

COMPARATIVE EFFICACY ANALYSIS OF INSECTICIDAL AND REPELLENCY PROPERTIES OF *HYMENOCARDIA ACIDA* WOOD ASH AND DIATOMACEOUS EARTH AGAINST *SITOPHILUS ZEAMAI*S (MAIZE WEEVIL)

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ABSTRACT

Stored product insect pests are problems throughout the world, as they diminish both the quantity and quality of grains. The resultant effect of this is an imminent threat to food security which might negatively impact our wellbeing. The maize weevil, *Sitophilus zeamais* is an insect pest which has spread all over the world, and causes damage to maize grains and other species of stored cereal. The aim of this study was to investigate the insecticidal efficacy and repellency properties of *H. acida* tree against the weevil on a 14 day exposure and trial. The study involved the use of treatment doses of 2g, 4g and 6g for *H. acida* and 0.5g, 1g, and 1.5g were used for diatomaceous earth as the Positive Control. Results showed that mortality was dose dependent, and there was a significant difference in mortality rates of the weevil across all treatments ($P < 0.05$). The treatment doses of HA ash significantly induced 94.17% mortality within the study period, and highest percentage progeny inhibition of 44.44% compared to DE with 27.78%. The study also recorded that HA ash showed the best efficacy in terms of grain damage (1.67%) and weight loss (0.92%). A highest repellency of 80% was observed in the treatment with 30% aqueous solution of *H. acida* ash than the control (70%). The study recommends that wood ash from *H. acida* could best be used as alternative method to reduce grain infestation and improve food security.

Keywords: Insecticidal, Repellency, Food Security, *Hymenocardia acida*, *Sitophilus zeamais*, Diatomaceous earth.

Introduction

The exponential growth of the global human population, projected to exceed 10 billion by 2050 (FAO, 2017), has intensified the need for food security worldwide. This challenge is particularly acute in developing region like Nigeria where post-harvest losses due to insect infestations significantly reduced the availability of stored grains and other staples. Among stored product pests, the maize weevil, *Sitophilus zeamais*, a cosmopolitan species, is particularly destructive, causing extensive damage to grains and other stored products (Pimatel et al., 2007). Infestations lead to both qualitative and quantitative losses, with significant economic implications.

The primary cause of the major issues with maize storage is insect infestations (Kebede et al., 2024). The maize weevil, *Sitophilus zeamais* Motschulsky (Coleoptera: Curculionidae), is a pest bug that has spread throughout the world and damages stored cereals, including maize grains. While an infestation may start in the field, most damage occurs in storage because both adults and larvae consume grains internally (Jean et al., 2015), Suleiman et al., 2015). Although losses are typically 4–5%, severe infestations of these pests in stored maize can result in weight losses of 15–30% in developing nations (Bergvinson, 2021). One of the most significant maize pests in Europe has been identified as *S. zeamais* (Trematerra et al., 1999), Asia (Peng, 1998) and USA (Sedlacek et al. 1998; Arbogast & Throne, 1997).

In the past, it was controlled with diclodiphenyltrichloroethane (DDT), malathion, pirimiphos-methyl, pyrethroids, and other chemicals (Corrúa et al., 2011), which often led to resistance and other negative effects (Jean et al., 2015). Research on alternative suppression techniques has focused on the use of various inert dusts, such as diatomaceous earth (Athanassiou et al., 2011) and various essential oils (Tofel et al., 2016). Inert dusts are any dry dusts that do not react chemically in the environment. However, limited data exists on *H. acidus* efficacy, dosage, and application against *Sitophilus zeamais*, making this study a pioneer in leveraging this plant for maize protection.

Liška et al. (2017) and Korunić (1998) divided them into a number of groups based on their level of activity, chemical composition, and physical makeup. These groups included non-silica dusts, groups that contained sand, kaolin, and wood ash, diatomaceous earths (or diatomite) primarily derived from diatomaceous earths and amorphous hydrated silica, with high-quality silica aerogels and synthetic silicates. These include wood ash and kaolin, which are commonly utilised locally (on small farms) and in less developed nations. According to Panagiotakopulu et al. (1995), the latter is among the oldest insecticides. Its significance has grown recently, especially in light of the issues surrounding synthetic pesticides (Guedes et al., 2017; Tofel et al., 2016).

According to a Slovenian study, common beech ash exerts a strong insecticidal effect on the granary weevil, *Sitophilus granarius* (L.) (Coleoptera: Curculionidae) (Bohinc & Tradan, 2017). The study found that lower relative humidity levels had an adverse effect on both progeny production and death following exposure intervals. Applying wood ash alone and expanding its use with other items showed the high efficacy of non-chemical methods (Marina et al., 2023). Akob and Ewete (2007) and Jean et al. (2015) have already shown how to apply wood ash alone.

The objective of this study is to compare the effectiveness of wood ash from the native tree species, *Hymenocardia acida* (Phyllanthaceae), and diatomaceous earth against *S. zeamais* which is underexplored for pest control, offering a potential breakthrough in using local Nigerian flora for eco-friendly biopesticides as a sustainable weevil management approach. The efficacy was assessed using the following metrics: adult mortality, offspring inhibition, population suppression of *S. zeamais*, and the weevil's damage reductions on the treated grains.

Materials and methods

Study Area:

The experimental study was conducted into two sites. *In vitro* insecticidal efficacy test was conducted in the Zoology Department, Federal University of Lafia Nasarawa State Nigeria, while the proximate analysis of wood ashes was conducted in the Chemistry Department Federal University of Lafia. The study was carried out between the months of July to September.

Lafia is Located within latitude 8.49⁰N and 8.52⁰E in Nasarawa State Lafia is bounded to the East by Nasarawa Egon and Kokona local government areas, on the West by Plateau State, to the North by Wamba Local government area and to the South by Doma and Obi local government areas. The climatic condition of lafia is typical of the Tropical Guinea Savanna Belt of North Central Nigeria with an annual rainfall that varies between 130cm to 140cm and an average annual temperature of 28.4⁰C (Figure 2.1).

Study design and procedures:

The study explored the *In vitro contact toxicity bioassay of the ash using the adult weevils as subjects* which was conducted between July and September 2024.

Collection and preparation of wood ash of *Hymenocardia acida* trees:

Branches of *H. acida* were collected respectively from Lafia town area. They were collected between July and September 2024. During collection, latex gloves was used in order to reduce contamination. Stems were air-dried under the sun for two weeks, and then burnt in a traditional kitchen and obtained a required weight of ash. The wood ash (250g) from plant species was packaged in glass jars and kept in a refrigerator (-4⁰c) for bioassay.

Diatomaceous earth: The DE used was fossil field 90.0 fine brownish powder containing amorphous silica particles with size range between 5 and 30 mm, a content of 73% amorphous SiO₂, a water content of about 2% in addition to 3% aerosol and other mineral compounds (Fossilfield company, Bein GmbH, Ei terfield, Germany).

Proximate analysis of wood ash collected from *Hymenocardia acida* trees and diatomaceous earth: The wood ash was collected from the trees and the analysis was done. Ash content, moisture content, volatile matter as well as the mineral composition (Ca, K and pH) were the major parameters assessed for the wood ash while percentage aerosol, water content and silica was also obtained for diatomaceous earth.

Insect culture and treatment of maize grains: The initial *S. zeamais* population was acquired from infected maize grains that were bought from Lafia Modern Market, and freshly harvested, uninfested maize varieties were acquired from the College of Agriculture, Lafia, and kept for three hours in ambient laboratory settings (20–29°C) before being allowed to cool for an additional hour. After being added to the uninfested (sterilised) maize grains and given time to oviposit, the adults of the original population of *S. zeamais* were taken out. A second jar of recently collected maize seeds was filled with the emerging adults (F1).

This is to guarantee that the F1 adults used as the experiment's culturing stock are of the same age (of 27 days) and size. According to Masuda et al. (2004), the insect culture was maintained at a temperature of 27 to 29 degrees Celsius and a relative humidity of 70 to 80%. All bioassays using maize as the substrate used live insects. To encourage the initial grain moisture content, the Shaba variety of maize was identified with the Department of Agronomy, Faculty of Agriculture, FULafia, and kept in a freezer at -4c for seven days.

Preparation of treatments: Each of the two (2) diatomaceous earth and wood ash replicates was weighed using 1-5% w/w (weight of ash per weight of grain) 2 g, 4 g, and 6 g of

wood ash from the samples, respectively. 50g of maize was placed in glass jars with 0.5g, 1g, and 1.5g of diatomaceous earth (Positive Control) added individually.

Adult mortality test and F1 progeny production: Group of 40 *S. zeamais* each for three replicates were separately added to glass first containing the treated maize. Control stand was also inoculated with 40 insects. All treatments were arranged in completely randomized design on shelf in the laboratory under ambient conditions (23.7+1.87°C, 59.0+7.63r.h.). Within 24 hours and on days 3, 7, and 14 following treatment, insect mortality was noted. To guarantee that the dusts were evenly distributed throughout the grain mass, each jar was shook by hand for five minutes. Diatomaceous earth was used as the positive control, and maize grains devoid of wood ash were used as the negative control (Nukenine et al., 2007).

Insect population and grain damage:

The dosages of wood ash and diatomaceous earth used in the previously stated toxicity test were considered for 150g grains. Forty (40) adult insects were placed in each jar of treated or untreated grains. Each procedure was performed three replicates. After being stored for two weeks, the number of live and dead insects in each jar was determined. Using Adams and Schullen's method, damage was assessed by counting and measuring the perforated and non-perforated grains as well as calculating the percentage of weight loss (Schullen et al., 1978) as follows:

$$\frac{(UNd)-(DNu)}{U(Nd+Nu)} \times 100$$

Where;

U= Weight of undamaged grains;

Nu= Number of undamaged grains;

D= Weight of damaged grains;

Nd= Number of damaged grains

Persistence of wood ash on maize grains:

Twenty (20) mature *S. zeamais* were exposed to the treated maize (same order as previously mentioned for bioassay above) which were stored for 7 days (Obeng-fovi et al., 2005) and the persistence of dust on grain was evaluated.

Repellency bioassay of the wood ash:

Weevil-infested maize seeds were placed in cotton wool soaked in aqueous solution of ashes with 20 live weevils per each replicate at different concentrations at 2-hour intervals for a 12-hour period for seven days (one day for each concentration) (Adesina, 2019) in three replicates for both the ash and the control. The percentage repellency activity of the aqueous solution of wood ash and diatomaceous earth was determined using a free choice (area preference) bioassay model, which was carried out in the demonstration cage, e.g. 25cmx 25cmx 10cm, because it is easily applicable and dependable (Gambari, 2021) thus:

- Percentage repellency (PR) = $(NC-NT) \div (NC+NT) \times 100\%$ ((Nerio et al. 2009)

NC= Number of insects in the non-treated (Control) after exposure interval

NT = Number of insects in treated area after exposure interval

- Concentration of aqueous ash solution was calculated as:

$$\frac{\text{Quantity of power (g)}}{1000 \text{ mls of water}} \times 100\%$$

Statistical Analysis:

The analysis of variance (ANOVA) approach was applied to the converted data using the statistical analysis system (Finney et al. 1979), which comprised the following: Abbott's Formula (Abbot et al., 1925) was used to adjust for control mortality before ANOVA, Tukey's test (P=0.05) was used for mean separation, and Probit analysis (Zar et al., 1999) was used to determine the

lethal content (LC) causing 50% (LC50) and 95% (LC95) mortality of *S. zeamais* following treatment application.

Results

Proximate analysis of the wood ash:

Plant species- *Hymenocardia acida* whose branches were gathered and burnt and the ash was subjected to proximate analysis as shown in Table 1. Ash content, moisture content, fixed carbon, pH, Ca, K and volatile matter were found in the tree as major parameters for the analysis. Moisture content, fixed content and volatile matter were found in high concentration, but ash content was low in concentration. The wood ash is highly alkaline (12.96) and contains significant amount of potassium (K) (15.70) and calcium (Ca) (24.02). The insecticidal efficacy of the ash is primarily attributed to its alkalinity and desiccative properties, which cause mortality to insects like maize weevil by physical action and disruption of their cuticles.

Proximate percentage composition of diatomaceous earth (Synthetic Fossil field 90.0):

The chemical composition of DE as indicated by the commercial label as shown in Table 1

Comparison of mortality efficacy of the wood ash against the weevil:

Both the two tested dusts generally caused significant dose-dependent mortality to adult *S. zeamais* (Table 2) compared to the control. Complete mortality (100%) of *S. zeamais* was achieved by DE at the doses of 0.5g, 1g, and 1.5g within 14 days of exposure. Maximum mortality caused to the weevil by the highest tested dose of wood ash (6g) was 100% of *H. acida* at 14th day of treatment.

Comparison of the larvicidal efficacy of the wood ash on F1 progeny suppression:

All the treatment groups caused significant reduction in progeny emergence relative to control, which was not dose-dependent (table 3.2). Diatomaceous earth suppressed F1 progeny emergence of (44.44%) at lowest dose level (0.5g). However, this decreased at highest content (1.5g) to 33.33%.

Comparison of the efficacy of the wood ash and DE on insect population, percentage reduction of grain damage and weight loss:

Both powders have significantly reduced grain damage from the *Zea mais* infestation, with treated samples recording a smaller number of damaged grains than the control (table 2). For example, at dose 1.5g, D.E as positive control averted weevil damage (0.33%). The samples treated with HA ash showed 1.67% grain damage, compared to 64.67% of the negative control, and correspondingly no weight was lost at the highest dose of HA with 0.94% reduction. At the tested dose of DE recorded a weight loss of 2.66% compared to 74.77% of the negative control (Table 2).

Comparison of the efficacy on persistence of wood ash on maize grains 7-days post-treatment

In general, the efficacy of D.E (Positive control) decreased 7 days post-treatment at the lowest dose (0.5g) with 96.67% mortality potency. Thus, dose appeared to influence insecticidal efficacy because at the highest tested dose rate, (1.5g), the potency of D.E remained constant (100%).

Lethal contents resulting in 50% (LC 50) and 95% (LC 95) mortality of *Sitophilus zeamais* adults in grains treated with wood ash of *Hymenocardia acida*:

The HA wood ash treated at the dose of 2g with an LC50 of 1.18g was observed to be the least effective in achieving 50% mortality rate compared to the control (0.23g) at day 14.

The HA ash was however more effective in achieving 95% mortality rate which showed an LC95 of 2.05g compared to the control (3.32g).

Comparison of repellency properties of wood ash at different concentrations against *Sitophilus zeamais*:

A highest repellency of 80% was recorded in the ash from *H. acida* than the control (70%). The effect gradually wore out for both the tested dusts in the following hours, until the 10th – 12th hours when there was no impact on the weevils (0%). There is therefore, an indirect relationship between time of exposure and repellency, and a direct relationship between concentration and repellency.

Table 1: Showing proximate analysis of HA wood ash and diatomaceous earth (synthetic)

Parameter wt (%)	<i>Hymenocardia acida</i>	Diatomaceous earth
Ash content	0.13	----
Moisture content	0.75	2.00
Fixed carbon	11.12	----
Volatile matter	81.25	----
pH	11.93	----
Ca	24.02	----
K	15.70	----
Amorphous silica (SiO ₂)	---	73.00
Aerosol	---	3.00

Table 2: Showing percentage mortality, progeny suppression, damage reduction, weight loss and persistence of wood Ashes (7days post-treatment)

Treatment groups	%Mortality	%Progeny suppression	%Damage reduction	%Weight loss	%Persistence
Control	0±0	27.22±6.83	64.67±2.03	74.77±2.9	0±0
DE 0.5g	100±0	44.44±5.56	2.33±0.33	4.34±2.12	96.67±1.67
DE 1g	100±0	33.33±33.33	2.67±0.67	9.09±3.97	100±0
DE 1.5g	100±0	16.67±16.67	0.33±0.33	2.66±2.52	100±0
HA 2g	94.17±2.2	27.78±14.7	9±0.58	7.64±0.55	93.33±1.67
HA 4g	100±0	16.67±16.67	3.67±0.33	4.29±0.79	95±2.89
HA 6g	100±0	50±28.87	1.67±0.33	0.94±0.3	100±0

*The Mean difference is significant at 0.05 level (Tukey's test); df= 29; F-value=1073.594

*Each datum represents the mean value of three replicates of 20 insects each

Table 3: Lethal contents resulting in 50% (LC 50) and 95% (LC 95) mortality of *Sitophilus zeamais*

Treatment	LC50	LC95
HA	1.18	2.05
DE	0.23	3.32

*The Mean difference is significant at 0.05 level (Tukey's test)

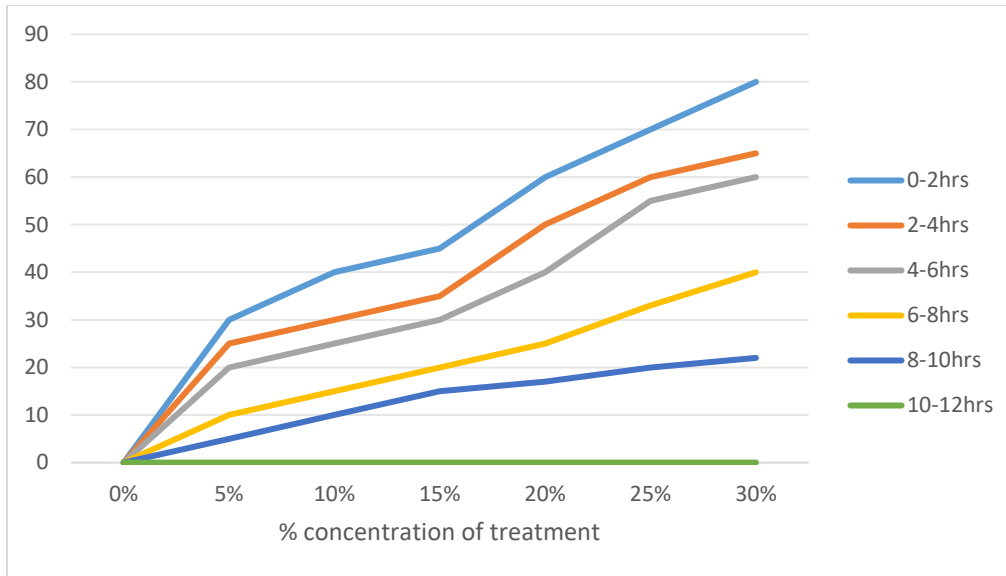


Figure 1: Percentage Repellency of *Sitophilus zeamais* at Varying Concentration of Wood ash from *H. acida* Tree

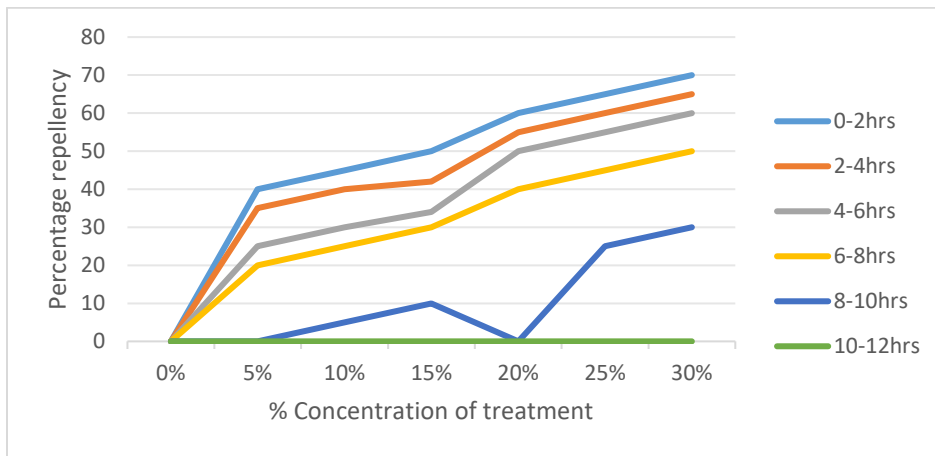


Figure 2: Percentage Repellency of *Sitophilus zeamais* at Varying Concentration of Powder Dusts from Control (Diatomaceous earth)

Discussion

This investigative study showed that the utilization and exploring of biopesticides from local trees to curb the menace of these insect pests, and their threat on the food security is a necessary way. This study showed that both the wood ash and diatomaceous earth were highly toxic and this toxicity was different according to the doses administered. The wood ash of DE was more effective than that of HA powder. This observation was well elucidated by the decreasing LC50 (0.23g) within treatment period. This is similar to the work of Gwinner *et al.* (1996) which reported that the effect of ash varies according to the type of tree. The HA wood ash caused 96.17% mortality to the weevil at the dose rate of 6g. Thus, the toxic action increased with ascending

exposure period for all the two dusts. The increase in adult mortality according to ascending exposure period and doses was due to the increase in the quantity of active ingredients contained in the insecticidal materials. This is similar to the work of Mewis and Ulrich (2001) which reported that Fossil Shield significantly reduced the survival of adult *Tribolium castaneum* and *Tribolium molitor* after seven days of exposure. Therefore, the exposure time is crucial for the effectiveness of DE, because insects' movement increases the contact of the cuticle with dust particles (Athanasassiou *et al.* 2005).

DE induced total mortality from the 7th day post-treatment at all doses except 0.5g. The same tendency was recorded by Demissie *et al.* (2008) using another commercial DE formulation (Silicosec) against *S. zeamais*. These authors reported that SilicoSec caused 100% mortality of *S. zeamais* within 7 days exposure period, at the rates 1% and 2%. The death of insects caused by diatomaceous earth could be attributed to the dehydration provoked by the abrasiveness of the small particles of this inert dust and by adsorption of oils in the body of the insect (Field *et al.* 2000) which breaks the layer of wax on the epicuticle, exacerbating the fatal loss of water as reported by Subramanyam and Roesli (Subramanyam *et al.*; 2000).

The percentage progeny suppression was not dose-dependent for both the HA and control (DE). DE powder (44.44%) has demonstrated more effectiveness than the HA which recorded 27.78% at the same period of exposure. This is being confirmed by the study of Abraham (2012) which observed that DE reduced the production of progeny by increasing adult mortality, reducing oviposition, ovicidal and larvicidal activities. Arthur and Throne (2003), showed that adult although weevils are killed by exposure to DE, some oviposition could still occur and progeny suppression may not be effective. This may explain the emergence of progeny at lowest content of DE (Table 2) as revealed by this study.

Reductions in insect population growth, percentage of grains damage and weight losses were observed for the two treatments. However, the potency of HA ash to suppress grain damage and weight loss at 1.67% and 0.94% is elucidated with significant difference with DE (0.33% and 2.33%) respectively.

This study also revealed, that, persistence of insecticide is related to time of exposure, it decreases when the storage period goes on. *H. acida wood* ash recorded decreased persistence rate 7 days post-treatment. Concerning the persistence of DE, the loss of efficacy was not really observed. Diatomaceous earth remains effective for long time compared to HA wood ash. This is being confirmed by the study of Khakame *et al.* (2012) which reported that Dryacide dust, a formulation of diatomaceous earth provided effective protection to stored maize grain against *S. zeamais* for 15 days. This revealed important to determine the action duration of each insecticidal material used for grain storage then the persistence of insecticide is also similar to the work of Ntonifor *et al.* (2011) which reported that 1 g of leaf powder of *Chenopodium ambrosioides* applied to 50 g of maize caused 100% mortality during the first week. But at the second week for the same concentration, the mortality rate decreased to 87.5%. Thus *C. ambrosioides* lost its potency and needed to be retreated. This report makes obligatory the monitoring of stored grains to avoid re-infestation by insect pests, especially when the persistent efficacy of used insecticidal material is no longer as the storage time.

The repellency for both the HA wood ash and control showed a significant difference at highest concentration of 30%, but demonstrated a similar trend. A highest repellency of 80% as seen in the ash from *H. acida* compared to the control (70%). The effect gradually wore out in the following hours, until the 10th – 12th hours when there was no impact on the weevils (0%). This is in line with the study of Trongtokit *et al.* (2005) which reported that, although essential oils are

effective when freshly applied, their protective effects usually dissipates relatively quickly with time. This study therefore, revealed that there is an indirect relationship between time of exposure and repellency, and a direct relationship between concentration and repellency which is in agreement with earlier research studies of Chaeib *et al.* (2009) and Marimuthu (2011) which indicated some plant extracts distilled from *C. citrates*, *C. zeylanicum* and *Z. officianale* had promising dose-dependent repellent properties against *Culex tritaeniorhynchus* and *Anophilus subpictus*, and a decreasing repellency at the initial period of exposure, but however, the effectiveness in killing mosquitoes increased with period.

Conclusion

The study showed that wood ashes are hazardous to maize grains that have been kept. After stopping population increase, the two ashes shielded the grains that had been preserved. Likewise, DE powder, which was employed, demonstrated its ability to effectively prevent maize weevil infection in stored maize grains. There was a positive correlation between the two ashes' reduction of damage and suppression of *S. zeamais* population growth. Both ashes' repellent properties were noted, albeit they wane with time and lost their effectiveness after ten hours. The most significant discovery here is that wood ash's repellency declined over time, but it was strongly correlated with the amount of wood ash solution used.

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