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

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ABSTRACT

Filter cake and Triplex are powdered by-products of aluminum sulfate and soap factories, respectively. The latter powder is from berries of the Endod plant, *Phytolacca dodecandra* L. This study was designed to determine the chemical composition of these two powders using scanning electron microscopy conjugated with energy-dispersive X-ray spectroscopy. Another aim was to evaluate their direct 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 °C and 65% r.h. for 7 d to determine mortality. Adult progeny production, percent of insect damaged kernels, and grain weight loss at each powder-concentration-time combinations were determined after 42 d. Both powders were free of heavy metals. Silicon and oxygen were the major components of both powders. 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. Reductions in adult progeny production, percent 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*.

1. 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 20–30% (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 such products are filter cake and Triplex. Filter cake is a by-product of aluminum sulfate factory (Awash Melkassa Aluminium Sulfate & Sulfuric 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 genotypes of maize with 1, 2.5, and 5% (w/w) of filter cake and reported 92% mortality of the maize weevil, *Sitophilus zeamais* Motschulsky, after exposing adults for 3 d on treated maize. Admixing Triplex with

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maize at a concentration of 0.25% (w/w) showed 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 maize in farmers' 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 organisms other than stored-product insects, 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, (Girma et al., 2008a,b; Abraham et al., 2008; Tefera et al., 2011), under laboratory conditions.

2. Materials and methods

2.1. 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° (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 energy-dispersive X-ray 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). Each powder was mounted on a scanning electron microscope stub using double-sided adhesive 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. A total of six samples was evaluated for each powder.

2.2. Concrete-poured Petri dishes

Rockite®, a ready-to-mix concrete product (Hartline Products Co., Inc., Cleveland, Ohio, USA), was mixed with tap water in 2:1 ratio (grams to milliliter) 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. Application of powders to concrete arenas and insect exposure

Concrete arenas of 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 at 28 °C and 65% r.h. on organic yellow maize (Heartland Mills, Marienthal, Kansas, USA) of 13.5% moisture content (wet basis). Adults of *S. zeamais* 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 ($n = 1440$) 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. In total, there were 120 arenas. After introducing insects Petri dishes were covered with lids for the intended exposure time. After the intended exposure time period insects 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 (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 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. Adults 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. At 42 d, adult progeny produced was counted 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 sample 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. In each replicate control and treated working samples, percent weight loss was calculated using the following equation (Adams and Schulten, 1978; Boxall, 1986):

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

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.

2.4. Data analysis

Atomic percent of each element from each sample obtained by SEM and EDS was 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