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Action of Different Exposures of Chilled Atmospheric Treatments on the Mortality of Granary Weevil and Embryo Viability of the Treated Wheat

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Abstract: The granary weevil, *Sitophilus granarius* (L.), is considered a serious pest in stored grain worldwide. As residual-based protection possibilities become scarcer, the development of eco-friendly control technologies that can be implemented in practice is becoming urgent. In this spirit, our objective was to assess the effectiveness of different levels of atmospheric cooling against *S. granarius* under laboratory conditions. We also analysed the effects of cooling on progeny generation and the viability of treated wheat. Thus, we investigated the consequences of atmospheric exposures to temperatures of −5, −10, −15, −20, and −25 °C for 60, 75, and 90 min on these factors, and also explored the effects of nearby ranges using extrapolation. The viability of the treated wheat embryo was analysed using a TTC test. Our results showed that the highest efficacy was observed at an atmospheric cooling temperature of −25 °C (with a parallel recorded temperature of −10.5 °C in the stored grains zone), with a 90 min exposure at suboptimal relative humidity (40%). At 60% relative humidity, the mortality averages were more dispersed, and the expected efficiencies above 95% occurred at longer exposures. The post-suppressive effect of cooling can be confirmed in all three exposures. The different cooling temperatures of the tested exposure times did not produce any appreciable changes in the viability of treated wheat embryos. In conclusion, our results show that the use of atmospheric cooling can be an effective solution for stored product protection.

Keywords: atmospheric cooling; control method; mortality; relative humidity; *Sitophilus granarius*; stored product; viability of winter wheat

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1. Introduction

The granary weevil, *Sitophilus granarius* (L., 1758) (Coleoptera: Curculionidae), is a cosmopolitan species and is one of the most devastating arthropod pests of stored cereals [1]. The damage caused by this species reduces the quality and quantity of stored grain [2]. It reduces the content of thiamin (vitamin B1), riboflavin (vitamin B2), and α-tocopherol (vitamin E) in grains [3,4], and it has been shown to change the fatty acid composition [4,5] and increase the uric acid content of stored cereal grains.

The global assortment decline of residual insecticides significantly reduces the range of management opportunities for stored product protection [6]. Thus, research into environmentally friendly control technologies is increasingly becoming a priority in line with the criteria of sustainable agricultural production and integrated pest management (IPM). Several different methods were tested or experimentally developed for the protection of stored products, such as the effect of different ionising radiations [7,8] or modifications of atmospheric parameters [9,10] on stored product pests, as well as the stored product protection aspects of natural substances (plant essential oils, diatomaceous earth, inert

dust, etc.) added to stored products for the same purpose [11,12]. For all these reasons, the translation of the results of new scientific experiments in this field into direct practice is of particular relevance today.

One sustainable control method is the artificial alteration of the atmospheric environment surrounding stored products [9]. The establishment of air temperature conditions other than the temperature preference of *S. granarius* has been shown to reduce the vitality of the pest. Decreased egg production, altered activity, and mating behaviour were reported [13]. Finally, the persistence of these effects may trigger pest population mortality. Several lines of research have addressed the possibilities of changing the atmospheric temperature for the protection of stored products, dealing exclusively with the possibility of applying heat stress against arthropods damaging stored products [14–16].

Insects are poikilothermal organisms. Thus, the temperature is a critical abiotic factor, causing significant physiological changes in them [17]. The results of experimental work by Fields [18] confirmed that the more extreme the temperature, the faster the insects die. Thus, according to his data, absolute mortality is induced within a few minutes at −20 or 55 °C. The lethal temperature varies considerably between species and depends on the developmental stage, relative humidity, and acclimatisation. The pest-killing effect of low temperatures has been used in stored product management in many countries, which has even allowed for the reduction of fumigation techniques [14]. The effect of air cooling on storage pests has been studied mainly on closely related species of *S. granarius* such as *S. oryzae* (L., 1763) and *S. zeamais* (Motschulsky, 1855) [19,20]. In their experimental work, Nakakita and Ikenata [14] showed that the oxygen consumption of adult *S. oryzae* and *S. zeamais* retrogressed with decreasing temperature. Metamorphosis and hatching from eggs stopped completely at 10 °C for all species. The knock-down effects of temperature changes were also analysed in combination with residual insecticide applications. The work of Kljajic et al. [21] showed that short-term cooling to −5 °C enhances the insect-mortality-inducing effects of dichlorvos and deltamethrin.

The side effects of using cooling-based eradication methods for pest control may cause changes in the viability of the treated stored grains, affecting embryonic survival [22]. This, in turn, may directly impair the germination ability of the seeds.

Although some results are available on the effects of low temperatures, even freezing, on *S. granarius* [18,23], there is rather little relevant information on mortality and seed viability under different negative temperatures and relative humidity settings. Previously, we investigated the effect of the upper pessimum range on *S. granarius* [24]. In connection with these related investigations, our experimental study aims to provide information on the efficacy of extremely low temperature freezing treatments in different relative humidity environments against *S. granarius* in winter wheat grain. Our objective was to determine the effect of different exposure times in combination with different freezing temperatures on the mortality of *S. granarius*, as well as assess progeny production. In addition, we were curious about the degressive effect of these treatments on the viability of treated wheat grains.

2. Materials and Methods

2.1. Experimental Sample Preparation

Infestation-free wheat grains were used for our experimental work (one-year-old with 13.5% moisture content, stored at 21 °C). At first, 100 g wheat grain was placed in glass jars. Then, 20 healthy *S. granarius* imagoes (mixed sex and age) were put into the glass jars containing the grain samples. These sample containers were covered with well-ventilated tissues and placed in a Pol-Eco Apartura KK 1450 climate chamber (Wodzislav-Slenski, Poland) at 26 ± 3 °C, 60 ± 5% relative humidity (RH) and a 16/8 photoperiod. These are the most favourable developmental conditions for *S. granarius* [25]. Two days elapsed between the experimental sample preparation and the beginning of the actual laboratory treatments to allow egg-laying to take place.

2.2. Insect Eradication Test

To survey the impact of the low temperatures combined with different relative humidities (RH) on insect mortality (besides the intact samples), a total of five target temperatures were adjusted. The examined temperature values, starting from the lower tolerance border value (−5 °C) of the species, were −5 °C, −10 °C, −15 °C, −20 °C, and −25 °C. The effects of these temperature values were analysed in combination with two different relative humidity values, 40 ± 5 and 60 ± 5 percent RH (hereafter 40 and 60% RH, respectively). The exposure time (exposure to freezing) was uniformly 75 min at 60% RH and 60, 75, and 90 min at 40% RH. After each cooling exposure, the temperature of the grains was measured with a ThermoPro TP-01H digital thermometer (ThermoPro, Atlanta, GA, USA).

Alfa Laval CCEH251 (1AS 230V BO PCE EP 7.0 CU) freezing chamber (Alfa Laval Krakow Sp. zoo, Krakow, Poland) was used for the cooling experiment. All treatments consisted of 4 repetitions, and right after the freezing, the samples were reinstated into the climate chamber at 26 ± 3 °C and the above-mentioned two different relative humidity values. Dead adults were counted after 48 h. At this point, all adults (dead or alive) from the treated samples were removed and placed in the climate chamber (26 ± 3 °C, 60 ± 5% RH, 14/8 photoperiod). Progeny survival was determined after 45 days, from the adult emergence analysis of samples originally containing eggs, larvae, and pupae.

2.3. Determination of Viability of Wheat Grain by TTC Test

To assess the viability of embryos, a TTC (triphenyl tetrazolium chloride) test was used, with a slightly modified version of the methodology recommended by Carvalho et al. [26]. During preparation, seeds were immersed in water, using 10 mL of water for each sample, for six hours at 20 °C. The seeds were then cut in half lengthwise along the embryo and placed in plastic containers between filter paper discs. Staining was done with a 1.0% TTC solution and placed in an incubator at 30 °C for 2 h. The amount of TTC solution used for staining each sample was 2.5 times the weight of the filter papers. Viable seeds were those in which the embryo was bright red and uniformly stained, with no large unstained areas.

The viability percentage of seeds (V%) was determined as follows: V% = (nv/tn) × 100, where nv = the number of viable embryos, tn = the total number of embryos.

2.4. Statistical Analysis

Abbott's formula [27] was applied to determine mortality values. The Shapiro–Wilk test was used to assess normality in the mortality data of granary weevil. The evaluation of the normal distribution of data was by Ghasemi and Zahediasl approach. Data were analysed using a two-way ANOVA in SPSS 11. 5 software (response variable: adult mortality; main effects: temperature, humidity). The nature of the relationship between 75 min cooling exposure set at different humidity levels and mortality was investigated using correlation regression analysis. Mortality values for progenies were also statistically analysed using the Student's t-test and one-way ANOVA.

Moreover, the effects of different levels of cooling at 75 min exposure on the viability of wheat grains at different humidity levels (40 and 60% RH) were compared using a Student's t-test ($p < 0.05$). In parallel, the registered viability values triggered by applied minimum temperatures and different cooling exposures at 40% RH were tested by one-way ANOVA. The Tukey HSD test ($p < 0.05$) was used to separate means uniformly.

3. Results

3.1. Insect Mortalities Triggered by Freezing

Table 1 shows the recorded stored grain temperature data. The 100 g of wheat has a significant temperature buffer effect. Even at the longest exposures to the set atmospheric minimum values, grain temperatures were up to 10 °C higher for several treatments. Lower temperature settings and longer exposures naturally create a lower temperature zone around the pest in the stored grain zone.

Table 1. Stored grain temperature [mean (°C) ± SE] recorded as a function of set atmospheric temperatures and exposures (n = 4).

Adjusted Atmospheric Temperature		**−5 ± 0.50**	**−10 ± 0.50**	**−15 ± 0.50**	**−20 ± 0.50**	**−25 ± 0.50**
stored grains temperature	at 60 min of exposure	7.8 ± 0.23	2.8 ± 1.25	0.2 ± 0.54	−3.9 ± 0.65	−6.4 ± 0.21
	at 75 min of exposure	5.3 ± 0.65	0.7 ± 0.44	−2.7 ± 0.76	−7 ± 0.26	−10.5 ± 0.97
	at 90 min of exposure	3.8 ± 1.11	−1.4 ± 0.85	−4.2 ± 1.28	−9.2 ± 1.06	−15.3 ± 1.52

Abbot's corrected mortalities at the different minimum temperatures and relative humidity values are shown in Figure 1. The mortality values were unequivocally increased as a function of the applied temperatures with 75 min exposure times at both 40 and 60 RH values. At both 40 and 60% RH, the effect of temperature decrease on observed mortality change can be statistically proven (df = 4; F = 12.112; $p = 5.62 \times 10^{-6}$) ($p < 0.05$). The data registered at different relative humidity settings are statistically different (Student's t-value: 0.015). In contrast, the consequences of different humidity values (df = 1; F = 0.367; $p = 0.548$), as well as the interaction of temperature and relative humidity (df = 4; F = 1.186; $p = 0.336$) on the mortality change cannot be confirmed by a two-way ANOVA ($p > 0.05$).

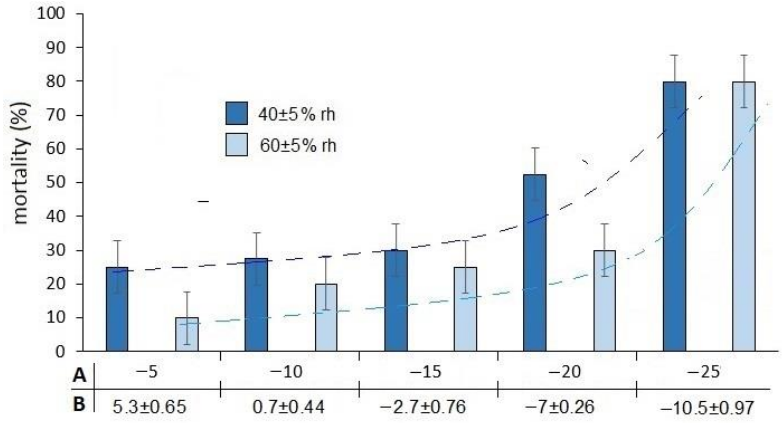

Figure 1. Mortality percentage (mean ± SE) of *S. granarius* adults (corrected by the Abbott method [27]) treated with two different relative humidity values as a function of the applied temperatures with a 75 min exposure time. The dotted lines indicate trends of exponential change. **A**: applied atmospheric minimum temperature; **B**: stored grain temperature (mean ± SE) which is recorded in parallel with the atmospheric temperature setting.

The tendencies of the triggered mortality are strictly exponential in the case of both relative humidity values. The relationships are strong for both applied relative humidity values, but for 40% relative humidity, the relationship is somewhat stronger (40% RH: $y = 15.856e^{0.2973x}$; $R^2 = 0.8902$; 60% RH: $y = 6.6245e^{0.4564x}$; $R^2 = 0.9190$) (e is a mathematical constant: Euler-type value). Additionally, in both 40 and 60 percent humidity settings, about 80 percent mortality of the experimental population was triggered by the applied temperature of −25 °C (stored grain temperature in the pest zone: −10.5 °C) for the 75 min exposures. At 60 percent relative humidity, mortality averages are more dispersed, and efficiencies between −5 and −20 temperature settings triggered much lower mortalities than in the case of the same settings at 40 percent relative humidity. In the case of a 75-min exposure, the complete extermination of the experimental insect population can be expected with atmospheric minimum values of −28.46 °C at 40% RH and −27.23 °C at 60% RH.

The mortality percentages produced by data extrapolation as a function of the interaction of exposures and applied temperatures are shown in Figure 2. The mortality rate has increased with exposure time and lower pessimum temperature values. Atmospheric temperatures approaching zero until −20 °C (stored grain temperature in the pest zone:

from 5 to −7 °C) have not triggered an acceptable efficacy in the case of 60 min of exposure yet. The effect of a 75 min exposure at −20 °C cooling produces mortality to between 70 and 80 percent. Mortality above 90 percent was only registered when cooling the samples to −25 °C for 75 min (stored grain temperature in the pest zone: −10.5 °C). Combinations of atmospheric cooling of the samples at or below −20 °C or for longer than 90 min in all cases triggered total eradication of the experimental pest's population.

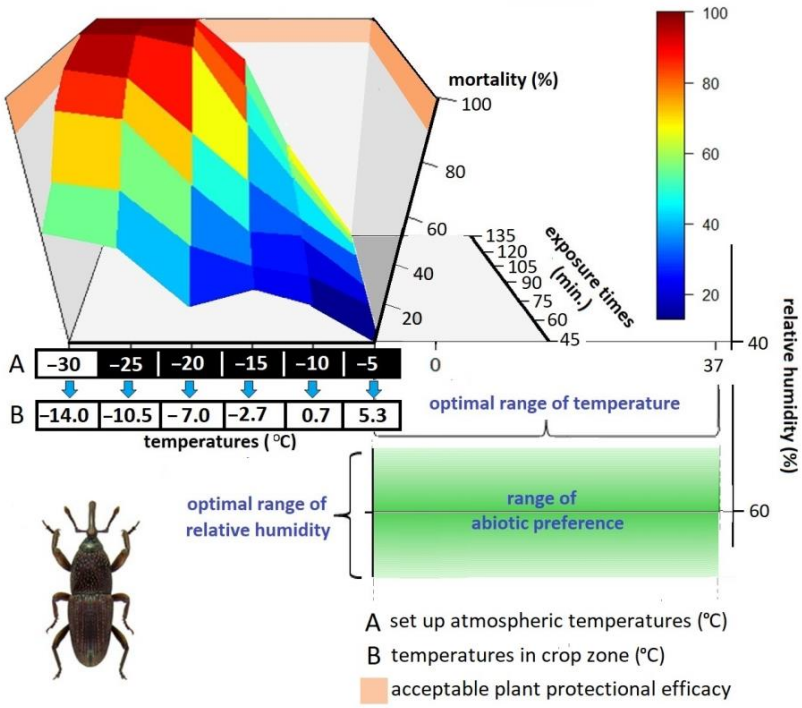

Figure 2. Extrapolation of the effect of set-up temperatures and their exposure times on the mortality of the granary weevil as a function of its ecological preference [25].

The continuously decreasing atmospheric temperature effects examined at different exposure times (60, 75, and 90 min) verifiably caused a clear decline in the number of adult progeny (Table 2). This observation was statistically confirmed for all exposure times ($p < 0.05$) by one-way ANOVA. After forty-five days, the number of progenies was statistically verified to be different for some exposure times of −20 and −25 °C atmospheric cooling ($p < 0.05$). In contrast, no similar correlation was demonstrated for different exposure times at lower cooling settings (−5, −10, and −15 °C). The longest exposure at the lowest temperature (−25 °C for 90 min) resulted in the greatest reduction in progeny, effectively wiping out all developmental stages of the experimental insect population and preventing the imago stage from developing.

Table 2. The observed progeny production of *S. granarius* 45 days after the cold treatment., Statistical inferences with $p < 0.05$ were considered significant.

Treatments		**No. Progeny**						
control				6.75 ± 0.75			statistical relationships (df = 19)	
set up the atmospheric temperature		−5 °C	−10 °C	−15 °C	−20 °C	−25 °C	F	*p*
exposure times	60 min	3.25 ± 0.62	1.75 ± 0.75	0.75 ± 0.47	0.75 ± 0.75	0.75 ± 0.25	11.277	*0.001*
	75 min	3.00 ± 2.04	2.00 ± 0.40	3.50 ± 2.06	1.00 ± 0.41	1.00 ± 0.41	1.834	*0.003*
	90 min	0.75 ± 0.25	0.75 ± 0.47	2.00 ± 1.68	0.50 ± 0.28	0 ± 0	21.091	4.7×10^{-5}
statistical relationships (df = 11)	F	1.434	1.434	0.008	11.301	*20.509*		
	p	0.243	0.243	0.928	*0.002*	*0.001*		

The mean number of adults ± SE are shown; italic characters denote statistically significant correlations).

3.2. Viability of the Treated Wheat

The embryo viability of the treated wheat grains was uniformly shown to be between 75–100 percent for each cooling level at different relative humidity values with the 75 min long exposure (Figure 3). The obtained values could not be determined to reflect statistically significant differences ($p > 0.05$).

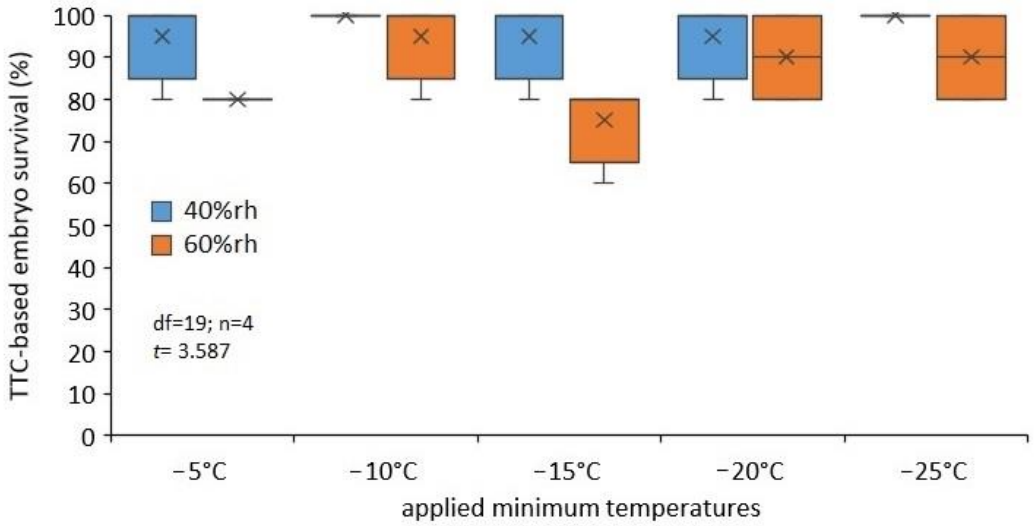

Figure 3. TTC-based (triphenyl tetrazolium chloride) embryo survival at 75 min of exposure by different applied minimum temperature and relative humidity values.

When examining the effects of different exposure times at different temperature settings on seed viability at 40% relative humidity (Figure 4), it can be seen that the dispersion of values is more significant as one moves towards longer exposures at the lowest cooling values. Independently of this observation, no effects of either the three examined exposure times or the interaction of the main influencing factors (different temperature and relative humidity parameters) on the seed viability could be determined by a two-way ANOVA ($p > 0.05$).

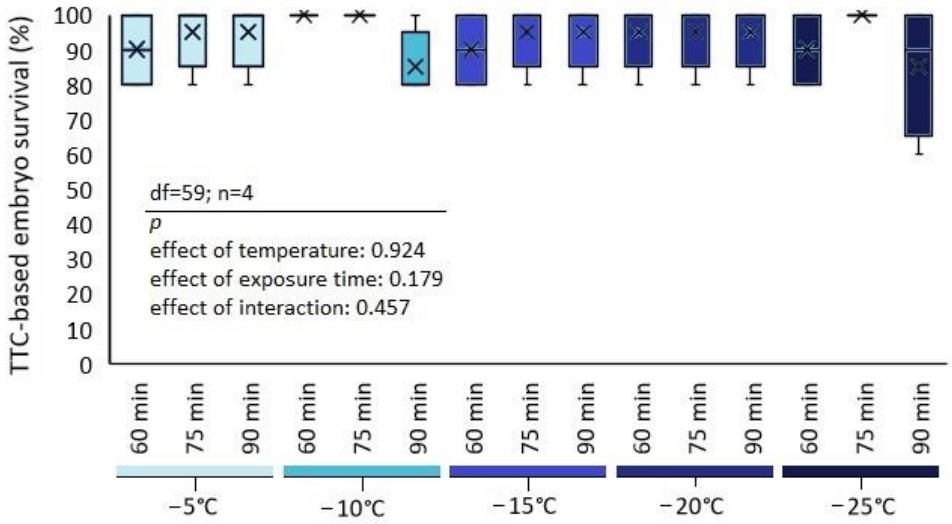

Figure 4. TTC-based embryo survival at 60, 75, and 90 min of exposure and 40% relative humidity at different applied minimum temperatures.

4. Discussion

The optimum life activities of insects, as poikilotherm organisms, are realised in the presence of a specific abiotic interval [17]. This is called the optimum range. The vitality values of the individual development stages measured under the influence of various abiotic parameters are best characterised by their individual isothermal curves [28]. Values different from this can already lead to disruption of their homeostasis. In the case of insects, this disorder manifests itself in the shift of ontogenetic processes and the slowing down of life activities. Finally, beyond a certain value, the affected organisms die due to irreversible physiological processes [17,29].

Grain stored at 20 °C will negatively affect the development of most arthropod crop pests; a notable exception is *S. granarius* which can complete its development even at 15 °C [18]. Consequently, the insecticidal effects are expected well below this temperature range. Our experiment demonstrated that, in contrast to suboptimal high temperature exposures, where shorter exposures to higher values caused total mortality [24], longer exposures to lower temperature values closer to the lower tolerance trigger similar mortality.

The artificial temperature decrease of the stored product atmosphere can be deemed to be a potential protection against *S. granarius*, a supposition confirmed by our data. The highest efficacy has been detected at an atmospheric setting of −25 °C (stored grain temperature: −10.5 °C) with a 90 min exposure at suboptimal relative humidity values (40 RH). Furthermore, the insect perishing efficacy can be enhanced by the decrease in minimum temperature and increased exposure time, but at the same exposure, further cooling of about 1–2 °C causes 100 percent mortality. Our laboratory experiment was carried out with only 100 g of grain. Naturally, under storage conditions, the weight of cereals is much greater. Therefore, the buffering effect of the grain mass is much greater under storage conditions. For this reason, in practice, the exposure time to low temperatures will be significantly extended to cause the target pest to perish.

In his study, Fields [18] calls the temperature ranges we examined the lethal range for stored product pests, dividing it into two more sections from −5 to −10 °C, where death is triggered in weeks to months (acclimated) and from −15 to −25 °C, where death occurred in minutes. Overall, the more extreme the temperature, the more quickly insects die, with death occurring within a few minutes at −20 °C.

Our experiment confirms this finding in that the stored cereal item as a medium can significantly dampen and postpone the insecticidal effect of atmospheric cooling. In our case, due to the buffering effect of the grain medium, freezing at −25 °C (stored grain temperature: −10.5 °C) induces about 80 percent insect mortality after 75 min of exposure. Our calculations indicate that an atmospheric cooling of −28.46 °C at 40% RH is required to induce total mortality, or −27.23 °C at 60% RH. These findings are in line with the statements of Marpaung [30], who reports on the preservation of maize embryo viability besides successfully using atmospheric cooling against *S. zeamais*.

According to Grgac et al.'s [31] work, the cell membranes are likely to be the first targets of freezing injury. The cell membranes lose their integrity in frost-sensitive insects subjected to freezing stress, whereas their integrity is maintained by accumulated cryoprotective molecules and proteins in frost-tolerant insects. Furthermore, the formation of pointed ice crystals perforates the cell membranes, which eventually leads to the death of injured cells [32]. Basically, to avoid these adverse effects, the compounds (trehalose, ribulose) that accumulate in the insect's cells aim to achieve a controlled, slow freezing of the water molecules [33].

Thorpe et al.'s [34] composite model indicates that there is an optimum aeration strategy characterised by specific temperature and humidity parameters that result in the lowest damaging insect population. Aeration with ambient air was also shown to render the population dynamics of *S. oryzae* relatively insensitive to grain moisture content and temperature. Consequently, the most outstanding insect killing efficiency can be expected from unannounced suboptimal temperature effects. This statement is confirmed by several entomological studies [18,35–37].

The basic requirement for crop protection technology is to be able to reduce the pest below economic damage thresholds [38] in a short time. As part of a biorational approach to insect pest control, modern methods of controlling stored products increasingly rely on the use of low or high temperatures as a physical control method, or a combination of these with some other pest control methods such as chemical control agents [39–41]. Our study showed that cooling has an insecticidal effect on the *S. granarius* imago and suppresses its progeny. An important specificity of this control method is that the germination capacity of treated wheat batches was not reduced due to the embryonic intactness of the tested setting parameters.

The weakness and disadvantage of the practical application of this method is the difficulty of handling large batches of stored cereal items. The technical realisation of the cooling technology for batches of cereal stored in a silo or silo tower can raise several issues, such as providing a uniform temperature throughout the batch. The latter concern may represent a significant additional cost in view of current energy prices [42], which may result in a significant increase in the cost of production of the produced crop, which may be a fundamental limitation for the successful practical adaptation of the technique. This methodology may also be a promising tool for the conservation of seed stock quality. The primary benefit of the method is therefore to provide a theoretical basis for stored product protection based on atmospheric temperature variation in the storage area, which could become the basis for a successful practical application method in the near future.

Overall, our results could contribute to the development of a reliable control method for plant product storage, which, even in combination with other control methods, could meet the criteria for integrated pest management (IPM).

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