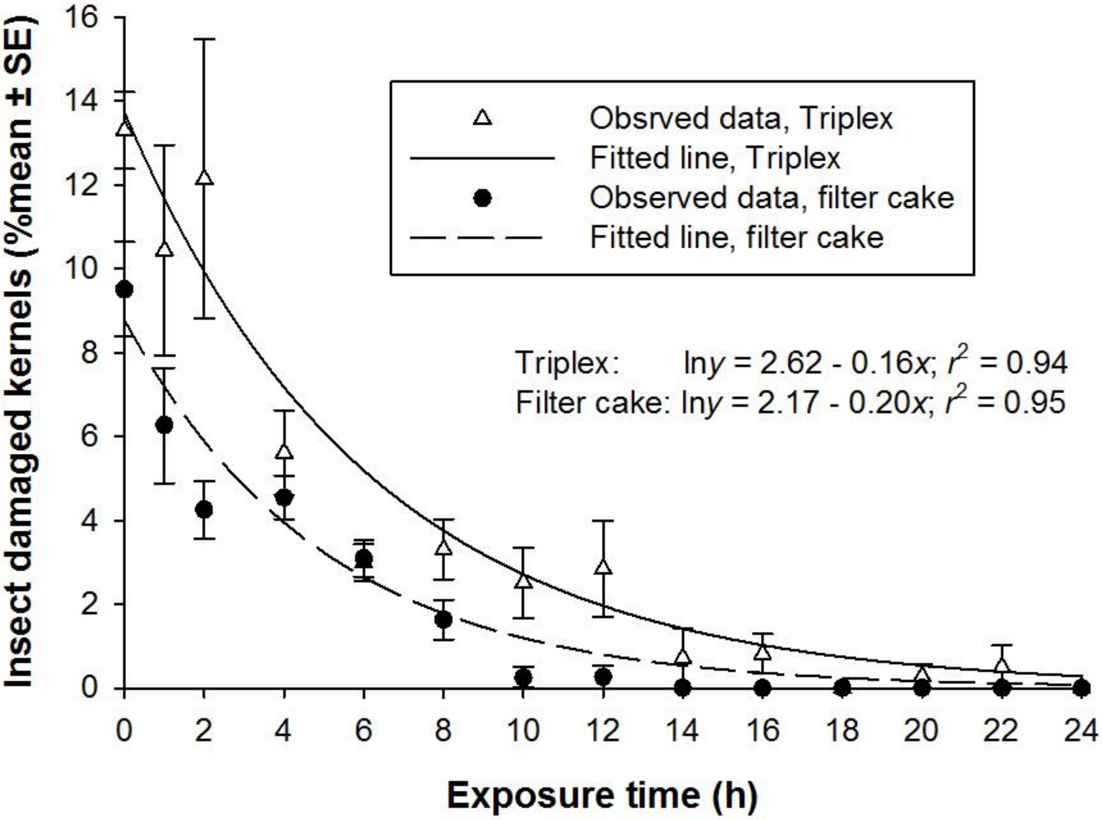

Figure 5.2. Percentage of kernels damaged by *S. oryzae* at 42 d after exposure to concrete arenas treated at 3 g/m^2 of filter cake and 9 g/m^2 of Triplex for various time periods.

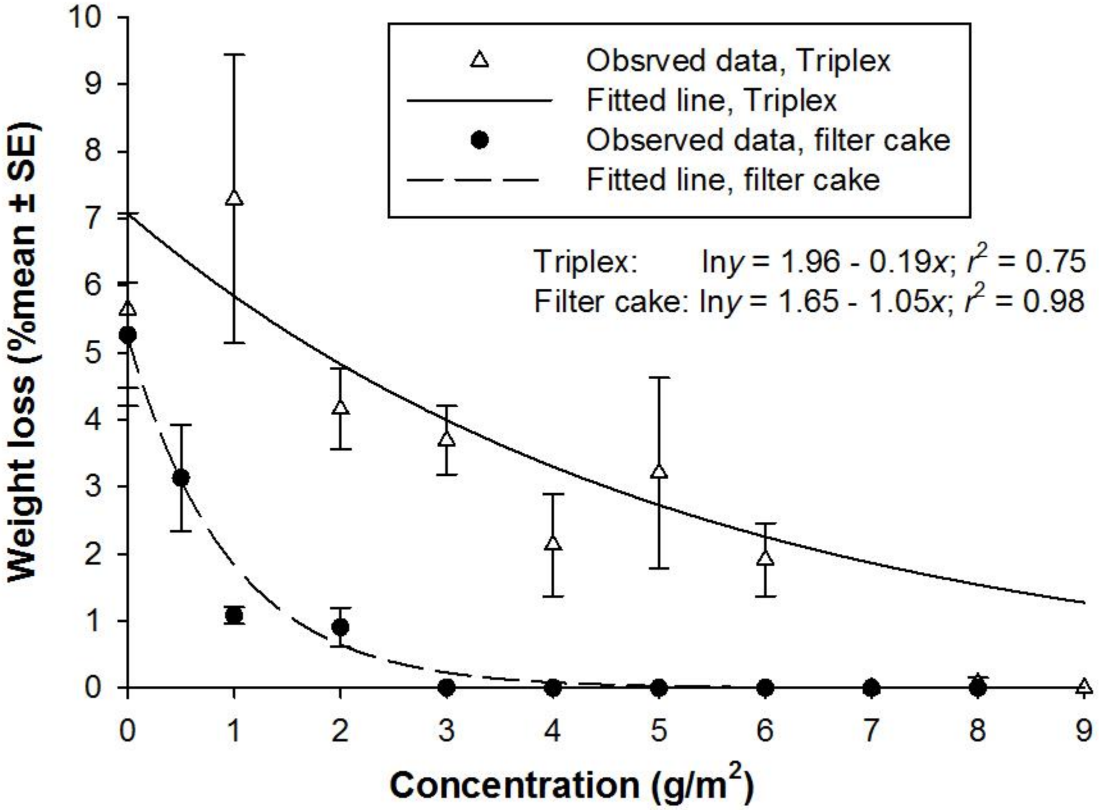

Figure 5.3. Percentage of grain weight loss at 42 d caused by *S. oryzae* after a 12 h exposure to various concentrations of filter cake and Triplex treated concrete arenas.

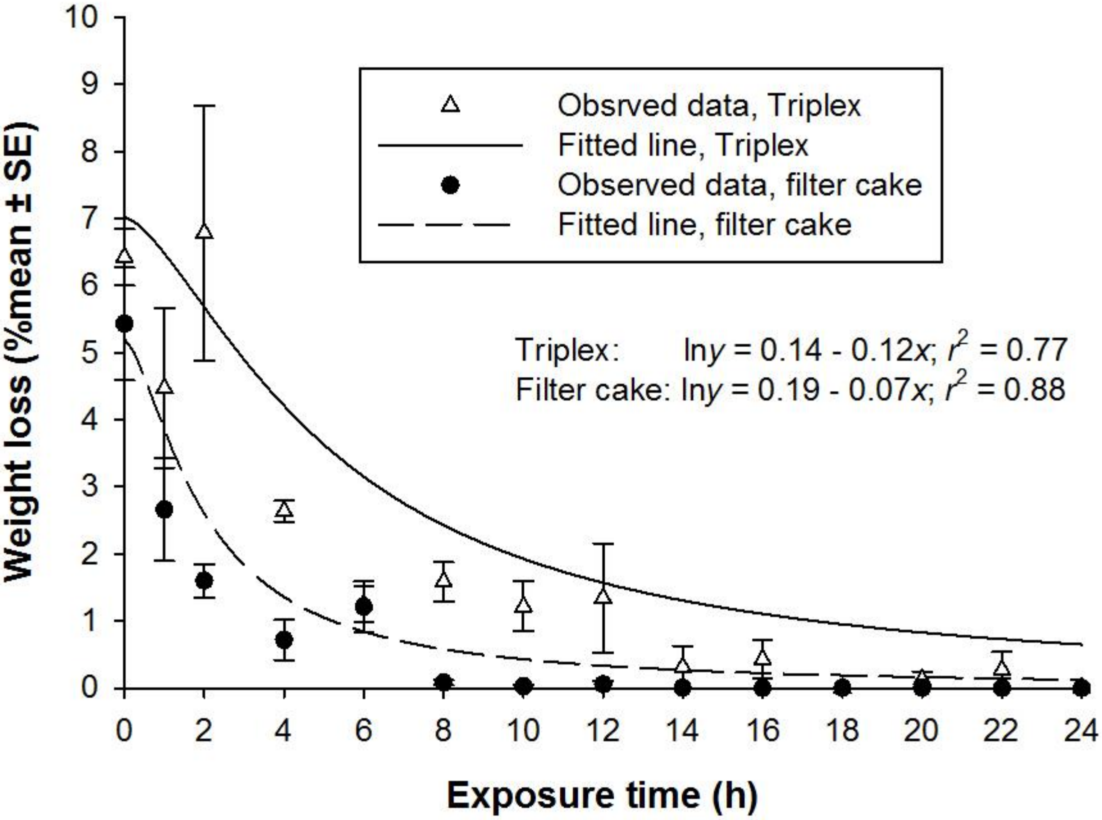

Figure 5.4. Percentage of grain weight loss at 42 d caused by *S. oryzae* after exposure to concrete arenas treated at 3 g/m2 of filter cake and 9 g/m2 of Triplex for various time periods.

Chapter 6 - Efficacy of filter cake and Triplex powders against three internally developing stored-product insect pests

6.1. Abstract

The efficacy of filter cake and Triplex powders applied to maize and wheat was evaluated in the laboratory against the lesser grain borer, *Rhyzopertha dominica* (Fabricius); maize weevil, *Sitophilus zeamais* Motschulsky; and rice weevil, *Sitophilus oryzae* (Linnaeus). These species are major insect pests associated with stored grain in Ethiopia. Efficacy of the two powders was determined by exposing 20 adults of each species to 100 g of maize and wheat treated with 0, 0.3, 0.5, 0.7, 1, 2, 3, 4 and 5 g/kg of filter cake, and 0, 0.5, 0.7, 1, 2, 3, 6, 8 and 10 g/kg of Triplex. Adult mortality was determined 14 d after exposure to untreated and treated grain. Adult progeny production at each species-powder-concentration combination was determined at 42 d. Complete mortality (100%) of *R. dominica* was achieved at 3–5 g/kg of filter cake treated maize; however, 100% mortality of *S. zeamais*, and *S. oryzae* was not achieved at any of filter cake concentrations on maize. Mortality was 100% for adults of *R. dominica* exposed to wheat treated with 2–3 g/kg of filter cake. The corresponding 100% mortality of *S. zeamais*, and *S. oryzae* was achieved when adults were exposed to wheat treated with 0.7–3 g/kg of filter cake. Mortality was less than 100% at all Triplex concentrations on maize and wheat for all three species. Adult progeny production of *R. dominica* was completely suppressed at filter cake concentrations of 1–5 g/kg on maize, whereas progeny production of *S. zeamais*, and *S. oryzae* was not completely suppressed at any of filter cake concentration on maize. No progeny of *R. dominica* was observed at 2–3 g/kg of filter cake treated wheat. Similarly, progeny production of *S. zeamais*, and *S. oryzae* was completely suppressed in 3 g/kg of filter cake treated wheat. Complete suppression of progeny production of the three species was not achieved at all concentrations of

Triplex on both maize and wheat. These powders have potential in managing *R. dominica*, *S. zeamais*, and *S. oryzae* following field tests in smallholder traditional storage structures in Ethiopia.

Keywords: Filter cake; Triplex; Maize; Wheat; *Rhyzopertha dominica*; *Sitophilus* species; Efficacy assessment

6.2. Introduction

Most of the sub-Saharan African countries store their grains in traditional structures which are not insect proof (Abraham et al., 2008; Nukenine, 2010). Post-harvest losses of dry durable commodities in sub-Saharan Africa are significant and are estimated to range from 20-40% (Zorya et al., 2011). Losses up to 50% in cereals and 100% in pulses have been reported, although average losses stand at about 20% (Nukenine, 2010). Grain storage losses in Ethiopia due to insect pests were estimated to be in the range of 10-21% (Abraham et al., 2008), consistent with losses in other sub-Saharan countries. Tefera et al. (2011) indicated the post-harvest loss to range from 20 to 30% in Ethiopia. Major insects that contribute to post-harvest loss of cereals and pulses in Ethiopia include grain weevils, grain borers, grain beetles, and grain moths (Abate et al., 2000; Abraham et al., 2008; Nukenine, 2010). The lesser grain borer, *Rhyzopertha dominica* (Fabricius); maize weevil, *Sitophilus zeamais* Motschulsky; and rice weevil, *Sitophilus oryzae* (Linnaeus) are cosmopolitan insect pests (Hills, 2008), and considered as major primary insect pests of stored grains in Ethiopia (Abraham et al., 2008; Nukenine, 2010; Befikadu, 2018).

Application of chemical pesticides has been recommended to protect stored grain from insect pests. However, farmers in Ethiopia complained of the difficulty of keeping their grains in stores infestation-free due to ineffectiveness of pesticides authorized for grain treatment (Williamson et al., 2008). The report also indicated that despite the use of pesticides in stores, Ethiopian farmers reported that most smallholder farmers' storages had become severely infested with weevils. In addition, Ethiopian smallholder farmers are confronted with a number of problems related to unsafe handling and use of pesticides due to improper training in safe use of pesticides, and inadequate infrastructure to regulate safe use of pesticides (Mengistie et al., 2017).

Therefore, safe and non-chemical alternatives should be provided to smallholder farmers in order to reduce hazards related to pesticides. New non-chemical technologies such as Purdue Improved Crop Storage (PICS) bags, GrainPro Super bags, plastic drums, and metal silos were introduced to sub-Saharan Africa to protect smallholder farmers' stored commodities (Obeng-Ofori, 2011; Murdock and Baoua, 2014; Chigoverah and Mvumi, 2016). In addition to these new technologies, some inert dusts, such as clay, sand, ground phosphate, ash, diatomaceous earths were in use for a long period of time in Africa (Headlee, 1924; Ebeling, 1971; Subramanyam and Roesli, 2000; Liška et al., 2017). Inert dusts are dry dusts that are chemically unreactive in nature (Liška et al., 2017; Bohinc et al., 2018), and are identified as alternatives to chemical pesticides (Subramanyam and Roesli, 2000). Protection of stored grain from insect pests using inert dusts such as wood ash, lime, sand, and tobacco has been practiced in Ethiopia (Abraham et al., 2008). Inert dusts are classified into different groups based on their physical and chemical composition (Subramanyam and Roesli, 2000; Stadler et al., 2010).

Silica based inert dusts are one of these groups which have toxic effect to insects due to their ability to adsorb lipids from insect's epicuticle leading to death by desiccation (Ebeling, 1971; Subramanyam and Roesli, 2000; Malia et al., 2016a). Limited studies investigated different alternatives to chemical pesticides in Ethiopia (Abraham et al., 2008; Girma et al., 2008a,b). Filter cake and Triplex were identified as two of the alternatives to chemical pesticides found in Ethiopia, and are locally available. Filter cake and Triplex are by-products of aluminum sulfate and soap factories, respectively (Girma et al., 2008a,b; Tadesse and Subramanyam, 2018a,b). Tadesse and Subramanyam (2018a) reported that filter cake and Triplex have higher atomic percentage of silicon and oxygen in the form of silicon dioxide, and they inferred that the mode of action may be similar to other silica-based inert dusts. They reported 100% mortality of *S. zeamais* adults after exposure of adults for 24 h to 7.5 g/m^2 of filter cake on concrete arenas. Complete mortality of *S. zeamais* and *S. oryzae* adults was reported by Tadesse et al. (2018a,b) after a 12 h exposure to 3–8 g/m^2 and 4–8 g/m^2 filter cake, respectively on concrete arenas. Tadesse and Subramanyam (2018b) reported significantly greater mortality, and lower progeny production after exposure of *S. zeamais* and *S. oryzae* adults for 7 and 14 d to 0.1, 0.5, 0.7 and 1g/kg of filter cake or Triplex treated wheat. However, there are limited data on efficacy of filter cake and Triplex against multiple species of internally developing stored-product insect pestsat a range of concentrations applied to maize and wheat. Unlike the previous study by Tadesse and Subramanyam (2018b), which used four concentrations of filter cake and Triplex (0.1, 0.5, 0.7 and 1g/kg), the present study uses a range of filter cake and Triplex concentrations with particle sizes that are less than 177 µm applied to maize and wheat against *R. dominica*, *S. zeamais*, and *S. oryzae* to determine the efficacy of the two powders.

6.3. Materials and Methods

6.3.1. Insect rearing

Organic maize and hard red winter wheat (Heartland Mills, Marienthal, Kansas, USA) were cleaned manually using a 4.76 and 2.38 mm diameter round-holed aluminum sieves, respectively (Seedburo Equipment Company, Des Plaines, Illinois, USA) to remove broken kernels and dockage. The cleaned maize and wheat were frozen at -13ºC for five days to kill any live insects present. A 400 g cleaned, untreated maize with moisture content 13.5% (wet basis) was transferred to each of the six 0.95 L glass jars. One hundred unsexed adults of mixed ages of laboratory strains of *R. dominica*, *S. zeamais*, and *S. oryzae* were transferred to separate glass jars. Similarly, 400 g of cleaned organic wheat with moisture content ~12% (wet basis) was transferred to each of the six 0.95 L glass jars. One hundred unsexed adults of mixed ages of laboratory strains of *R. dominica*, *S. zeamais*, and *S. oryzae* were transferred to separate glass jars. All the glass jars containing grain and insects were covered with filter paper and wire-mesh screen lids to facilitate air diffusion. The originally added adults were removed after 14 d of incubation in an environmental growth chamber (Percival Scientific, Inc., Model I-36VL, Perry, Iowa, USA) at 28°C and 65% r.h. These jars were checked after six weeks to obtain newly emerged adults for use in our experiments.

6.3.2. Concentration response tests

A 100 g of cleaned, untreated maize and wheat with moisture contents 13.5 and ~12.5% (wet basis), respectively, were transferred to separate 0.45 L glass jars, and jars were covered with filter paper and wire-mesh screen lids to facilitate air diffusion. A brass frame steel US standard sieve #80 (mesh size of 177 µm) (Seedburo Equipment Company) was used to sift filter cake and Triplex powders. Powders that passed through the sieve were used to treat maize and wheat in the glass jars. Filter cake concentrations of 0 (untreated maize), 0.3, 0.5, 0.7, 1, 2, 3, 4 and 5 g/kg were added to separate jars containing maize, and Triplex concentrations of 0 (untreated maize), 1, 2, 3, 6, 8, and 10 g/kg were added to separate jars containing maize. Similarly, filter cake concentrations of 0 (untreated wheat), 0.3, 0.5, 0.7, 1, 2, and 3 g/kg were added to separate jars containing wheat. Triplex concentrations of 0 (untreated wheat), 0.5, 07, 1, 2, and 3 g/kg were added to another separate jars containing wheat. Each jar was manually shaken for one minute to ensure uniform distribution of powders on maize and wheat kernels.

6.3.3. Addition of insects to jars

Insects reared on maize were used in all tests using maize, and those reared on wheat were used in all tests using wheat. Twenty newly emerged adults of *R. dominica* were transferred to separate jars containing maize treated with 0, 0.3, 0.5, 0.7, 1, 2, 3, 4 and 5 g/kg of filter cake, and 0, 1, 2, 3, 6, 8 and 10 g/kg of Triplex. Another 20 newly emerged adults of *R. dominica* were transferred to separate jars containing 0, 0.3, 0.5, 0.7, 1, 2 and 3 g/kg of filter cake, and 0, 0.5, 0.7, 1, 2 and 3 g/kg of Triplex treated wheat. Similarly, 20 adults of each of *S. zeamais* or *S. oryzae* were transferred to maize treated with 0, 1, 2, 3, 4, and 5 g/kg of filter cake, and 0, 1, 2, 3, 6, 8 and 10 g/kg of Triplex. Twenty adults of *S. zeamais* or *S. oryzae* were transferred to wheat treated with 0, 0.3, 0.5, 0.7, 1, 2 and 3 g/kg of filter cake, and 0, 0.5, 0.7, 1, 2 and 3 g/kg of Triplex. All the jars containing grain and insects were kept at 28°C and 65 % r.h. for 14 d to determine mortality. Separate jars were used for each species-grain-powder-concentration combinations with three replications. After a 14 d exposure, maize and wheat samples from each jar was sifted using a 4.76 and 2.38 mm circular round-holed aluminum sieves, respectively, to separate insects from the grain. Adults that did not respond when gently prodded by a Camel's hair brush were considered dead.

A separate experiment with the same species-grain-powder-concentration combinations as explained above was set and held in an environmental chamber at 28°C and 65% r.h for 42 d to determine adult progeny production. The progeny produced was counted from each jar after 42 d.

6.3.4. Data analysis

The number of adults that died at 14 d was reported as percentage. The mean ± SE mortality of *R. dominica* adults on untreated maize was 1.6 ± 1.6 and 0% in tests with filter cake and Triplex, respectively. Mortality of *R. dominica* adults on untreated wheat was 0%. The mean ± SE mortality of *S. zeamais* adults on both untreated maize and wheat was 01.6 ± 1.6% in tests with filter cake and Triplex, respectively. The mean ± SE mortality of *S. oryzae* adults on untreated maize was 1.6 ± 1.6 and 3.3 ± 3.3% in tests with filter cake and Triplex, respectively. Therefore, in cases where mortality on untreated maize or wheat was greater than 0%, mortality data of each species exposed to filter cake and Triplex treated maize and wheat were corrected for responses in the control treatment (Abbott, 1925). Arcsine transformation was used for normalizing heteroscedastic treatment variances of uncorrected and corrected mortality data. Adult progeny production was determined after subtraction of the 20 originally added adults. Adult progeny production data was transformed to $\log_{10}(x+1)$ scale (Bartlett, 1947) to normalize heteroscedastic treatment variances. However, untransformed raw data of corrected mortality and adult progeny are presented in tables. Corrected mortality and progeny production data were subjected to three-way analysis of variance (ANOVA) to determine significant differences ($P < 0.05$) of main (concentration, species, and grain), and interactive effects (SAS Institute, 2012). Data on corrected mortality and progeny production were later subjected to one-way ANOVA to determine significant differences ($P < 0.05$) among concentrations and grains, and means were separated by Ryan-Einot-Gabriel-Welsch multiple range test (REGWQ) (SAS Institute, 2012).

6.4. Results

6.4.1. Mortality responses of insects at different concentrations

Three–way ANOVA showed that the mortality due to the main effects of concentration (F = 7.46, df = 10, 206) and grain (F = 49.32, df = 1, 206) were significant ($P < 0.0001$), whereas significant differences in mortality were not observed among species (F = 2.88; df = 2, 206; P = 0.0588). The mortality differences due to the associated interactions, concentration x grain (F = 2.32; df = 5, 206; P = 0.0453), and species x grain (F = 3.56; df = 2, 206; P = 0.0307) were also significant. All the remaining two and three way interactive effects were not significant (F, range = 0.32 to 1.3; df = 4, 206 and 20, 206; P, range = 0.1762 to 0.8669).

The mean ± SE mortality of *R. dominica* adults exposed to filter cake treated maize ranged from 2.3 ± 1.1 to 100% and those exposed to Triplex treated maize ranged from 0 to 45.0 ± 5.8%. Complete mortality of *R. dominica* was observed when adults were exposed to maize treated with 2–5 g/kg of filter cake, however, mortality of adults was less than 50% at the highest concentration of Triplex (10 g/kg).

The mean ± SE mortality of *R. dominica* adults exposed to filter cake treated wheat ranged from 73.3 ± 4.4 to 100%, and those exposed to Triplex treated wheat ranged from 40.0 ± 10.4 to 83.3 ± 4.4%. Complete mortality of adults was achieved when they were exposed to 2 and 3 g/kg of filter cake treated wheat, whereas the mortality was 83.3% at the highest concentration of Triplex (3 g/kg).

In tests with maize, one–way ANOVA showed significant differences in mortality of *R. dominica* among concentrations of filter cake (F = 79.69; df = 7, 23; $P < 0.0001$) and Triplex (F = 8.78; df = 5, 17; P = 0.0011). In tests with wheat, one–way ANOVA showed significant differences among concentrations of filter cake (F = 12.47; df = 5, 17; P = 0.0002) and Triplex

(*F* = 5.78; df = 4, 14; *P* = 0.0113). Mortality of *R. dominica* adults was significant between maize and wheat treated with 0.3 and 0.5 g/kg of filter cake (*F* = 13.55; df = 1, 5; *P* = 0.0212 at 0.3 g/kg and *F* = 131.38; df = 1, 5; *P* = 0.0003 at 0.5 g/kg). However, no significant differences were observed in *R. dominica* adult mortality at each of the remaining concentrations between maize and wheat (*F*, range among grains = 0.58–1.0; df = 1, 5; *P*, range = 0.3739–0.9955) (Table 6.1). Significantly greater mortality of *R. dominica* was observed when adults were exposed to Triplex treated wheat at concentrations of 1, 2 and 3 g/kg compared to those exposed to similar concentrations on maize (*F*, range among grains = 51.89–75.56; df = 1, 5; *P*, range = 0.0010–0.0020) (Table 6.2).

The mean ± SE mortality of *S. zeamais* adults exposed to maize treated with filter cake and Triplex ranged from 50.0 ± 2.9 to 90.0 ± 2.9 % and 2.8 ± 2.8 to 64.4 ± 2.9%, respectively. Complete mortality was not achieved when adults were exposed to maize at all concentrations of filter cake and Triplex. The mean ± SE corrected mortality of *S. zeamais* adults on wheat treated with filter cake and Triplex ranged from 51.7 ± 10.9 to 100% and 57.6 ± 4.5 to 91.5 ± 3.4%, respectively. On wheat 100% mortality was achieved at filter cake concentrations of 0.7–3 g/kg, whereas adult mortality was 91.5% at the highest Triplex concentration of 3 g/kg.

On maize, one–way ANOVA showed significant differences in mortality of *S. zeamais* among concentrations of filter cake (*F* = 6.80; df = 4, 14; *P* = 0.0065) and Triplex (*F* = 31.93; df = 5, 17; *P* < 0.0001). On wheat one–way ANOVA showed significant differences in mortality among concentrations of filter cake (*F* = 31.35; df = 5, 17; *P* < 0.0001) and Triplex (*F* = 9.89; df = 4, 14; *P* = 0.0017). Significantly greater mortality of *S. zeamais* adults was observed in 1, 2 and 3 g/kg of filter cake treated wheat compared to those observed at the same concentrations on maize (*F*, range among grains = 17.23 –737.75; df = 1, 5; *P*, range < 0.0001 – 0.0143) (Table

6.1). Significantly greater mortality of *S. zeamais* adults was observed at Triplex concentrations of 1, 2 and 3 g/kg on wheat compared to adults exposed to similar concentrations on maize (*F*, range among grains = 82.62 – 173.47; df = 1, 5; *P*, range = 0.0002 – 0.0008) (Table 6.2).

The mean ± SE mortality of *S. oryzae* adults on maize treated with filter cake ranged from 69.0 ± 3.0 to 91.4 ± 8.6% and mortality of adults exposed to Triplex ranged from 32.2 ± 4.5 to 94.9 ± 2.9%. On maize, irrespective of the powder used, mortality of adults was less than 100% at all concentrations tested.

The mean ± SE corrected mortality of *S. oryzae* adults on wheat treated with filter cake ranged from 41.7 ± 14.8 to 100% and those exposed to Triplex ranged from 56.7 ± 4.4 to 90.0 ± 2.9%. On filter cake treated wheat, 100% mortality was achieved at concentrations of 0.7–3 g/kg. However, the mortality of *S. oryzae* adults was 90.0% at the highest concentration (3 g/kg) of Triplex.

On maize one–way ANOVA did not show significant differences in mortality of *S. oryzae* adults among concentrations of filter cake (*F* = 1.45; df = 4, 14 ; *P* = 0.2867). However, significant differences in mortality were found among concentrations of Triplex (*F* = 16.30; df = 5, 17; *P* < 0.0001). On wheat one–way ANOVA showed significant differences in mortality of *S. oryzae* adults among concentrations of filter cake (*F* = 20.64; df = 5, 17; *P* < 0.0001) and Triplex (*F* = 8.02; df = 4, 14; *P* = 0.0037). Significantly greater mortality of *S. oryzae* adult was observed on wheat at filter cake concentrations of 1 and 2 g/kg compared with mortality observed at similar concentrations on maize (*F* = 332.02; df = 1, 5; *P* < 0.0001 at 1 g/kg and *F* = 37.03; df = 1, 5; *P* = 0.0037 at 2 g/kg) (Table 6.1). Similarly, significantly greater mortality of *S. oryzae* adults on wheat was observed in 1, 2 and 3 g/kg concentrations of Triplex compared with mortality observed on maize at similar concentrations (*F*, range among grain = 9.92–91.34; df = 1, 5; *P*, range = 0.0008–0.0345) (Table 6.2).

6.4.2. Adult progeny production at 42 d

Three–way ANOVA showed that the progeny production was significantly different among concentrations (*F* = 32.93, df = 11, 251, *P* < 0.0001) and among the species (*F* = 85.15, df = 2, 251, *P* < 0.0001). However, progeny production between the two types of grain was not significant (*F* = 1.15; df = 1, 251; *P* = 0.2839). The concentration × grain interaction (*F* = 10.26; df = 6, 251; *P* < 0.0001) and species × grain interaction (*F* = 14.64; df = 2, 251; *P* < 0.0001) were significant. All of the remaining two way and three way interactive effects were not significant (*F*, range = 0.63 to 0.56; df = 6, 251 and 22, 251; *P*, range = 0.7101 to 0.9453).

The mean ± SE number of *R. dominica* adult progeny produced after 42 d of exposure to maize treated with filter cake ranged from 0 to 29.0 ± 9.6 adults per jar and those exposed to Triplex ranged from 1.0 ± 0.6 to 31.3 ± 8.8 adults per jar. More progeny were produced at lower than high concentrations of both powders. On maize, progeny production was not observed when adults were exposed to 1–5 g/kg of filter cake (1000 reduction in progeny production). However, on maize 100% reduction in progeny production was not achieved at all of Triplex concentrations. The mean ± SE number of progeny of *R. dominica* produced after 42 d of exposure to wheat treated with filter cake ranged from 0 to 204.7 ± 26.1 adults per jar and those exposed to Triplex ranged from 5.3 ± 0.9 to 207.0 ± 10.7 adults per jar. On wheat, progeny production was not observed when adults were exposed to filter cake concentration of 2–3 g/kg. However, on wheat 100% reduction in progeny production was not achieved when adults were exposed to all Triplex concentrations.

On maize, one–way ANOVA showed significant differences in number of *R. dominica* adult progeny producted among concentrations of filter cake (*F* = 15.77; df = 8, 26; *P* < 0.0001), and Triplex (*F* = 10.04; df = 9, 29; *P* < 0.0001). On wheat, one–way ANOVA showed significant differences in *R. dominica* progeny production among concentrations of filter cake (*F* = 20.96; df = 6, 20; *P* < 0.0001) and Triplex (*F* = 142.44; df = 5, 17; *P* < 0.0001). Significantly less number of adult progeny of *R. dominica* was produced at higher concentrations of filter cake (Table 6.3) and Triplex (Table 6.4) compared to lower concentrations on both grains, a result of greater mortality at higher concentrations.

The mean ± SE number of *S. zeamais* adult progeny produced after 42 d of exposure to maize treated with filter cake ranged from 26.0 ± 8.7 to 201.0 ± 18.4 adults per jar and those exposed to Triplex ranged from 57.7 ± 3.5 to 192.7 ± 32.5 adults per jar, with the highest number of progeny being produced at lower than higher concentrations. On maize, complete suppression of *S. zeamais* progeny production was not achieved at all filter cake and Triplex concentrations. The mean ± SE number of *S. zeamais* progeny produced after 42 d of exposure to wheat treated with filter cake ranged from 0 to 587.7 ± 124.3 adults per jar and those exposed to Triplex ranged from 15.7 ± 3.2 to 672.0 ± 53.4 adults per jar, with the highest number of progeny being produced at lower than higher concentrations. On wheat, no adult progeny was produced when adults were exposed to 3 g/kg of filter cake. However, on wheat, complete suppression of progeny production was not achieved when adults were exposed all Triplex concentrations.

On maize, one–way ANOVA showed significant differences in *S. zeamais* adult progeny production among concentrations of filter cake (*F* = 14.32; df = 5, 17 ; *P* = 0.0001), and Triplex (*F* = 20.94; df = 6, 20; *P* < 0.0001). On wheat, one–way ANOVA showed significant differences in *S. zeamais* adult progeny production among concentrations of filter cake (*F* = 34.41; df = 6, 20; *P* <0.0001), and Triplex (*F* = 80.81; df = 5, 17; *P* < 0.0001). Significantly less number of adult progeny of *S. zeamais* was produced at higher than lower concentrations of filter cake (Table 6.3) and Triplex (Table 6.4) on both grains.

The mean ± SE number of *S. oryzae* adult progeny of produced after 42 d of exposure to maize treated with filter cake ranged from 5.0 ± 5.0 to 131.3 ± 19.7 adults per jar and those exposed to Triplex ranged from 25.7 ± 4.9 to 162.0 ± 2.5 adults per jar, with the highest number of progeny being produced at lower concentrations. On maize, complete suppression of progeny production was not achieved at all concentrations of filter cake and Triplex. The mean ± SE number of *S. oryzae* adult progeny of produced after 42 d of exposure to wheat treated with filter cake ranged from 3.7 ± 1.9 to 535.7 ± 29.2 adults per jar and those exposed to Triplex ranged from 8.3 ± 2.0 to 656.3 ± 182.1 adults per jar, with the highest number of progeny being produced at lower concentrations. On wheat, complete suppression of progeny production was not achieved at all filter cake and Triplex concentrations.

On maize, one–way ANOVA showed significant differences in *S. oryzae* adult progeny production among concentrations of filter cake (*F* = 8.07; df = 5, 17; *P* = 0.0015) and Triplex (*F* = 5.65; df = 6, 20; *P* = 0.0036). On wheat, one–way ANOVA showed significant differences in *S. oryzae* adult progeny production among concentrations of filter cake (*F* = 18.35; df = 6, 20; *P* <0.0001) and Triplex (*F* = 22.81; df = 5, 17; *P* < 0.0001). Significantly less number of adult progeny of *S. oryzae* was produced at higher than lower concentrations of filter cake (Table 6.3) and Triplex (Table 6.4) on both grains.

On maize, one–way ANOVA showed significant differences in adult progeny production among the species at all concentrations of filter cake (*F*, range among species = 8.25 to 789.11; df = 2, 8; *P*, range < 0.0001 to 0.0189) and Triplex (*F*, range among species = 13.40 to 616.35;

df = 2, 8; *P*, range < 0.0001 to 0.0061). On wheat, one–way ANOVA did not show significant differences in adult progeny production among the species at all concentrations of filter cake (*F*, range among species = 0.83 to 3.62; df = 2, 8; *P*, range = 0.0794 to 0.4801;). On wheat, one–way ANOVA showed significant differences in adult progeny production among the species at all concentrations of Triplex (*F*, range among species = 6.91 to 17.31; df = 2, 8; *P*, range = 0.00032 to 0.0278; one–way ANOVA). In general, *R. dominica* produced less number of progeny on both grains treated with filter cake and Triplex compared to *S. oryzae* and *S. zeamais*.

6.5. Discussion

Our data demonstrated the efficacy of filter cake and Triplex against *R. dominica*, *S. zeamais* and *S. oryzae* adults in response to powder concentrations and grain types. Admixing of powder with maize and wheat was done manually, where uneven distribution of powder over the maize and wheat kernel surfaces was more likely to occur. Accumulation of powder over insect's body is directly proportional to concentration of the powder (Le Patourel et al., 1989), and insect's behavior such as mobility (Malia et al., 2016b). The toxicity of such powders will depend on particle accumulation from the maize and wheat on to the insect's body and adsorption of lipid by powder particles from the insect cuticle (Le Patourel et al., 1989; Filipović et al., 2010). Therefore, uneven distribution of filter cake and Triplex on maize and wheat might have contributed to unexplained variations in mortality and progeny production responses of the tested species.

We hypothesize that filter cake and Triplex have properties similar to silica-based inert dusts, because of their higher atomic percentage of silicon and oxygen in the form of silicon dioxide (Tadesse and Subramanyam, 2018a). The proportion of silicon and oxygen as silicon dioxide in both filter cake and Triplex was smaller than that found in diatomaceous earth powders, and therefore, our results cannot be compared directly with the research done on diatomaceous earth powders by numerous researchers (Arthur, 2002; Athanssiou et al., 2003, 2008; Kljajić et al., 2010; Nukenine et al., 2010; Jean et al., 2015). However, mortality of all tested species tended to increase with increasing filter cake and Triplex concentrations on maize and wheat. Similar results were reported by Athanssiou et al. (2003, 2008) and Arthur (2002). It was suggested by several researchers that the insecticidal efficacy of different diatomaceous earth was affected by grain type (Athanassiou et al., 2003, 2008; Athanassiou and Kavallieratos, 2005; Kavallieratos et al., 2005; Vassilakos et al., 2015; Fazlabad et al, 2017). For example, Athanassiou et al. (2008) reported that the susceptibility of *S. oryzae* to diatomaceous earth was lower in maize than wheat. A similar report by Athanassiou et al. (2003) indicated that *S. oryzae* adults were less susceptible to SilicoSec® when exposed to treated maize compared to exposure on treated pealed rice and barley. Kavallieratos et al. (2005) reported significant differences in mortality of *R. dominica* adults among eight grain types after a 14 d exposure to Insecto and SilicoSec® treated grains. They reported that mortality of *R. dominica* was higher in Insecto and SilicoSec® treated wheat compared to the rest of the grains. A similar phenomenon was observed in our experiment. We observed significantly greater mortality of *R. dominica*, *S. zeamais* and *S. oryzae* adults exposed to filter cake and Triplex treated wheat compared to maize. Complete mortality of *R. dominica* adults was observed on maize and wheat treated with 3–5 and 2–3 g/kg of filter cake, respectively. Complete mortality of *S. zeamais* and *S. oryzae* adults was observed when adults were exposed to wheat treated with 0.7–3 g/kg of filter cake. Greater mortality of *R. dominica, S. zeamais* and *S. oryzae* adults was observed on wheat compared to maize at all concentrations of filter cake and Triplex.

Complete suppression of adult progeny production of *R. dominica* was achieved when adults were exposed to maize and wheat treated with 1–5 and 1–3 g/kg of filter cake, respectively. Although adults might have been killed by exposure to filter cake and Triplex some oviposition may still occur as these powders do not kill adults rapidly (Tadesse and Subramanyam, 2018b). Therefore, progeny production was observed especially at the lower powder concentrations tested. In our experiment, we found that progeny of *R. dominica*, *S. zeamais*, and *S. oryzae* were found dead on both maize and wheat treated with ≥ 1 g/kg of filter cake. However, complete suppression of adult progeny production was not achieved at any of Triplex concentrations on both grains. Adult progeny production tended to decrease with increasing filter cake and Triplex concentrations on both maize and wheat. The greater mortality of *R. dominica*, *S. zeamais* and *S. oryzae* adults at increasing concentrations of filter cake and Triplex might have caused significant reduction in adult progeny production. A similar phenomenon was reported by Tadesse and Subramanyam (2018b) after a 42 d exposure of *S. zeamais* and *S. oryzae* adults to filter cake and Triplex treated wheat. Tadesse et al. (2018a,b) also reported a decreasing trend of *S. zeamais* and *S. oryzae* adult progeny production with an increase in concentration of filter cake and Triplex on concrete surfaces.

Tadesse and Subramanyam (2018a,b) and Tadesse et al. (2018a,b) reported that filter cake was consistently more efficacious than Triplex against *S. zeamais* and *S. oryzae* under laboratory conditions. The present study showed that filter cake was consistently more efficacious against *R. dominica*, *S. zeamais* and *S. oryzae* compared to Triplex on both maize and wheat. However, traditional storages in Ethiopia, such as *gota* and *gotera* are made with teff straw, mud, and cow dung (Abraham et al., 2008). A survey conducted in three major grain producing areas of Ethiopia indicated that 93.3% (*n* = 300) used traditional storages, which predisposes stored grains to insect infestation (Gabriel and Hundie, 2006). Therefore, including filter cake and Triplex as part of an integrated pest management program in smallholder farmers' traditional storage structures will be useful to protect maize and wheat from *R. dominica*, *S. zeamais* and *S. oryzae* infestations. Filter cake and Triplex can be recommended to be used in combination of Purdue Improved Crop Storage (PICS) bags, GrainPro Super bags, plastic drums, and metal silos (Obeng-Ofori, 2011; Murdock and Baoua, 2014; Chigoverah and Mvumi, 2016). Field studies in Ethiopia need to be conducted in smallholder farmers' traditional storages, which include above-ground structures like *gota* and *gotera*, and underground pits (Tadesse and Eticha 2000, Blum and Bekele 2001, Dessalegn et al. 2017) to make recommendations to smallholder farmers.

6.6. References

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Table 6.1. Mortality of *R. dominica*, *S. zeamais*, and *S. oryzae* adults after a 14 d exposure to filter cake treated maize and wheat.

Concentration (g/kg)	Corrected adult mortality (%mean ± SE)[a,b]					
	R. dominica		*S. zeamais*		*S. oryzae*	
	Maize	Wheat	Maize	Wheat	Maize	Wheat
0.3	2.3 ± 1.1D,b	73.3 ± 4.4B,a	–[c]	51.7 ± 10.9B	–	41.7 ± 14.8B
0.5	55.9 ± 6.1C,b	80.0 ± 2.9B,a	–	96.7 ± 1.7A	–	93.3 ± 3.3A
0.7	84.7 ± 5.9B	90.0 ± 5.0AB	–	100.0 ± 0.0A	–	100.0 ± 0.0A
1	98.3 ± 1.7A	98.3 ± 1.7A	50.0 ± 2.9B,b	100.0 ± 0.0A,a	69.0 ± 3.0b	100.0 ± 0.0A,a
2	98.3 ± 1.7A	100.0 ± 0.0A	70.0 ± 12.6AB,b	100.0 ± 0.0A,a	79.3 ± 6.0b	100.0 ± 0.0A,a
3	100.0 ± 0.0A	100.0 ± 0.0A	76.7 ± 1.7AB,b	100.0 ± 0.0A,a	82.8 ± 8.6	100.0 ± 0.0A
4	100.0 ± 0.0A	–	85.0 ± 2.9A	–	82.8 ± 6.2	–
5	100.0 ± 0.0A	–	90.0 ± 2.9A	–	91.4 ± 8.6	–

[a] Means within a commodity followed by different upper letters were significantly different (*F*, range = 6.80–79.69; df = 7, 23 for *R. dominica*; and 4, 14 for *S. zeamais* on maize; and 5, 17 for the remaining grain-species combinations; *P*, range < 0.0001 – 0.0065; one–way ANOVA with means were separated by REGWQ)

[b] Means between maize and wheat by species followed by different lower letters were significantly different (*F*, range = 13.55–635.10; df = 1, 5; *P*, range < 0.0001 – 0.0212; one–way ANOVA with means were separated by REGWQ)

[c] These concentrations were not tested

Table 6.2. Mortality of *R. dominica*, *S. zeamais*, and *S. oryzae* adults after a 14 d exposure to Triplex treated maize and wheat.

Concentration (g/kg)	Corrected adult mortality (%mean ± SE)[a,b]					
	R. dominica		*S. zeamais*		*S. oryzae*	
	Maize	Wheat	Maize	Wheat	Maize	Wheat
0.5	–[c]	40.0 ± 10.4B	–	57.6 ± 4.5B	–	56.7 ± 4.4C
0.7	–	41.7 ± 10.1B	–	78.0 ± 4.5A	–	70.0 ± 5.8BC
1	3.3 ± 1.7B,b	55.0 ± 5.8AB,a	2.8 ± 2.8B,b	86.4 ± 4.5A,a	32.2 ± 4.5C,b	85.0 ± 2.9AB,a
2	5.0 ± 2.9B,b	65.0 ± 5.8AB,a	10.2 ± 3.4B,b	88.1 ± 1.7A,a	57.6 ± 6.8BC,b	86.7 ± 6.0AB,a
3	3.3 ± 1.7B,b	83.3 ± 4.4A,a	11.9 ± 4.5B,b	91.5 ± 3.4A,a	57.6 ± 4.5BC,b	90.0 ± 2.9Aa
6	11.7 ± 1.7B	–	57.6 ± 4.5A	–	61.0 ± 4.5BC	–
8	16.7 ± 7.3B	–	61.0 ± 4.5A	–	79.7 ± 5.9B	–
10	45.0 ± 5.8A	–	64.0 ± 2.9A	–	94.9 ± 2.9A	–

[a] Means within a commodity followed by different upper letters were significantly different (*F*, range = 5.78–31.93; df = 5, 17 for maize, 4, 14 for wheat; *P*, range < 0.0001 – 0.0113; one–way ANOVA with means were separated by REGWQ)

[b] Means between maize and wheat by species followed by different lower letters were significantly different (*F*, range = 9.92–173.47; df = 1, 5; *P*, range < 0.0001 – 0.0345; one–way ANOVA with means were separated by REGWQ)

[c] These concentrations were not tested

Table 6.3. Number of adult progeny produced by *R. dominica*, *S. zeamais*, and *S. oryzae* after a 42 d exposure to filter cake treated maize and wheat.

Commodity	Concentration (g/kg)	Number of adult progeny (mean ± SE)[a,b]		
		R. dominica	*S. zeamais*	*S. oryzae*
Maize	0	29.0 ± 9.6A,b	201.0 ± 18.4Aa	131.3 ± 19.7Aa
	0.3	4.7 ± 2.6B	–[c]	–
	0.5	3.0 ± 0.6B	–	–
	0.7	0.3 ± 0.3B	–	–
	1	0.0 ± 0.0B,c	66.7 ± 2.9B,a	20.3 ± 2.8B,b
	2	0.0 ± 0.0B,c	57.0 ± 3.2BC,a	14.3 ± 3.4BC,b
	3	0.0 ± 0.0B,c	34.7 ± 8.3BC,a	13.3 ± 4.8BC,b
	4	0.0 ± 0.0B,c	33.0 ± 7.8BC,a	7.7 ± 3.8BC,b
	5	0.0 ± 0.0B,b	26.0 ± 8.7C,a	5.0 ± 5.0C,b
Wheat	0	204.7 ± 26.1A,b	587.7 ± 124.3A,a	535.7 ± 29.2A,a
	0.3	100.3 ± 60.4AB	169.3 ± 26.3AB	204.0 ± 23.8AB
	0.5	54.7 ± 14.0AB	57.0 ± 19.5B	102.3 ± 33.2AB
	0.7	13.0 ± 6.2BC	31.3 ± 4.7B	64.3 ± 18.4BC
	1	1.7 ± 1.7CD	5.3 ± 2.7C	13.7 ± 5.4CD
	2	0.0 ± 0.0D	1.3 ± 1.3C	6.0 ± 3.8D
	3	0.0 ± 0.0D	0.0 ± 0.0C	3.7 ± 1.9D

[a] Means among concentrations by species followed by different upper letters were significantly different (*F*, range = 8.07–34.41; df = 8, 26 for *R. dominica*, 5, 17 for *S. zeamais* and *S. oryzae*

in maize, and 6, 20 for all species in wheat; *P*, range < 0.0001 – 0.0065; one–way ANOVA with means were separated by REGWQ multiple range test).

[b] among species at each concentration followed by different lower letters were significantly different (*F*, range = 8.25–789.11; df = 2, 8; *P*, range < 0.0001 – 0.0189; one–way ANOVA with means were separated by REGWQ multiple range test).

[c] These concentrations were not tested.

Table 6.4. Number of adult progeny produced by *R. dominica*, *S. zeamais*, and *S. oryzae* after a 42 d exposure to Triplex treated maize and wheat.

Commodity	Concentration (g/kg)	Mortality (%mean ± SE)[a,b]		
		R. dominica	*S. zeamais*	*S. oryzae*
Maize	0	31.3 ± 8.8A,c	192.7 ± 32.5A,a	162.0 ± 2.5A,b
	0.3	17.7 ± 5.8AB	–[c]	–
	0.5	8.0 ± 1.2BC	–	–
	0.7	9.0 ± 2.6BC	–	–
	1	7.3 ± 2.4BC,b	120.7 ± 3.5B,a	77.7 ± 2.6AB,a
	2	6.7 ± 1.8BC,b	119.0 ± 8.9B,a	53.7 ± 24.0B,a
	3	5.3 ± 1.8BCD,c	103.3 ± 2.2BC,a	34.0 ± 9.5B,b
	6	4.3 ± 0.3BCD,c	101.0 ± 2.3BC,a	44.3 ± 3.9B,b
	8	2.0 ± 0.6CD,c	78.3 ± 6.9CD,a	32.3 ± 1.9B,b
	10	1.0 ± 0.6D,c	57.7 ± 3.5D,a	25.7 ± 4.9B,b
Wheat	0	207.0 ± 10.7A,b	672.0 ± 53.4A,a	656.3 ± 182.1A,a
	0.5	95.7 ± 8.1B,c	159.3 ± 10.2B,b	304.3 ± 55.9A,a
	0.7	44.3 ± 5.5C,b	129.6 ± 25.2BC,a	220.7 ± 52.8AB,a
	1	17.7 ± 2.0D,b	72.3 ± 2.0CD,a	81.0 ± 38.3BC,a
	2	8.3 ± 1.5E,b	58.0 ± 8.0D,a	27.7 ± 14.7CDa,b
	3	5.3 ± 0.9E	15.7 ± 3.2E	8.3 ± 2.0D

[a] Means among concentrations by species followed by different upper letters were significantly different (*F*, range = 5.65–142.44; df = 9, 29 for *R. dominica*, 6, 20 for *S. zeamais* and *S. oryzae*

in maize, 5, 17 for all species in wheat; *P*, range < 0.0001 – 0.0113; ; one–way ANOVA with means were separated by REGWQ multiple range test).

[b] Means among species at each concentration followed by different lower letters were significantly different (*F*, range = 6.91–616.35; df = 2, 8; *P*, range < 0.0001 – 0.0278; ; one–way ANOVA with means were separated by REGWQ multiple range test).

[c] These concentrations were not tested.

Chapter 7 - Efficacy of filter cake and Triplex powders against three externally developing stored product insect pests

7.1. Abstract

The efficacy of filter cake and Triplex powders applied to maize and wheat was evaluated in the laboratory against the red flour beetle, *Tribolium castaneum* (Herbst); sow-toothed grain beetle, *Oryzaephilus surinamensis* (Linnaeus); and Indian meal moth, *Plodia interpunctella* (Hübner). These species are the common insect pests associated with stored grains in Ethiopia. Efficacy of these powders was determined by exposing 20 adults of each species to 100 g of maize and wheat treated with 0, 0.3, 0.5, 0.7, 1, 2, and 3 g/kg of filter cake and 0, 0.5, 0.7, 1, 2, 3, 6, 8 and 10 g/kg of Triplex. Mortality of *T. castaneum* and *O. surinamensis* adults was determined 14 d after exposure to untreated and treated grains. Adult progeny production of *T. castaneum* and *O. surinamensis* at each species-powder-concentration combination was determined at 42 d. Number of live larvae at 21 d and number of adult of *P. interpunctella* that emerged at 42 d were determined by exposing 100 eggs to 0, 0.2, 0.3, 0.5, 0.7, 1, 2, and 3 g/kg of filter cake and 0, 0.3, 0.5, 0.7, 1, 2, 3, 6, and 8 g/kg of Triplex treated maize and wheat. On both commodities, mortality was 100% for adults of *T. castaneum* and *O. surinamensis* exposed to 2–3 and 1–3 g/kg of filter cake, respectively. At 2-3 g/kg of Triplex treated wheat, 100% mortality was achieved only with *O. surinamensis*. On both maize and wheat, adult progeny production of *T. castaneum* and *O. surinamensis* was completely suppressed at filter cake concentrations of 0.7–3 and 1–3 g/kg, respectively. On both maize and wheat, adult progeny production of *T. castaneum* was not observed at Triplex concentrations of 1–3 g/kg. Complete suppression of *O. surinamensis* adult progeny production was achieved at Triplex concentrations of 2–3 g/kg only on maize. Both the number of live larvae at 21 d and adults of *P. interpunctella* that emerged at

42 d were completely suppressed when eggs were exposed to filter cake concentrations of 2–3 and 0.5–3 g/kg on maize and wheat, respectively. Complete suppression of both number of live larvae at 21 d and adults emerged at 42 d were achieved at Triplex concentrations of 6–8 and 3 g/kg on maize and wheat, respectively. Both powders work better on wheat than on maize. Filter cake was consistently more efficacious compared to Triplex and can be used an alternative to chemical insecticides to manage *T. castaneum*, *O. surinamensis*, and *P. interpunctella* in smallholder farmers' traditional storage structures in Ethiopia.

Keywords: Filter cake; Triplex; maize; Wheat; *T. castaneum, O. surinamensis*; *P. interpunctella*; Efficacy assessment

7.2. Introduction

The average yield of cereal crops in Ethiopia showed an increasing trend between 2001 and 2017 (Cochrane and Bekele, 2018). The Ethiopian Central Statistical Agency (CSA) survey data of the '*meher*' crop of 2015-16 indicated that cereal grains contributed 86.7% of the total grain production in Ethiopia (CSA, 2016). Maize and wheat are two of the most important cereal grains in Ethiopia (Workneh, 2003), and in 2015-16 contributed to 26.8 and 15.8% of total cereal grain production (CSA, 2016). However, inefficient postharvest management practices in Ethiopia lead to 20 to 30% loss of harvested grains, including maize and wheat (Tefera, 2016). Storage insect pests are responsible for the quantitative and qualitative losses of cereal grains worldwide (Phillips and Throne, 2010). Grain storage losses in Ethiopia due to insect pests was estimated to be in the range of 10 to 21% (Abraham et al., 2008). Major insects that contribute to post-harvest loss of cereals and pulses in Ethiopia include grain weevils, grain borers, grain beetles, and grain moths (Abate et al., 2000; Abraham et al., 2008; Nukenine, 2010).

Smallholder farmers in Ethiopia apply pesticides to stored grains to manage insect infestations (Williamson et al., 2008). In Ethiopia, lack of information and resources, weak interaction, lack of motivation of state actors, and lack of technical knowledge in safe handling of pesticides have contributed to improper and misuse of pesticides by end users (Mengistie et al., 2017; Loha et al., 2018). Unsafe handling of pesticides has resulted in ill health episodes, hospitalizations, and fatalities soon after a pesticide application (Williamson et al., 2008; G/Mariam and Gelaw, 2016).

Major issues with the use of pesticides is toxicity to mammals, persistence of residues on grains, and development of resistance in insects (Arthur, 1996). Safer alternatives to pesticides have been explored by several researchers (Athanassiou and Kavallieratos, 2005; Athanassiou et al., 2005, 2008). Inert dusts have been identified as safer alternatives to chemical pesticides for a long period of time (Ebeling, 1971). Inert dusts are dry dusts that are chemically unreactive in nature (Liška et al., 2017; Bohinc et al., 2018), and are classified into different groups based on their physical and chemical composition (Subramanyam and Roesli, 2000; Stadler et al., 2012). Some inert dusts, such as clay, sand, ground phosphate, ash, diatomaceous earths have been for a long period of time in North America and Africa (Headlee, 1924; Ebeling, 1971; Subramanyam and Roesli, 2000; Liška et al., 2017). One group of inert dusts are silica based and produce a toxic effect to insects by adsorbing epicuticular lipids from insect's body leading to death by desiccation (Ebeling, 1971; Subramanyam and Roesli, 2000; Malia et al., 2016).

Wood ash, lime, sand, and tobacco were used with limited success for stored product insect control in Ethiopia (Workneh, 2003; Abraham et al., 2008). However, in Ethiopia limited studies have investigated inert dusts for management of insects in stored grains (Abraham et al., 2008; Girma et al., 2008a,b; Tadesse and Subramanyam, 2018a,b; Tadesse et al., 2018a,b). Filter cake and Triplex, by-products of aluminum sulfate and soap factories, respectively from Ethiopia were recently identified as two of the alternatives to chemical pesticides (Girma et al., 2008a,b; Tadesse and Subramanyam, 2018a,b).

Filter cake and Triplex have higher atomic percentage of silicon and oxygen in the form of silicon dioxide (Tadesse and Subramanyam, 2018a), and the mode of action may be similar to other silica-based dusts (Ebeling, 1971; Subramanyam and Roesli, 2000; Malia et al., 2016). Mortality of *S. zeamais* adults was100% after a 24 h exposure to 7.5 g/m^2 of filter cake dusted concrete arenas (Tadesse and Subramanyam, 2018a). In another study on concrete arenas using smaller particle sizes of filter cake, Tadesse et al. (2018a) reported complete mortality of *S.*

zeamais adults after a 12 h exposure to 3–8 g/m^2 of filter cake. In the case of *S. oryzae* complete mortality of adults was observed after a 12 h exposure to 4–8 g/m^2 of filter cake applied to concrete arenas (Tadesse et al., 2018b). Significantly greater mortality, and lower adult progeny production of *S. zeamais* and *S. oryzae* at 42 d were achieved after exposure to 0.1, 0.5, 0.7 and 1g/kg of filter cake and Triplex treated wheat for only 7 and 14 d (Tadesse and Subramanyam, 2018b).

To the best of our knowledge, there are no data on efficacy of filter cake and Triplex against the red flour beetle, *Tribolium castaneum* (Herbst); saw-toothed grain beetle, *Oryzaephilus surinamensis* (Linnaeus); and Indian meal moth, *Plodia interpunctella* (Hübner) at a range of concentrations applied to maize and wheat. All the three species are externally developing insect pests (Phillips and Throne, 2010). These three species are listed common insect pests of stored grains and grain products throughout the world (Hills, 2008), and specifically grouped as primary pests of stored grains in Ethiopia (Abraham et al., 2008; Nukenine, 2010; Befikadu, 2018). Therefore, this study was designed to determine the efficacy of filter cake and Triplex with particle size less than 177 µm against *T. castaneum*, *O. surinamensis* and *P. interpunctella* on maize and wheat.

7.3. Materials and Methods

7.3.1. Insect rearing

Cultures of *T. castaneum* were reared on hard red winter wheat flour (Heartland Mills, Marienthal, Kansas, USA) fortified with 5% (w/w) brewer’s yeast. *O. surinamensis* cultures were reared on organic rolled oats (Heartland Mills). *P. interpunctella* cultures were reared on poultry-mash based diet prepared from poultry-mash (1000 g), glycerol (150 ml), honey (75 ml) and distilled water (75 ml) (Subramanyam and Cutkomp, 1987). Each species was reared separately in 0.9 L glass jars filled with 400 g of the respective diets at 28°C and 65% r.h in an environmental growth chamber (Percival Scientific, Inc., Model I-36VL, Perry, Iowa, USA).

7.3.2. Sample preparation

A 100 g of cleaned, untreated organic maize and wheat with moisture contents 13.5 and ~12.5% (wet basis), respectively, were transferred to separate 0.45 L glass jars. Jars were covered with filter paper and wire-mesh screen lids to facilitate air diffusion. In tests with *T. castaneum* 2% (w/w) of dockage (broken kernels) was added to each jar containing maize and wheat to support larval-to-adult development. Survival, development, and multiplication of externally developing stored product insects in wheat, including *T. castaneum* is influenced by dockage (Sinha, 1975).

A brass US standard sieve #80 (mesh size of 177 µm) (Seedburo Equipment Company, Des Plaines, Illinois, USA) was used to sift filter cake and Triplex powders. Powders that passed through the sieve were used to treat maize and wheat in glass jars. Filter cake concentrations of 0 (untreated maize), 0.2, 0.3, 0.5, 0.7, 1, 2 and 3 g/kg were added to separate jars containing maize and wheat. Similarly, Triplex concentrations of 0 (untreated maize), 0.3, 0.5, 0.7, 1, 2, 3, 6, 8 and 10 g/kg were added to separate jars containing maize and wheat. Each jar was manually shaken for one minute to ensure uniform distribution of powders on maize and wheat kernels.

7.3.3. Concentration response tests

Twenty unsexed 1-3 weeks old adults of *T. castaneum* were transferred to separate 0.45 L jars containing maize and wheat treated with 0, 0.3, 0.5, 0.7, 1, 2 and 3 g/kg of filter cake, and 0, 0.3, 0.5, 0.7, 1, 2, 3, 6, 8 and 10 g/kg of Triplex. Twenty unsexed 1-3 weeks old adults of *O. surinamensis* were transferred to 0, 0.3, 0.5, 0.7, 1, 2 and 3 g/kg of filter cake and Triplex treated maize and wheat, separately. All the jars containing *T. castaneum* and *O. surinamensis* were kept for 14 d at 28°C and 65 % r.h. to determine mortality. Separate jars were used for each species-grain-powder-concentration combinations with three replications. After a 14 d exposure, maize and wheat from each container were sifted using a 4.76 and 2.38 mm circular round-holed aluminum sieve, respectively, to separate adults. Adults that did not respond when gently prodded by a Camel's hair brush were considered dead.

P. interpunctella eggs laid within 24 h were collected for use in the bioassay experiments. For egg collection, *P. interpunctella* adults from culture jars were briefly anesthetized with carbon dioxide (Silhacek and Miller, 1972). Anesthetized adults were rapidly transferred to 0.95 L glass jars with wire mesh lid. The wire mesh used had 841 µm openings, which would allow eggs laid by *P. interpunctella* females to pass through. The jars were inverted over 9 cm diameter glass Petri dishes to collect eggs. Eggs collected with 24 h were used in tests. One hundred eggs were transferred to each jar containing maize treated with 0, 0.3, 0.5, 0.7, 1, 2 and 3 g/kg of filter cake and wheat treated with 0, 0.2, 0.3, 0.5, 0.7, 1, 2 and 3 g/kg of filter cake, separately. Another 100 eggs were transferred to each jar containing maize treated with 0, 1, 2, 3, 6 and 8 g/kg of Triplex and wheat treated with 0, 0.2, 0.3, 0.5, 0.7, 1, 2 and 3 g/kg of Triplex. All the jars were held for 21 d in an environmental chamber at 28°C and 65 % r.h to determine number of live larvae that developed from eggs.

A separate experiment with the same species-grain-powder-concentration combinations as explained above was set up, and held in an environmental chamber at 28°C and 65% r.h for 42 d to determine adult progeny production of *T. castaneum* and *O. surinamensis*, and number of adults that emerged from *P. interpunctella* eggs. The adult progeny produced was counted from each jar after subtracting the original 20 added adults of *T. castaneum* and *O. surinamensis*. Number of adults of *P. interpunctella* that emerged from each jar was counted after freezing all the jars at -13°C for 12 h.

7.3.4. Hatchability of P. interpunctella eggs

One hundred eggs of *P. interpunctella* were transferred to 25 mm diameter and 10 mm high glass Petri dishes, and held at 28°C and 65% r.h in an environmental chamber for 5 d to determine egg hatchability. A total of six replications were used for the hatchability test. Number of eggs that hatched were counted based on empty egg shells. Egg hatchability was expressed as percentage of eggs that hatched out of total eggs. The mean ± SE percentage hatchability of *P. interpunctella* eggs was 95.0 ± 3.5%.

7.3.5. Data analysis

The number of adults that died at 14 d at each concentration was reported as a percentage. Mortality of *T. castaneum* and *O. surinamensis* adults on untreated maize and wheat was 0%. The percentage mortality data were transformed to angular values to normalize heteroscedastic treatment variances (Zar, 1984). The adult progeny production data were transformed to $\log_{10}(x+1)$ scale (Bartlett, 1947), to normalize heteroscedastic treatment variances. Number of *P. interpunctella* live larvae at 21 d and number of adults that emerged at 42 d data were transformed to angular values (Zar, 1984). However, untransformed data are presented in tables for all the three species. Percentage mortality, adult progeny production, number of live larva and number of adult emergence data were subjected to three-way ANOVA to determine significant differences ($P < 0.05$) among the main (concentration, species, and grain), and interactive effects (SAS Institute, 2012). Data showing significant differences on any of the main effects were subjected to one-way ANOVA to determine significant differences ($P < 0.05$) among levels on each main effect, and means were separated by Ryan-Einot-Gabriel-Welsch multiple range test (REGWQ) (SAS Institute, 2012).

7.4. Results

7.4.1. Mortality responses of T. castaneum and O. surinamensis adults to powders

The mean ± SE percentage mortality of *T. castaneum* adults on maize treated with filter cake and Triplex ranged from 1.7 ± 1.7 to 100 and 0 to 61.7 ± 6.0%, respectively (Table 7.1). The corresponding mortality of *T. castaneum* adults on wheat treated with filter cake and Triplex ranged from 1.7 ± 1.7 to 100 and 1.7 ± 1.7 to 78.3 ± 4.4%, respectively. Complete mortality of *T. castaneum* adults was achieved when adults were exposed to both maize and wheat treated with 2–3 g/kg of filter cake; however, complete mortality was not achieved at all Triplex concentrations on maize and wheat.

The mean ± SE percentage mortality of *O. surinamensis* adults on maize treated with filter cake and Triplex ranged from 13.3 ± 4.4 to 100 and 0 to 88.3 ± 9.3%, respectively. The corresponding mortality of *O. surinamensis* on wheat treated with filter cake and Triplex ranged from 11.7 ± 3.3 to 100 and 3.3 3.3 to100%, respectively. Complete mortality of *O. surinamensis* was achieved on maize and wheat treated with 1–3 g/kg of filter cake and 2–3 g/kg of Triplex only on wheat. Complete mortality was not achieved at all Triplex concentrations on maize.

Three–way ANOVA showed that the mortality due to the main effects of concentration (F = 24.38, df = 8, 161; $P < 0.0001$), grain (F = 12.60, df = 1, 161; P = 0.0005), and species (F =

36.52, df = 1, 161; *P* < 0.0001) were significant. The interaction of concentration and grain (*F* = 0.54; df = 8, 161; *P* = 0.8263), concentration and species (*F* = 0.77; df = 5, 161; *P* = 0.5759), grain and species (*F* = 0.17; df = 1, 161; *P* = 0.6804), and concentration, grain, and species (*F* = 0.32; df = 5, 161; *P* = 0.8990) were not significant.

On maize, one–way ANOVA showed significant differences in mortality of T. *castaneum* among concentrations of filter cake (*F* = 87.92; df = 5, 17; *P* < 0.0001) and Triplex (*F* = 38.75; df = 8, 26; *P* < 0.0001). Significant differences among concentrations were observed on wheat treated with filter cake (*F* = 59.24; df = 5, 17; *P* < 0.0001) and Triplex (*F* = 44.15; df = 8, 26; *P* < 0.0001). Significantly greater mortality of *T. castaneum* was observed on wheat at 0.7 g/kg of filter cake concentration compared to mortality at the same concentration on maize (*F* = 24.92; df = 1, 5; *P* = 0.0075). However, no significant difference in *T. castaneum* adult mortality was observed between maize and wheat treated with the remaining concentrations of filter cake (*F*, range = 0.01–0.57; df = 1, 5; *P*, range = 0.0.4918–0.9999) (Table 7.1). Similarly, significantly greater mortality of *T. castaneum* adults was observed on wheat treated with Triplex concentrations of 1–8 g/kg compared to mortality at same concentrations on maize (*F*, range = 13.45–109.20; df = 1, 5; *P*, range = 0.0005–0.0214) (Table 7.2). The remaining Triplex concentrations produced similar mortalities of *T. castaneum* on maize and wheat (*F*, range = 0.01–5.20; df = 1, 5; *P*, range = 0.0847–0.9999).

On maize, one-way ANOVA showed significant differences in mortality of *O. surinamensis* adults among concentrations of filter cake (*F* = 61.53; df = 5, 17; *P* < 0.0001) and Triplex (*F* = 32.43; df= 5, 17; *P* < 0.0001). On wheat, one-way ANOVA showed significant differences in *O. surinamensis* adult mortality among concentrations of filter cake (*F* = 147.38; df= 5, 17; *P* < 0.0001) and Triplex (*F* = 35.74; df = 5, 17; *P* < 0.0001). Mortality of *O. surinamensis* adults were significantly greater on wheat treated with 0.5, 0.7, 1 and 2 g/kg of Triplex compared to mortality at similar concentrations on maize (*F*, range = 7.86–153.33; df = 1, 5; *P*, range = 0.0002 – 0.0486) (Table 7.2). However, no significant differences in mortality of *O. surinamensis* adults was observed between maize and wheat treated with filter cake (*F*, range = 0.06–1.16; df = 1, 5; *P*, range = 0.3413–0.8215) (Table 7.1).

On maize, significantly greater mortality of *O. surinamensis* adults were observed at filter cake concentrations of 0.3, 0.5 and 0.7 g/kg (*F*, range = 7.71–21.61; df = 1, 5; *P*, range = 0.0097–0.0500) and Triplex concentrations of 0.7, 1, 2 and 3 g/kg (*F*, range = 14.04–40.75; df = 1, 5; *P*, range = 0.0031–0.0200) compared to mortality *T. castaneum* adults at the same concentrations. On wheat, mortality of *O. surinamensis* adults was significantly greater at filter cake concentration of 0.3 g/kg (*F* = 7.86; df = 1, 5; *P* = 0.0486) and Triplex concentrations of 0.5, 0.7, 1, 2 and 3 g/kg (*F*, range = 7.86–407.07; df = 1, 5; *P*, range < 0.000–0.0486) compared to mortality of *T. castaneum* adults at the same concentrations.

7.4.2. Adult progeny production at 42 d

The mean ± SE number of *T. castaneum* adult progeny production after 42 d of exposure to maize treated with filter cake ranged from 0 to 21.0 ± 1.7 adults per jar and production in Triplex treatments it ranged from 0 to 32.7 ± 10.5 adults per jar. More adult progeny production was observed at lower than high concentrations of both powders. On maize, *T. castaneum* adult progeny production was not observed when adults were exposed to 0.7–3 g/kg of filter cake and 1–3 g/kg of Triplex. The mean ± SE number of *T. castaneum* adult progeny produced after 42 d of exposure to wheat treated with filter cake ranged from 0 to 48.7 ± 9.4 adults per jar and production in Triplex treatments it ranged from 0 to 51.7 ± 6.0 adults per jar. On wheat, *T.*

castaneum adult progeny production was not observed when adults were exposed to 1–3 g/kg of filter cake and 2–3 g/kg of Triplex.

The mean ± SE number of *O. surinamensis* adult progeny production after 42 d of exposure to maize treated with filter cake ranged from 0 to 261.7 ± 42.0 adults per jar and in Triplex treatments it ranged from 0 to 260.3 ± 7.4 adults per jar. More adult progeny production was observed at lower than high concentrations of both powders. On maize, *O. surinamensis* adult progeny production was not observed when adults were exposed to 1–3 g/kg of filter cake and 2–3 g/kg of Triplex. The mean ± SE number of *O. surinamensis* adult progeny production after 42 d of exposure to wheat treated with filter cake ranged from 0 to 296.7 ± 62.7 adults per jar and in Triplex treatments it ranged from 1.3 ± 0.9 to 265.7 ± 3.2 adults per jar.

On wheat, adult *O. surinamensis* progeny production was not observed when adults were exposed to 0.7–3 g/kg of filter cake. However, complete suppression of *O. surinamensis* adult progeny production was not achieved in all Triplex concentrations.

Three–way ANOVA showed that the progeny production was significantly different among concentrations (F = 65.04, df = 6, 167; P < 0.0001) and species (F = 83.96, df = 1, 167; P < 0.0001), whereas no significant differences observed between maize and wheat (F = 0.60; df = 1, 167; P = 0.4385). Concentration and species was the only significant interaction (F = 4.83; df = 6, 167; P = 0.0002). The interaction of concentration and grain (F = 0.59; df = 6, 167; P = 0.7363), grain and species (F = 0.44; df = 1, 167; P = 0.5064), and concentration, grain, and species (F = 1.81; df = 6, 167; P = 0.1012) were not significant.

On maize, one–way ANOVA showed significant differences in *T. castaneum* adult progeny production among concentrations of filter cake (F = 91.89; df= 6, 20; P < 0.0001) and Triplex (F = 11.55; df= 6, 20; P < 0.0001). On wheat, one-way ANOVA showed significant differences among concentrations of filter cake (*F* = 10.71; df = 6, 20; *P* = 0.0002) and Triplex (*F* = 50.83; df = 6, 20; *P* < 0.0001).

On maize, one–way ANOVA showed significant differences in *O. surinamensis* adult progeny production among concentrations of filter cake (*F* = 137.52; df= 6, 20 ; *P* < 0.0001) (Table 7.3) and Triplex (*F* = 192.94; df= 6, 20; *P* < 0.0001) (Table 7.4). On wheat, one-way ANOVA showed significant differences in *O. surinamensis* adult progeny production among concentrations of filter cake (*F* = 109.93; df = 6, 20; *P* = 0.0001) and Triplex (*F* = 82.04; df = 6, 20; *P* < 0.0001).

On maize, *O. surinamensis* produced significantly greater number of adult progeny at filter cake concentrations of 0, 0.3 and 0.5 g/kg (*F*, range = 47.23–161.81; df = 1, 5; *P*, range < 0.0001–0.0023) and Triplex concentrations of 0.3–0.7 g/kg (*F*, range = 39.93–390.55; df = 1, 5; *P*, range < 0.0001–0.0032) compared to adult progeny of *T. castaneum* at the same concentrations. On wheat, no significant difference in adult progeny production was observed between *T. castaneum* and *O. surinamensis* at all filter cake concentrations (*F*, range = 0.01–4.82; df = 1, 5; *P*, range = 0.0931–0.3744). However, on wheat, *O. surinamensis* produced significantly more adult progeny at Triplex concentrations of 0.3, 0.5, 0.7, 1 and 2 g/kg (*F*, range = 39.67–334.12; df = 1, 5; *P*, range < 0.0001 – 0.0032) compared to *T. castaneum*.

7.4.3. Live larvae at 21 d and adult emergence at 42 d of P. interpunctella

Mean ± SE percentage of *P. interpunctella* live larvae at 21 d on maize were 59.0 ± 6.4 and 54.7 ± 7.3% for filter cake and Triplex control treatments, respectively. The corresponding mean ± SE percentage of *P. interpunctella* live larvae at 21 d on wheat was 65.3 ± 0.7 and 63.0 ± 8.1% for filter cake and Triplex control treatments, respectively. Mean ± SE percentage of *P.*

interpunctella live larvae after exposure to filter cake and Triplex treated maize ranged from 0 to 18.0 ± 2.5% and 0 to 24.7 ± 1.8%, respectively. The corresponding percentage of live larvae on filter cake and Triplex treated wheat ranged from 0 to 27.7 ± 1.2% and 0 to 53.0 ± 6.1%, respectively. *P. interpunctella* live larvae at 21 d were not observed after exposure to 2–3 and 0.5–3 g/kg filter treated maize and wheat, respectively. Complete suppression of *P. interpunctella* live larvae was achieved at Triplex concentrations of 6–8 and 2–3 g/kg on maize and wheat, respectively. From all the jars, 21.6% of the total live larvae at 21 d were in the pupal stage. However, we considered both stages as live larvae for three and one–way ANOVA.

Mean ± SE percentage of *P. interpunctella* adults that emerged on maize were 77.3 ± 10.0 and 71.0 ± 4.9% for filter cake and Triplex control treatments, respectively. The corresponding mean ± SE percentage of *P. interpunctella* of adults that emerged on wheat were 62.7 ± 2.2 and 59.7 ± 4.3% for filter cake and Triplex control treatments, respectively. Mean ± SE percentage of *P. interpunctella* adults that emerged on maize treated with filter cake and Triplex ranged from 0 to 18.3 ± 5.0% and 0 to 11.0 ± 6.1%, respectively. The corresponding mean ± SE percentage of *P. interpunctella* adults that emerged on wheat treated with filter cake and Triplex ranged from 0 to 31.3 ± 3.4% and 0 to 47.0 ± 4.4%, respectively. No adults emerged on maize and wheat treated with 1–3 and 0.5–3 g/kg of filter cake, respectively. Complete suppression of adult emergence was achieved at Triplex concentrations of 6–8 and 3 g/kg on maize and wheat, respectively.

Three–way ANOVA showed significant differences in live larvae at 21 d and adult emergence at 42 d among concentrations (F = 45.47, df = 9, 167; P < 0.0001), whereas the remaining main effects (F, range = 0.02–3.17; df = 1, 167; P, range = 0.0772–0.8999) and all two way interactive effects (concentration, F = ; df = P = ;; grain, F = ; df = ; P = ; days, F = ; df

= ; P = ; and concentration, grain, and days, F = ; df = ; P =) were not significant. On maize, one–way ANOVA showed significant differences in percentage of *P. interpunctella* live larvae among concentrations of filter cake (*F* = 76.68; df= 6, 20 ; *P* < 0.0001) (Table 7.5) and Triplex (*F* = 94.15; df= 5, 17; *P* < 0.0001) (Table 7.6). On wheat, one-way ANOVA showed significant differences in percentage of *P. interpunctella* live larvae at 21 d among concentrations of filter cake (*F* = 934.28; df = 7, 23; *P* < 0.0001) and Triplex (*F* = 64.37; df = 6, 20; *P* < 0.0001).

On maize, one-way ANOVA showed significant differences in percentage of *P. interpunctella* adults that emerged among concentrations of filter cake (*F* = 43.13; df = 6, 20; *P* < 0.0001) and Triplex (*F* = 54.77; df = 5, 17; *P* < 0.0001). On wheat, one-way ANOVA showed significant differences in percentage of *P. interpunctella* in adults emerged among concentrations of filter cake (*F* = 61.49; df = 7, 23; *P* < 0.0001) and Triplex (*F* = 32.05; df = 6, 20; *P* < 0.0001). Filter cake was highly efficacious against *P. interpunctella* in suppressing live larvae and adult emergence.

7.5. Discussion

The toxicity of silica based powders against insects depends on particle accumulation from the grain kernel to insect's body and adsorption of lipid by powder particles from the insect cuticle (Le Patourel et al., 1989; Filipović et al., 2010). Insect behavior such as mobility (Malia et al., 2016) and concentration of the powder applied to grains (Le Patourel et al., 1989) are critical for the amount of particles to be picked up by insect's body. The efficacy of filter cake and Triplex varied among concentration and between the two grain types based on responses of *T. castaneum*, *O. surinamensis* and *P. interpunctella*. Filter cake and Triplex have higher atomic percentage of silicon and oxygen in the form of silicon dioxide (Tadesse and Subramanyam, 2018a), and therefore, we hypothesize the mode of action of these powders to be similar to that of other silica-based inert dusts.

Due to the smaller proportion of silicon and oxygen as silicon dioxide in both powders compared to diatomaceous earth powders, our result cannot be compared directly with the work done on diatomaceous earth (DE) powders by numerous researchers (Arthur, 2001, 2002; Arnaud et al., 2005; Athanassiou et al., 2003, 2008; Kljajić et al., 2010; Nukenine et al., 2010; Doumbia et al., 2014; Jean et al., 2015; Fazlabad et al., 2017). However, efficacy of filter cake and Triplex against all the species tested tend to increase with increasing concentration on maize and wheat. Similar phenomena were reported by several researchers with DE powders evaluated against *T. castaneum* (Fazlabad et al., 2017), *O. surinamensis* (Arthur, 2001) and *P. interpunctella* (Mewis and Ulrichs, 2001).

Several researchers suggested the toxicity of DE was affected by type of grain (Athanassiou et al., 2003; Athanassiou and Kavallieratos, 2005; Kavallieratos et al., 2005; Athanassiou et al., 2008; Vassilakos et al., 2015; Fazlabad et al., 2017). Our study demonstrated that the mortality of *T. castaneum* and *O. surinamensis* adults was significantly greater on filter cake and Triplex treated wheat compared to maize. Vayias et al. (2006) reported that the high lipid content of maize kernels may cause an increased oil adsorption by DE particles, and thus the DE may become less effective in adsorbing lipids from the insect cuticle. This may be the plausible reason for the relatively lower efficacy of filter cake and Triplex on maize compared to wheat. The mortality of *T. castaneum* adults did not exceed 37 and 66% after a 14 d exposure to 1.5 g/kg of Paya®, a DE formulation, on maize and rice, respectively, whereas mortality was 100% at same concentration of Paya® on wheat (Fazlabad et al., 2017). On both maize and wheat, 100% mortality of *T. castaneum* and *O. surinamensis* adults was achieved at 2–3 and 1–3 g/kg of filter cake, respectively. However, complete mortality of *T. castaneum* was not achieved at all concentrations of Triplex on maize and wheat. *T. castaneum* was reported as one of the least susceptible stored-product pests to DE (Korunic, 1998; Fields and Korunic, 2000; Arnaud et al., 2005). Fields and Korunic (2000) reported less DE attachment to its cuticle than *S. oryzae* and *R. dominica* on their scanning electron micrographs analysis of the ventral abdominal surfaces.

Complete suppression of adult progeny production of *T. castaneum* was achieved when adults were exposed to 0.7–3 g/kg of filter cake and 1–3 g/kg of Triplex treated maize and wheat. Similarly, 100% suppression of *O. surinamensis* adult progeny production was achieved at 1–3 and 0.7–3 g/kg of filter cake treated maize and wheat, respectively. *O. surinamensis* produced significantly greater adult progeny compared to *T. castaneum* in both filter cake and Triplex treatments. On maize, complete suppression of *O. surinamensis* adult progeny production was achieved at 2–3 g/kg of Triplex. However, suppression of *O. surinamensis* adult progeny production at the same concentrations of Triplex on wheat was 98.5-99.6%. This is surprising considering the fact that filter cake is more efficacious against adults than Triplex. These anomalous results could be due to using unsexed adults of mixed ages in bioassays.

Adult progeny production tended to decrease with increasing filter cake and Triplex concentrations on both maize and wheat. An increase in mortality of *T. castaneum* and *O. surinamensis* adults with increasing concentration of filter cake and Triplex may have caused significant reduction in adult progeny production at higher concentrations. We observed dead larval stages of both species when counting adult progeny. This indicated that larvae did not complete their development to adulthood. Subramanyam et al. (1998) reported 96–97 and 86–97% mortality of *T. castaneum* and *O. surinamensis* first instars, respectively, after exposure to 0.5 and 1 g/kg of Insecto® treated maize. They also reported that adult emergence was inversely proportional to powder concentration. A similar phenomenon was reported by Tadesse and Subramanyam (2018b) after a 42 d exposure of *S. zeamais* and *S. oryzae* adults to filter cake and Triplex treated wheat. Tadesse et al. (2018a,b) also reported a decreasing trend of *S. zeamais* and *S. oryzae* adult progeny production with increased concentration of filter cake and Triplex on concrete surfaces.

The survival of *P. interpunctella* in storage commodities depends on larval survival (Kaliyan et al., 2005). Live larvae of *P. interpunctella* at 21 d were not observed at filter cake concentrations of 2–3 and 0.5–3 g/kg on maize and wheat, respectively. Similarly, at 42 d, *P. interpunctella* adult emergence was not observed at filter cake concentrations of 1–3 and 0.5–3 g/kg on maize and wheat, respectively. Different stages of *P. interpunctella* have different sensitivity to insecticides. For example, Mewis and Ulriches (2001) reported that first instars of *P. interpunctella* died after a 1 d exposure to the DE Fossil shield® treated wheat, however, no treatment effect was observed on the third and fourth instars. Subramanyam et al. (1998) reported that less adult emergence of *P. interpunctella* was observed from first and third instars compared to fifth instars exposed to Insecto®-treated maize. The high surface area to volume ratio in the earlier stages explain their sensitivity to dusts, manifested by water loss through cuticle. The chemical composition of the cuticle depends on the developmental stage and could influence the effect of powders against insects (Mewis and Ulriches, 2001).

Under laboratory conditions, *T. castaneum*, *O. surinamensis,* and *P. interpunctella* were highly susceptible to filter cake. *O. surinamensis* was less susceptible to Triplex compared to *T. castaneum* and *P. interpunctella* on both maize and wheat. Admixing of powder to maize and wheat was done manually, where uneven distribution of powder over the maize and wheat kernel surfaces may have occurred. This might have contributed to unexplained variation in mortality and progeny production responses of the tested species.

New packaging technologies such as Purdue Improved Crop Storage (PICS) bags, GrainPro Super bags, plastic drums, and metal silos were introduced to Ethiopia (Obeng-Ofori, 2011; Murdock and Baoua, 2014; Chigoverah and Mvumi, 2016) to protect smallholder farmers' stored commodities. Using filter cake and Triplex in combination with these technologies will result in better protection of stored grains from insect pest infestations. The traditional storages in Ethiopia, such as *gota* and *gotera* are made with *teff* straw, mud, and cow dung (Abraham et al., 2008), which may influence the efficacy of filter cake and Triplex against stored product insects. Therefore, further research should evaluate the efficacy of applying filter cake or Triplex to maize and wheat under smallholders' traditional storage conditions in Ethiopia.

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Table 7.1. Mortality of *T. castaneum* and *O. surinamensis* adults after a 14 d exposure to filter cake treated maize and wheat.

Concentration (g/kg)	Corrected adult mortality (% mean ± SE)[a,b]			
	T. castaneum		*O. surinamensis*	
	Maize	Wheat	Maize	Wheat
0.3	1.7 ± 1.7C	1.7 ± 1.7D	13.3 ± 4.4C	11.7 ± 3.3D
0.5	10.0 ± 2.9BC	16.7 ± 7.3C	56.7 ± 10.1B	43.3 ± 7.3C
0.7	18.3 ± 7.3B,b	68.3 ± 3.3B,a	63.3 ± 8.8B	73.3 ± 4.4B
1	93.3 ± 4.4A	93.3 ± 6.7A	100.0 ± 0.0A	100.0 ± 0.0A
2	100.0 ± 0.0A	100.0 ± 0.0A	100.0 ± 0.0A	100.0 ± 0.0A
3	100.0 ± 0.0A	100.0 ± 0.0A	100.0 ± 0.0A	100.0 ± 0.0A

[a] Means within a commodity followed by different upper letters were significantly different (*F*, range = 59.24–147.38; df = 5, 17; $P < 0.0001$; one-way ANOVA with means were separated by REGWQ multiple range test)

[b] Means between maize and wheat by species followed by different lower letters were significantly different ($F = 24.92$; df = 1, 5; $P = 0.0075$; one-way ANOVA with means were separated by REGWQ multiple range test).

Table 7.2. Mortality of *T. castaneum* and *O. surinamensis* adults after a 14 d exposure to Triplex treated maize and wheat.

Concentration (g/kg)	Corrected adult mortality (% mean ± SE)[a,b]			
	T. castaneum		*O. surinamensis*	
	Maize	Wheat	Maize	Wheat
0.3	0.0 ± 0.0C	1.7 ± 1.7D	0.0 ± 0.0C	3.3 ± 3.3B
0.5	0.0 ± 0.0C	1.7 ± 1.7D	1.7 ± 1.7BC,b	11.7 ± 3.3B,a
0.7	1.7 ± 1.7C	1.7 ± 1.7D	11.7 ± 3.3BC,b	80.0 ± 15.3A,a
1	1.7 ± 1.7C	15.0 ± 2.9C	43.3 ± 7.3B,b	98.3 ± 1.7A,a
2	3.3 ± 1.7C,b	36.7 ± 4.4BC,a	78.3 ± 10.1A	100.0 ± 0.0A
3	5.0 ± 0.0C,b	55.0 ± 5.8AB,a	88.3 ± 9.3A	100.0 ± 0.0A
6	25.0 ± 5.8B,b	61.6 ± 7.3AB,a	–[c]	–
8	48.3 ± 4.4A,b	76.7 ± 6.0A,a	–	–
10	61.7 ± 6.0A	78.3 ± 4.4A	–	–

[a] Means within a commodity by species followed by different upper letters were significantly different (*F*, range = 32.43 – 44.15; df = 8, 26 for *T. castaneum*, and df = 5, 17 for *O. surinamensis*; $P < 0.0001$, one-way ANOVA with means were separated by REGWQ multiple range test).

[b] Means between maize and wheat by species followed by different lower letters were significantly different (*F*, range = 7.86–153.33; df = 1, 5; *P*, range = 0.0002–0.0486; one-way ANOVA with means were separated by REGWQ multiple range test).

[c] These concentrations were not tested.

Table 7.3. Adult progeny production of *T. castaneum* and *O. surinamensis* after a 42 d exposure to filter cake treated maize and wheat.

Concentration (g/kg)	Progeny production (mean ± SE)[a,b]			
	Maize		Wheat	
	T. castaneum	*O. surinamensis*	*T. castaneum*	*O. surinamensis*
0	21.0 ± 1.7A,b	261.7 ± 42.0A,a	48.7 ± 9.4A,b	296.7 ± 62.7A,a
0.3	11.7 ± 2.2B,b	54.7 ± 3.9B,a	7.6 ± 5.4B	42.0 ± 15.9B
0.5	1.0 ± 0.6C,b	28.3 ± 7.4B,a	6.7 ± 3.5B	1.3 ± 0.9C
0.7	0.0 ± 0.0C	1.7 ± 0.9C	0.0 ± 0.0B	0.0 ± 0.0C
1	0.0 ± 0.0C	0.0 ± 0.0C	0.0 ± 0.0B	0.0 ± 0.0C
2	0.0 ± 0.0C	0.0 ± 0.0C	0.0 ± 0.0B	0.0 ± 0.0C
3	0.0 ± 0.0C	0.0 ± 0.0C	0.0 ± 0.0B	0.0 ± 0.0C

[a] Means within a commodity by species followed by different upper letters were significantly different (*F*, range = 10.71–137.52; df = 6, 20; *P*, range < 0.0001–0.0002; one-way ANOVA with means were separated by REGWQ multiple range test).

[b] Means within a commodity among species followed by different lower letters were significantly different (*F*, range = 44.72–161.81; df = 1, 5; *P*, range = 0.0002–0.0026; one-way ANOVA with means were separated by REGWQ multiple range test).

Table 7.4. Adult progeny production of *T. castaneum* and *O. surinamensis* after a 42 d exposure to Triplex treated maize and wheat.

Concentration (g/kg)	Progeny production (mean ± SE)[a,b]			
	Maize		Wheat	
	T. castaneum	*O. surinamensis*	*T. castaneum*	*O. surinamensis*
0	32.7 ± 10.5A,b	260.3 ± 7.4A,a	51.7 ± 6.0A,b	265.7 ± 3.2A,a
0.3	9.0 ± 1.5AB,b	244.7 ± 18.2A,a	16.7 ± 3.4B,b	256.7 ± 5.2A,a
0.5	3.7 ± 2.7BC,b	217.3 ± 21.4A,a	7.7 ± 1.4B,b	219.7 ± 2.9A,a
0.7	2.0 ± 2.0BC,b	174.3 ± 28.7A,a	2.3 ± 1.2C,b	89.0 ± 2.5B,a
1	0.0 ± 0.0C	2.0 ± 1.2B	0.0 ± 0.0C,b	26.0 ± 7.5C,a
2	0.0 ± 0.0C	0.0 ± 0.0C	0.0 ± 0.0C,b	4.0 ± 1.2D,a
3	0.0 ± 0.0C	0.0 ± 0.0C	0.0 ± 0.0C	1.3 ± 0.9D

[a] Means within a commodity by species followed by different upper letters were significantly different (*F*, range = 11.55–192.94; df = 6, 20; $P < 0.0001$; one-way ANOVA with means were separated by REGWQ multiple range test).

[b] Means within a commodity among species followed by different lower letters were significantly different (*F*, range = 28.70 – 390.55; df = 1, 5; $P < 0.0001 – 0.0059$; one-way ANOVA with means were separated by REGWQ multiple range test).

Table 7.5.Live larvae and adult emergence of *P. interpunctella* adults after a 21 and 42 d exposure to filter cake treated maize and wheat.

Concentration (g/kg)	Mean ± SE **number of**			
	Live larvae at 21 d		Adults at 42 d	
	Maize	Wheat	Maize	Wheat
0	59.0 ± 6.4a	65.3 ± 0.7a	77.3 ± 10.0a	62.7 ± 2.2a
0.2	–[b]	27.7 ± 1.2b	−	31.3 ± 3.4b
0.3	18.0 ± 2.5b	15.7 ± 2.0c	18.3 ± 5.0b	12.3 ± 7.8c
0.5	3.0 ± 0.6c	0.0 ± 0.0d	3.3 ± 1.5c	0.0 ± 0.0d
0.7	2.3 ± 0.9c	0.0 ± 0.0d	1.3 ± 0.3c	0.0 ± 0.0d
1	1.7 ± 0.9c	0.0 ± 0.0d	0.0 ± 0.0c	0.0 ± 0.0d
2	0.0 ± 0.0c	0.0 ± 0.0d	0.0 ± 0.0c	0.0 ± 0.0d
3	0.0 ± 0.0c	0.0 ± 0.0d	0.0 ± 0.0c	0.0 ± 0.0d

[a] Means within a commodity followed by different letters were significantly different (*F*, range = 43.13–934.28; df = 6, 20 for maize and 7, 23 for wheat; $P < 0.0001$; one-way ANOVA with means were separated by REGWQ multiple range test)

[b] These concentrations were not tested.

Table 7.6. Number of live larvae at 21 d and adults of *P. interpunctella* at 42 d after exposure to Triplex treated maize and wheat.

Concentration (g/kg)	Mean ± SE number of:[a]			
	Live larvae at 21 d		Adults at 42 d	
	Maize	Wheat	Maize	Wheat
0	54.7 ± 7.3a	63.0 ± 8.1a	71.0 ± 4.9a	59.7 ± 4.3a
0.3	–[b]	53.0 ± 6.1ab	–	47.0 ± 4.4ab
0.5	–	40.7 ± 4.1b	–	41.0 ± 1.7abc
0.7	–	36.7 ± 2.9b	–	31.7 ± 2.7bc
1	24.7 ± 1.8b	19.0 ± 1.5c	11.0 ± 6.1b	22.0 ± 9.0c
2	2.0 ± 0.6c	0.0 ± 0.0d	6.0 ± 1.2bc	2.0 ± 0.6d
3	0.7 ± 0.3c	0.0 ± 0.0d	1.7 ± 0.9bc	0.0 ± 0.0d
6	0.0 ± 0.0c	–	0.0 ± 0.0c	–
8	0.0 ± 0.0c	–	0.0 ± 0.0c	–

[a] Means within a commodity followed by different letters were significantly different (*F*, range = 32.05–94.5; df = 5, 17 for maize and df = 6, 20 for wheat; $P < 0.0001$; one-way ANOVA with means were separated by REGWQ multiple range test).

[b] These concentrations were not tested.

Chapter 8 - Future work

Our studies demonstrated on the effectiveness of both filter cake and Triplex powders against the tested insect species under laboratory conditions. However, traditional storages in Ethiopia, such as *gota* and *gotera* are made with teff straw, mud, and cow dung (Abraham et al., 2008). Therefore, studies under the real Ethiopian field conditions should be conducted in smallholder farmer's traditional storages, which include above-ground structures like *gota* and *gotera*, and underground pits (Tadesse and Eticha 2000, Blum and Bekele 2001, Dessalegn et al. 2017). In addition to this, how the powders affect the grain physical and bulk density properties at effective concentrations should be investigated. Persistence, settling effect, and removal during processing are important factors that should be studied especially on maize and wheat.

According to the European parliament and council directive No. 95/2/EC (1995), silicon dioxide is an approved food additive and can be used as an anti-caking agent in dry powdered foodstuffs with a maximum level of 10 g/kg. In addition, it is approved for use in plastic material coming into contact with food, without hazards to public health. The US Food and Drug Administration (FDA) has classified silicon dioxide as Generally Recognized as Safe (GRAS) and has approved its use as a dietary food additive at levels of up to 2% by weight in food (21 CFR 172.480)
https://www.accessdata.fda.gov/scripts/cdrh/cfdocs/cfCFR/CFRSearch.cfm?fr=172.480.
Moreover, the expert group on vitamins and minerals of the UK food standards agency (2003) approved supplements that contain up to 500 mg of silicon. Our studies indicated that the concentrations used were not exceeded the recommended amounts by FDA, UK and EU. However, our study did not confirm the type of silicon dioxide found in both filter cake and Triplex.

Work place exposure to respirable crystalline silica produces silicosis, silicotuberclosis and also lung cancer (Goldsmith et al, 1997). It is only the amorphous form of silicon dioxide (synthetic amorphous silica (SAS)) authorized as a food additive by the European Union (Younes et al., 2018). Amorphous silica are also approved for stored product pest management. Our elemental analysis study confirmed only the absence of heavy metals in both powders. Collins and Marty (1997) reported that crystalline silica may be a cause for cancer. We did not confirm whether the silicon dioxide content of both powders is crystalline silica or amorphous. A confirmatory study is needed on both powders.

The food additive, silicon dioxide, is a material comprised of aggregated nano sized primary particles. These aggregates can further agglomerate to form larger structures depending on the size and moisture content. The sizes of the aggregates and agglomerates are normally greater than 100 nm (Younes et al., 2018). Subramanyam and Roesli (2000) reported that for use as insecticides, the particle size of silica containing products such as diatomaceous earth (DE) should be in the range of 1-50 µm with a mean particle size of 10 µm. Kurunic (1998) reported that DE should have pure amorphous silica having particles of equal diameter (< 10 µm) and less than 1% crystalline silica. However, our study on filter cake and Triplex used < 177 µm particle size. Therefore, knowing the particle size distribution and selecting the safest and effective uniform particle size of the two powders is important to be considered in future studies.

Studies in rats indicated no accumulation of silicon after repeated oral applications of the food additive silicon dioxide (Younes et al., 2018). The same report indicated that in humans, there was little indication of absorption of silicon dioxide after ingestion; however, silicon dioxide (of unknown origin) was occasionally found in human liver and spleen tissues. There was evidence for a low acute oral toxicity of food additive silicon dioxide and for low toxicity after repeated oral administration of silicon dioxide, no adverse effects were detected even at high dose levels up to 9,000 mg/kg body weight per day. Silicon dioxide as a food additive did not raise a concern with respect to genotoxicity. However, our research did not do any research on rats or other animals on the toxic effect of filter cake and Triplex. Therefore, studies on the effects of filter cake and Triplex in animal's liver, kidney and spleen, after oral administration is strongly recommended.

The main products of AMASSASC are aluminum sulfate and sulfuric acid. Aluminum sulfate is mainly used for the treatment of drinking water, cola, textile pulp, and paper production industries. Daniel (2006) reported that raw material inputs required for the production of aluminum sulfate are locally supplied except aluminum hydroxide, which has to be imported. Kaolin, a layered silicate mineral ($Al_2Si_2O_5(OH)_4$) is the main raw material and it is supplied by the Ethiopia Mineral resource Development Share Company (EMDSC) from Borena Zone of Oromia Region. Similarly, the inputs for the production of sulfuric acid are supplied from domestic sources except sulfur, which has to be imported. Thus, the project is partially based on local resources and it is expected to contribute considerably to the development and exploitation of local resources. However, we did not get access to the information on how the manufacturing processes are taken care of and how much by-product is produced per year. Therefore, establishing a collaborative work with AMASSASC will help researches to determine the potential amount of filter cake available to smallholder farmers, as it was consistently more efficacious than Triplex in my study.

I consistently observed on my study that Triplex was less effective against stored product insects compared to filter cake. Combing other silica based insecticides with Triplex may enhance the efficacy against insects. Similar to AMASSASC, we did not get access to the information on how much Triplex is produced per year from MIDROC soap factory. Therefore, establishing collaborative work with MIDROC soap factory is strongly recommended.

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