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Efficacy of combining sulfuryl fluoride fumigation with heat to control the ham mite, *Tyrophagus putrescentiae* (Schrank) (Sarcoptiformes: Acaridae)

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ABSTRACT

Sulfuryl fluoride (SF) could not control all life stages of *Tyrophagus putrescentiae* when applied at 23 °C using the highest allowable gas level. Recent work on heat treatments found 40 °C was ineffective against mites unless held for more than 48 h, a heating time that can damage ham quality. The objective of the laboratory trials reported here was to determine the lowest temperature and shortest exposure time at which SF could control mites while staying below the US EPA maximum concentration-time product (CTP) label rate of 1500 gh m^{-3} A 36-h fumigation with a CTP of 1400 gh m^{-3} killed 100% of all mite life stages at 40 °C. Mite eggs, which were determined to be the most SF-tolerant life stage, were exposed to lower CTPs of 300 or 1000 gh m^{-3} SF at 40 °C, control was estimated to require up to 125.71 h at 300 gh m^{-3} and 60.93 h at1000 gh m^{-3}. Applying heat with SF just below the maximum allowable CTP will increase the efficacy of this fumigant, but also increases the risk of exceeding the legal limit for the gas. However, at target CTPs of either 300 or 1000 gh m^{-3}, which would represent preferred commercial application rates of SF, good levels of mite control could not be achieved at 40 °C within a 48-h exposure.

1. Introduction

Dry-cured hams are valuable processed meat products that are obtained from the hind leg of a hog carcass. A typical American dry-cured ham is cured with salt, sugar and other spices applied under refrigeration (4 °C) for approximately 6 weeks, after which it is aged for six to 24 months. A cured ham loses at least 18% of its original weight during curing and aging, and obtains a unique flavor from this process. The nutrient composition, water activity and flavor characteristics make dry-cured hams a suitable food for various pests such as mites and beetles (Rentfrow et al., 2008).

Tyrophagus putrescentiae (Schrank) (Sarcoptiformes: Acaridae) is a mite pest that infests dry-cured ham through the aging process and associated aging and storage structures. *T. putrescentiae*, commonly known as ham mite or cheese mite, is an invasive species to cheese, dry meat, pet food, and grain (Rentfrow et al., 2008; Thind, 2005; Thind and Clarke, 2001). Ham mites reproduce rapidly and complete a generation within 8–21 days under ideal growth conditions. The average fecundity rate of females is four eggs/day at 25 °C and 90% R.H. (Boczek, 1991), and they can live for 80 days, which results in a high population buildup. Mites that reproduce and feed inside and outside the ham, particularly under optimal environmental conditions, can considerably decrease the economic value of dry-cured ham (García, 2004; Žďárková and Voráček, 1993). Efforts to control mites infesting Iberian hams by using low relative humidity (≤60%) resulted in complete loss of 10.9% of hams by all life stages of *T. putrescentiae* and *Tyrolichus casei* Oudemans (Sarcoptiformes: Acaridae) that had penetrated into cavities inside the hams where humidity was higher and more conducive to development (García, 2004). In highly infested ham plants in the USA, large mite populations are detectible by sight on ham and rack surfaces (Zhao et al., 2016a,b) and can elicit regulatory action with great expense to the ham producer.

Methyl bromide (MB) is a very effective fumigant pesticide that has been used for decades to control ham mites in the US (Marriott and Schilling, 2004). However, MB is classified as a significant ozone depleting substance under the Montreal Protocol (United States Environmental Protection Agency, 2016). Therefore, MB is

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being phased out for all industries in the US and consequently, many MB alternatives, including fumigation with sulfuryl fluoride (SF) and applications of extreme temperatures, have been explored (Boina et al., 2008; Fields, 1992).

SF is a colorless, odorless and non-flammable fumigant that was registered in the US for use in stored-products in 2005 (Phillips et al., 2012; Reichmuth et al., 1998). Compared to MB and hydrogen phosphide, SF is a non-ozone depleting and is a non-corrosive gas that has a faster diffusion rate than MB (Baltaci et al., 2009; Navarro, 2006). A satisfactory SF application can be achieved by using an adequate concentration (C) of SF in $g.m^{-3}$ for an effective exposure time (T) in hours. Therefore, the SF application label rate specifies a "concentration-time product", which is a value equal to the product (P) of C and T, known commonly as the CTP. The US Environmental Protection Agency (EPA) established a maximum application dosage of SF use on stored-products, including dried hams and other foods, at a CTP of 1500 gh m^{-3}, which allows some flexibility with exposure time and gas concentration in a commercial application as long as the documented CTP is lower than the specified maximum (Phillips et al., 2012).

Previous studies showed that SF controlled the post-embryonic stages, i.e., larvae, pupae and adults, of stored-product pests such as the red flour beetle, *Tribolium castaneum* (Herbst) (Coleoptera: Tenebrionidae); Mediterranean flour moth, *Ephestia kuheniella* Zeller (Lepidoptera: Pyralidae); cigarette beetle, *Lasioderma serricornne* (F.) (Coleoptera: Anobiidae), and other beetles and moths (Athanassiou et al., 2012; Bell and Savvidou, 1999; Reichmuth et al., 2003; Small, 2009). However, eggs of stored-product insects are more difficult to control with SF under the same conditions than are the post-embryonic life stages. Similar results were found in using SF for control of stored-product mite eggs. Phillips et al. (2008) studied SF efficacy for control of *T. putrescentiae* life stages at 23 °C, a typical temperature in ham-aging rooms, during experimental fumigation. They estimated the lethal concentration of SF needed to kill 95% of mite eggs was about 90 g m^{-3} SF at 23 °C after 48 h, which is equivalent to a CTP of 4320 gh m^{-3}, or nearly 3-fold higher than the maximum allowable CTP of 1500 gh m^{-3}.

Potential solutions studied for improving the mortality of insect eggs can be the use of SF at higher concentrations, or applying SF in combination with either heat, modified atmosphere or/and other fumigants such as hydrogen cyanide (Baltaci et al., 2009; Bell and Drinkall, 2000; Emekci, 2010; Reichmuth et al., 2003; Reichmuth and Klementz, 2008; Su and Scheffrahn, 1990). We recently studied the mortality of extreme cold and hot temperatures applied to all life stages of *T. putrescentiae* for different exposure times, and found that eggs were the most tolerant life stage to heat, but that they could be controlled at 45 °C in 21 h or at 40 °C in 96 h (Abbar et al., 2016). It was concluded that heat alone to control ham mites may put hams at risk with exposure to 45 °C and that longer exposures at lower temperatures could unduly interrupt commercial activities. Temperature has a considerable impact on the fumigation efficacy for insect control. Higher temperature increases insect metabolism and respiration and treated insects consequently take in more fumigant at a faster rate (Fields et al., 2012). Therefore, using heat in combination with SF may allow for a lower SF concentration to control post-embryonic stages of stored-product insect pests. Bell et al. (1998) and Reichmuth et al. (2003) reported that increased temperatures reduced the amount of SF needed to control adults of *E. kuehniella*, *T. castaneum, Tenebrio molitor* L. (Coleoptera: Tenebrionidae) and *Oryzaephilus surinamensis* (L.) (Coleoptera: Silvanidae). The objective of the research reported below was to investigate the efficacy of combining SF with high temperatures to control eggs and mobile life stages of *T. putrescentiae* as a fumigation alternative to MB, with the intention of using a CTP for SF that is both effective and within regulatory standards.

2. Materials and methods

2.1. Mite cultures

Mites were reared in 0.95 l glass canning jars (85 mm diameter, 160 mm height) on a diet that was a mixture of commercial dried dog food and several ingredients as in previous studies (Abbar et al., 2016). Approximately 80 g of dry whole dog food kibbles were added to each jar and mixed with 100 ml of cooked, thick gravy-like mixture. The gravy ingredients for six rearing jars included dog food (160 g), 475 ml tap water, 5 g of each of agar (Mooragar, Incorporated Loomis, CA), yeast (MP Biomedicals, LLC, Solon, OH), alphacel (MP Biomedicals, LLC, Solon, OH), mixed vitamins (Vanderzant modification vitamin mixture for insect diet; MP Biomedicals, LLC, Solon, OH), 25 ml of antifungal salt solution in 15% ethanol (methyl-*p*-hydroxybenzoate; ICN Biomedicals, Inc. Aurora, OH), and 25 ml of glycerol (Fisher Scientific, Pittsburgh, PA). Commercial dog food (160 g) was coarsely ground, mixed with water and brought to a boil. With the exception of antifungal salt, all other ingredients were added to the mixture and cooked to a boil again. The antifungal salt solution was then added to the gravy and cooked for 30 min to reach the desired thickness. The hot mixture was then added to dry whole dog food in the rearing jars and cooled to room temperature (23 °C) prior to inoculation with mites. After mites were added to diets, the jars were sealed with labeled filter paper (90 mm, Fisher Scientific®, Pittsburgh, PA) in the metal lid ring. *T. putrescentiae* colonies for this research originated from a local pet food plant and had been in culture for more approximately four years at 25 °C and 70% RH in darkness at our laboratory at Kansas State University.

2.2. Pre and post-fumigation bioassay preparation and mite mortality assessment

Methods used here were similar to those used in our previous work (Abbar et al., 2016). Mite life stages were separated into eggs and mobile stages for all SF fumigation bioassays and placed into ventilated glass vials (4.0 ml; Fisher Scientific®, Shell vial, 12 × 35 mm, Pittsburgh, PA), each including 0.2 mg of soft dog food. Each experimental vial included either twenty mobile stage mites, comprised mostly of 7–10 day old adults and no more than 25% nymphs, or 10 eggs that were 1-2-d old. Eggs and mobile mites were added to each vial from the mite colony using a one-haired brush. Eggs were carefully placed onto double-side adhesive tape that was stuck to a narrow piece of black paper (4 × 25 mm). Vials were covered with a fine mesh cloth (125 μm opening in the weave) to retain mites and to provide ventilation for SF during the bioassay and humidity during both the fumigation and post-fumigation periods. After fumigation, the vials were kept in fresh air within the incubator at 25 °C and 70% RH. Mortality of adults and nymphs was assessed based on lack of movement under physical stimulus after a 2-day recovery period, and unhatched eggs were considered dead four days after fumigation. Nymphs from hatched eggs were able to easily walk off of the sticky black paper, as with our past experiments conducted in this way (Abbar et al., 2016).

2.3. SF fumigation

SF gas from a ProFume™ gas cylinder (99.9% purity, supplied at that time by Dow AgroScience LLC, Indianapolis, IN) was transferred to Tedlar® gas bags (CEL Scientific Crop, Santa Fe Spring, CA). The appropriate amount of SF was drawn and injected into each air-tight fumigation jar following the removal of an equal volume of air to achieve the desired SF concentration. SF at 99.9% purity was used as a standard for quantitative analysis. SF was introduced using a