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REVIEW PAPER

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Review on stored grain insect pheromones

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Abstract

Using pheromones to control and monitor stored grain pests is a technology applied in different countries. The present review identified the primary compounds used to prevent or monitor stored grain pests, their chemical structures, functional groups and attraction mechanisms. We discuss the aspects of historical evolution, the geographic distribution of research on stored grain pests, the methodological approaches used in developing this research, the strategies used to control and monitor these pests, and the chemical synthesis of the compounds used as pheromones. We found 109 published articles that reported data on pheromones. Aggregation and sexual pheromones were the most used for control and monitoring. The surveys were distributed across six continents; most studies were conducted in North America. Laboratory studies were the most common, followed by field studies. Management using pest monitoring was the most common. Different synthetic routes were observed when conducting the studies. These works showed the improvement of these synthetic routes to obtain pheromone constituents. This review highlighted the main aspects of using pheromones for controlling or monitoring stored grain pests.

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Introduction

Agribusiness is one of the main segments of the Brazilian economy, contributing significantly to national and international trade and standing out by exporting commodities in the sector. The 2020/2021 Brazilian crop reached a production of 253.2 million tons of grains, with Brazil becoming the world leader in soybean production, estimated at 136 million tons. Brazil is also one of the largest producers of rice and corn, with an average output of 93 and 12 million tons, respectively (CONAB, 2021). The 2022 harvest is forecast at 261.6 million tons of grains. This is a 3.8% growth in production for 2020/2121, and with an increase of 3.4% in the planted area, it is estimated that it could reach 6.4%. Such voluminous production requires safe and efficient storage of these products to guarantee quality products to the consumer market. The Food and Agriculture Organization of the United Nations (FAO) recommends that grain-producing countries have more than 20% static storage capacity for the quantity produced (CONAB, 2021), indicating the importance of storing and preserving the products to minimize losses caused by deterioration and insects to ensure a better food supply to consumers. Global losses caused by insect pests in stored products are estimated at 9% in developed countries and over 20% of production in developing countries, both qualitative and quantitative. The presence of insects, fragments of dead insects and excretions that cause contamination make the grains unfit for human consumption (Pimentel, 1991; Tripathi, 2018; Weaver and Petroff, 2004), which justifies the precautions for preventing insects in stored grain. In Brazil, most problems with stored grain pests are caused by beetles (Coleoptera) and moths (Lepidoptera) (Ashworth, 1993; Lorini, 2015).

These insects are commonly controlled using chemical insecticides such as phosphine and a group of pyrethroids such as deltamethrin and bifenthrin (Fig. 1). However, the emergence of pests resistant to these compounds is a global problem (Hagstrum and Phillips, 2017; Lorini, 2015; Urrutia *et al.,* 2021). For example, phosphine, one of the main insecticides used to control stored grain pests, is recognised for having little residual effects and has shown a reduction in its efficiency due to the emergence of resistant insect populations, which may be related to the presence of a specific gene, or the selection pressure caused by the consistent application of the product. Furthermore, the continuous and sometimes excessive use of insecticides can cause harm to human health and non-target organisms and cause environmental pollution (Wakil *et al.,* 2021).

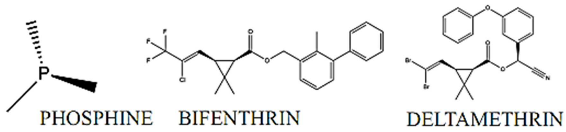


Fig. 1. Most commonly used chemical insecticides to control stored grain insect pests.

Countries such as Brazil, a large grain producer, should prioritise the search and adoption of methods capable of rationally reducing the use of conventional techniques to control pests that affect stored products (Sammani *et al.,* 2020a). The main advances capable of combating or alleviating the problems caused by conventional pest control of stored grains are hermetic storage, use of modified/controlled atmosphere, diatomaceous earth, use of compounds of botanical origin, bioinsecticides or bioinsecticides of bacterial origin such as compounds obtained from the bacterium *Saccharopolyspora spinosa*, and the use of pheromones to monitor and control these pests (Mudiyanselage *et al.,* 2020). Pheromones are compounds of natural origin used alone or in combinations that stimulate different behavioural responses in recipient individuals such as aggregation behaviours or sexual attractiveness. For example, they can be used as attractants in traps, serving as bait in pest monitoring and control (Campos and Phillips, 2013; Chambers *et al.,* 1990). Among the main control and monitoring, techniques are mating interruption or sexual disruption, mass capture and attract-and-kill techniques (Campos and Phillips, 2013; El-Sayed *et al.,* 2006). The advantages of using pheromones are that they have high specificity and are not harmful to non-target organisms such as natural enemies, and they do not present toxicity to humans and the environment (Campos and Phillips, 2013; Chambers *et al.,* 1990).

In the present study, we review the current knowledge on insects that act as pests in stored grains. We also seek and determine which are the primary pests controlled and monitored through pheromones and describe the products used to manage the insect pests of grain storage.

Research questions

1. Which pheromones are described?
2. Which pests are monitored?
3. Which pests are controlled?
4. Which pheromones are attractive?
5. What are repellents?
6. Which pheromone structures have already been described?
7. What pheromones are commercially available, and how are they used?
8. What is the control form?

Materials and methods

Experimental database

A search was carried out for articles that evaluated the control or monitoring of stored grain pests through pheromones from February to April 2022, covering studies published from 1969 to 2022 using Scopus, Scielo, and PubMed, respectively Web of Science and Elsevier. The employer descriptors were "storage pests" or "stored product insects" and "aggregation pheromone" and "grain" or "grain storage" or "grain weevil" and "insect damage to stored grains" or "pest control" or "pests in storage" and "semiochemicals" or "sexual pheromone" and "stored product". This resulted in 715 articles. The articles had to comply with the inclusion criteria established in the protocol, which was studies that reported the efficiency of pheromones in pest control that were fully available in the databases and showed how to use pheromones in monitoring/controlling, attracting or repelling stored grain pests. The exclusion criteria were works from dissertations, course conclusion work, theses, and reviews, that were not in English or did not show the form of application of pheromones in the control/monitoring of stored grain pests. In total, 128 manuscripts were selected for the systematic review. After selection, the articles were read in their entirety, and the information extracted included the year of publication, the country where the research was carried out, type of evaluation (control/monitoring), type of pheromone (including chemical class, commercial or standard name, structure or chemical name, commercial use, efficiency and if used in combination), the herbivore and parasitoid species and whether there was attraction and repellence, whether is it was a field or laboratory study, type of trap and bioassay and statistical test applied (result, standard deviation, standard error, N sample).

Results and discussion

Results and Discussion

After reading the articles, 20 compounds used to control or monitor stored grain pests were identified using pheromone baits (Table 2. Fig. 2).

Table 1. Pests of higher occurrence in stored grains in Brazil.

Name	Common name	Main grains and derivatives affected
Coleoptera		
Cryptolestes ferrugineus (Stephens, 1831) (Coleoptera: Laemophloeidae)	Rusty Grain Beetle	Flour, Bran, various kinds of cereal
Lasioderma serricorne (F., 1792) (Coleoptera: Anobiidae)	Cigarette beetle	Tobacco, Soy, Tobacco, Maize (corn)
Oryzaephilus surinamensis (L., 1758) (Coleoptera: Silvanidae)	Grain beetle	Maize, all occurrence cultures
Rhyzopertha dominica (F., 1792) (Coleoptera: Bostrichidae)	Beetle	Rice, Barley, Soybeans, Maize, Wheat, Sorghum
Sitophilus zeamais Motschulsky, 1855 (Coleoptera: Curculionidae)	Sawtoothed grain beetle	Rice, Barley, Maize, Wheat, Sorghum
Tribolium castaneum (Herbst, 1797) (Coleoptera: Tenebrionidae)	Red flour beetle	Rice, Oats, Soybean Meal, Maize, Wheat
Lepidoptera		
Ephestia kuehniella Zeller, 1879 (Lepidoptera: Pyralidae)	Mediterranean flour moth	Maize, Rice, Wheat Flour, Bran, Cornmeal
Plodia interpunctella (Hübner, 1813) (Lepidoptera: Pyralidae)	Indianmeal moth	Rice, Maize, Wheat, Beans, Tobacco, Soybeans
Sitotroga cerealella (Olivier, 1789) (Lepidoptera: Gelechiidae)	Angoumois grain moth	Rice, Barley, Rice, Maize, Wheat

Table 2. Classification, organic function, chemical and commercial name of the pheromones identified in the cited literature.

Name / Chemical structure	Commercial name	Organic function	References
Aggregation pheromone			
1-Nonanol; (Z)-3-noneol; (Z)-2-octenol; (s)-(+)-1-octen-3-ol; (R)-(-)-1-octen-3-ol	*	Alcohol	Pierce *et al.* (1989); Pierce, *et al.* (1991b); Chambers *et al.* (1990)
(4*R*,8*R*) dimethyldecanal	DMD	Aldehyde	Obeng-Ofori and Coaker.,(1990a); Barak and Burkholder (1985); Lindgren *et al.* (1985); Bloch Qazi *et al.* (1998); Hawkin *et al.* (2011); Duehl *et al.* (2011); Obeng-Ofori and Coaker., (1990b); Loschiaho *et al.* (1986); Dissanayaka,*et al.* (2020c); Phillips and Doud,(2020); Sajeewani *et al.* (2020); Dissanayaka *et al.* (2020a); Dissanayaka *et al*, (2020b); Sammani *et al.*. (2020a); Rajan *et al.* (2018); Daglish *et al.* (2017); Ridley *et al.* (2016); Jittanun and Chongrattanameteekul ,(2014); Buckman *et al.* (2013); Arthur *et al.* (2014); Campbell, (2012); Toews *et al.* (2009); Larson *et al.* (2008); Small, (2007); Campbell, (2013); McKay *et al.* (2019)
(4*S*, 5*R*)-5-Hydroxy-4-methyl-3-heptanone	Sitophynone	Ketone	Wakefield *et al.* (2004); Trematerra and Girgenti (1989); Walgenbach *et al.* (1986); Phillips and Throne ,(2010); Walgenbach *et al.* (1983); Likhayo and Hodges,(2000); Carvalho *et al.* (2013); Athanassiou *et al.* (2006); Tang *et al.* (2009)
4-(p-acetoxyphenyl)-butan-2-one	*	Ketone	Perez *et al.* (2020)
(*Z,Z*)-5,8-tetradecadien-13-olide; (*Z, Z*)-3,6-dodecadien-11-olide; 3Z,6Z-dodecadien-12-olide	*	Lactone	Pierce *et al.* (1987)
(*S*)-(+)-1-methylbutyl-E-2-methyl-2-pentenoate; (2*S*, 3*R*)-ethylpropyl-2-methyl-3-hydroxypentanoate	(Dominicalure-1 [DL-1]) e (Dominicalure-2 [DL-2])	Ester	Sammani *et al.*. (2020a); Dissanayaka *et al.* (2020c); Dissanayaka *et al.* (2020a); Rajan *et al.* (2018);Daglish *et al.* (2017); Dowdy *et al.* (1993); McKay *et al.* (2017); Toews *et al.* (2006); Mahroof *et al.* (2010)
1-ethylpropyl (2*S*, 3*R*)- 2-methyl-3-hydroxypentanoate	Sitophyllate	Ester	Likhayo and Hodges ,(2000); Athanassiou *et al.* (2006); Chambers *et al.* (1996)
Isopropyl (2*E*,4*E*)-2,4-heptadienoate; 1-methyl (*E*)-2-methyl-2-heptenoate	Trunk-call e Trunc-call II	Ester	Obeng-Ofori and Coaker, (1990a); Ramírez-Martínez *et al.* (1994); Smith *et al.* (1999); Fadamiro *et al.* (1996); Fadamiro *et al.* (1998); Omondi *et al.* (2011); Cork *et al.* (1991)
(*Z*)-3-dodecen-11-olide; 4,8-dimethyl, *E, E*-4,8-decadienolide	(Ferrulactona I) e (Ferrulactona II)	Lactones	Chambers *et al.* (1990); Holloway *et al.* (2018); Losey *et al.* (2019)
(2*Z*-6E)-7-ethyl-33,11-dimethyl-2,6,10-dodecatriene	*	Unsaturated hydrocarbon	Chiluwal *et al.* (2018)
Sex pheromone			
Octadecanal	*	Aldehyde	Vuts *et al.* (2015)
(*Z*)-14-methyl-8-hexadecanal	Trogodermal	Aldehyde	Morrison *et al.* (2020); Castañé *et al.* (2020); Larson *et al.* (2008); Arthur *et al.* (2014); McKay *et al.* (2017)
(4*S*, 6*S*, 7*S*)-7-hdroxy-4,6-dimethyl-3-nonanone	Serricornine	Ketone	Fardisi and Mason, (2013); Perez *et al.* (2020); Arthur *et al.* (2014); McKay *et al.* (2017); Larson *et al.* (2008)
(*Z, E*)-9,12-tetradecadienayl acetatate	ZETA OU TDA	Ester	Mullen and Dowdy, (2001); Trematerra *et al.* (2011); Sambaraju and Phillips ,(2008); McKay *et al.* (2017); Campos *et al.* (2013); Campos *et al.* (2014); Trematerra *et al.* (2013); Small, (2007); Perez *et al.* (2020)
(2*Z*, 6*E*)-7-ethyl-3,11-dimethyl-2,6,10-dodecatrienal	Homofarnesal	Aldehyde	Chiluwal *et al.* (2017)
R, *E*- methyl-2, 4,5-tetradacatrienoate; methyl (2*E*, 4*E*, 7*Z*)- 2,4,7- decatrienoate	*	Ester	Vuts *et al.* (2015)
(3*E*, 6Z)- 3,7,11-trimethyldodeca-1,3,6,10-tetraene	α-Farnesene	Unsaturated hydrocarbons	Vuts *et al.* (2015)

*Not informed in the articles.

Research on pheromone traps, both in pest control and monitoring, has been growing since the 1990s and has helped to minimize the use of synthetic insecticides. Pheromone traps can be applied to detect insect presence, population density and flight activity. The benefits of pheromones include their ability to generate an alert of the incidence of the insect and its distribution in both area and time (Zarbin *et al.,* 2009). The most used types of pheromones in control or monitoring are aggregation and sexual pheromones. The prevalence of these two groups of semiochemicals is explained by their being the most frequent in stored grain pests and, consequently, the most studied, in addition to being efficient in pest monitoring and control strategies (Moreira *et al.,* 2005). Among the compounds most cited in research (Fig. 3) is (*R, R*)-4,8-dimethyldecanal, an aggregation pheromone used to control *T. castaneum* and *R. dominica*. Other aggregation pheromones are (*S*)(+) -(*E*)-2-methyl-2-pentenoate of 1-methyl butyl and (*S*)(+)(*E*)-2,4-dimethyl-2-pentenoate of 1- methyl butyl, applied to control or monitor *R. dominica*. One of the leading sex pheromones is o (*Z, E*)-9,12-tetradecadienyl acetate, used to monitor and control the Indianmeal moth*, P. interpunctella*. Table 2 shows the pheromones with their chemical structure, IUPAC and commercial names and chemical function, and (Fig. 3) shows the citation percentages of pheromones in the consulted literature.

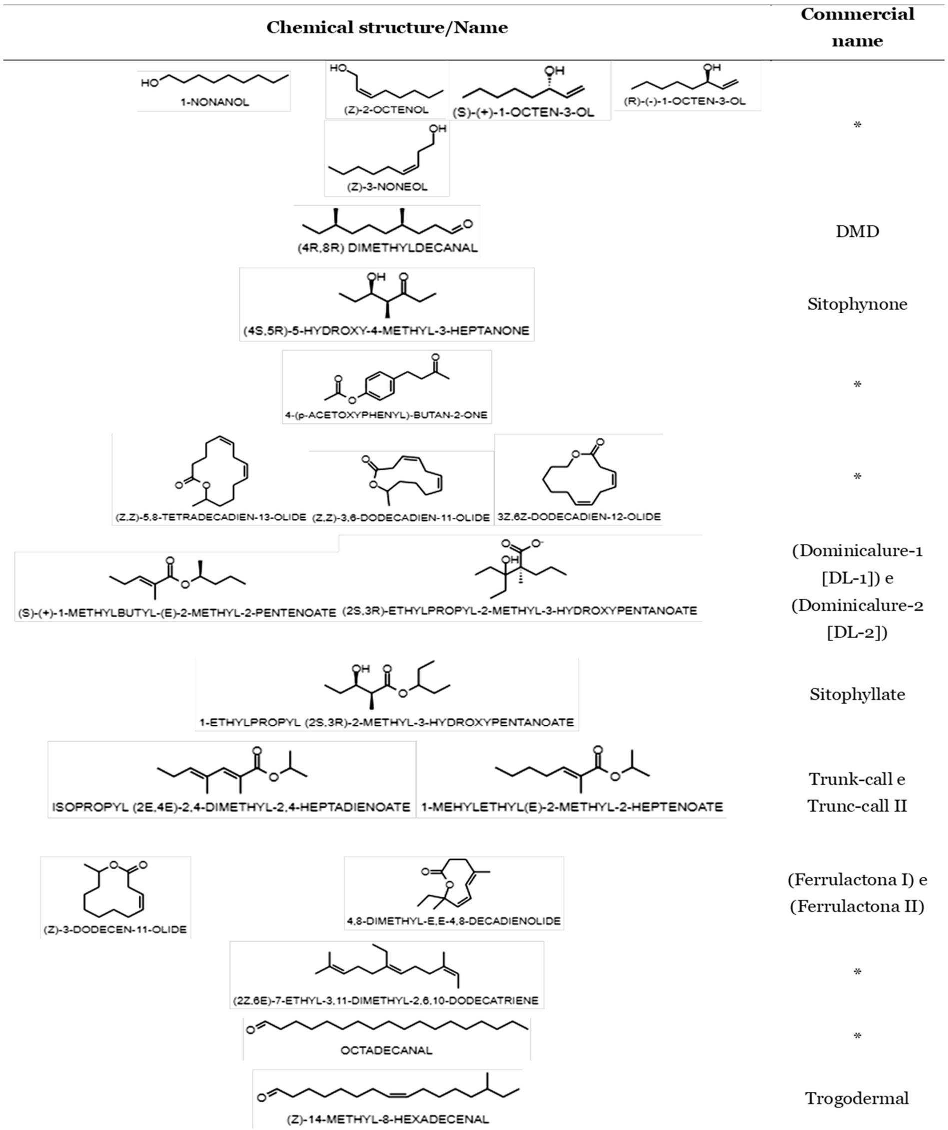


Fig. 2. Chemical structure and trade name of identified pheromones in the consulted literature for stored grains pests. *Not informed in the articles.

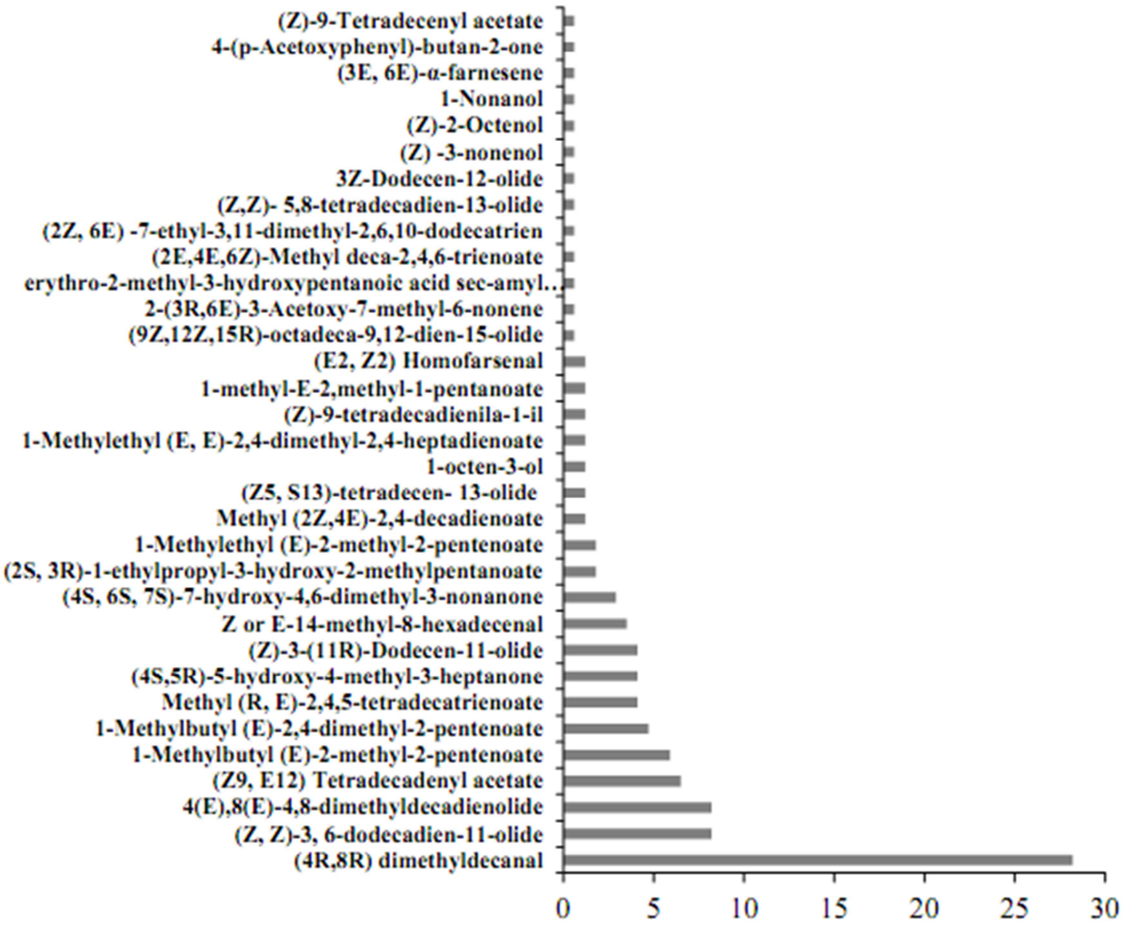


Fig. 3. Citation index of compounds identified as pheromones in the consulted literature.

Research with pests in stored grains - historical evolution

The database search strategy resulted in 715 articles. In the screening, 30 duplicate articles were excluded. After analysing the title, keywords and abstracts, 287 articles were excluded for failing to meet the inclusion criteria. The 398 remaining articles were read in total, which resulted in the exclusion of another 289 studies, including bibliographic reviews and research that presented only the synthesis of stored grain pest pheromones but still needed to demonstrate their application. In total, 109 manuscripts were included in the systematic review (Fig. 4)

There has been a significant increase in the number of studies published since the 1970s (Fig. 3). The number of publications demonstrates an expansion of research on stored grain pests. The first identified and isolated insect pheromone was the sex pheromone released by the moth *Bombyx mori* L., 1758 (Lepidoptera: Bombycidae) (Butenandt *et al.,* 1961). The first study on stored grain pests was published in France in 1974 by Burkholder and Boush (1974) and referred to the use of pheromones to capture insects of the genera *Attagenus*, *Trogoderma, Anthrenus* and *Lasioderma.*

Studies on pheromones for various stored product pests have been identified and are included in the synthesis. In the last 50 years, several pheromones have become commercially available, and they are used for monitoring and detecting stored grain pests and control (Phillips and Throne, 2010). Although pheromones of some species have been identified since the 1970s, for example, the lack of commercially available products for their control, the search for healthy foods and care for the environment mitigated the search for new pheromones for the application of pheromones already determined for some species.

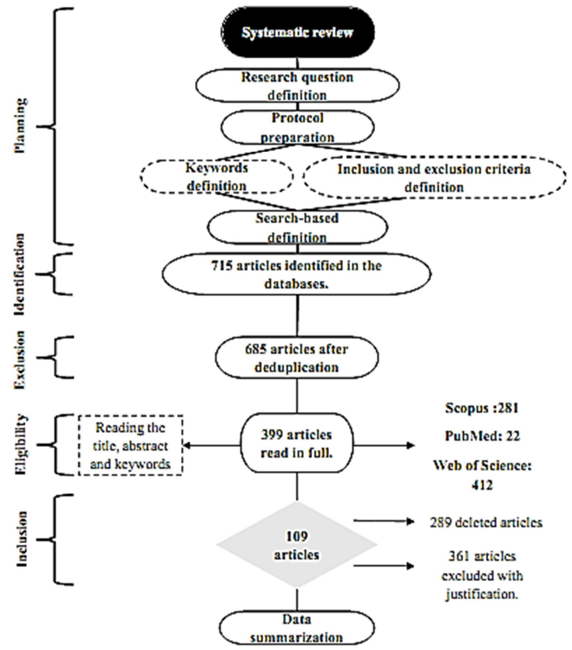


Fig. 4. Data summary

After the description of the first pheromones, the number of publications remained low until the 1980s, when the search for the control of these insects increased and continued over time, always aiming to investigate the possible pheromones of the different species of insect pests of stored grains, highlighting among them *S. granarius* (L., 1758), *S. oryzae* (L., 1763), *S. zeamais* and *T. castaneum*. Scientists were always looking to control these insects through behaviour to reduce the presence of insecticides in food, whether for human or animal use. The aggregation pheromone produced by males was identified in a study on *S. zeamais*. This same compound was determined for two other species of the same genus, *S. granarius* and *S. oryzae* (Burkholder and Boush, 1974; Faustini, 1982; Obeng-Ofori and Coaker, 1990a).

The identified aggregation pheromones were used to monitor and detect beetles in stored products and grains (Barak and Burkholder, 1985). Since the 1990s, as shown in (Fig. 5), the number of publications has increased significantly, thus indicating that research on insect behaviour to monitor and control of numerous species has important implications for the integrated management of stored grain pests.

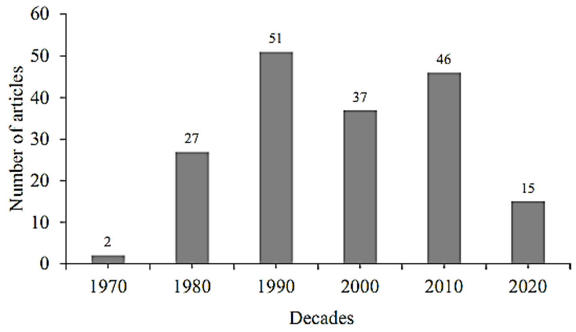


Fig. 5. Time trend of pheromone research of stored product pests over the last 50 years.

Geographic distribution of studies

Research studies were carried out in 27 countries (Fig. 6) distributed across six continents, with 37.42% of these studies originating in North American countries, mainly in the United States (n = 50) and Canada (n = 12). A total of 28.91% of the studies were carried out in 11 countries on the European continent, with most studies concentrated in the United Kingdom (n = 20), followed by Italy (n = 6). Already 14.05% of the studies originated from the Asian continent, highlighting studies carried out in Japan (n = 22). About 2.79% were carried out in South America, 3.91% in Oceania and 2.23% in Africa. Brazil, which has a humid tropical climate, is the largest grain exporter and the second largest producer but has minimal participation in these studies, highlighting the need to invest in this area to protect crops. Insects associated with stored agricultural products cause numerous direct and indirect losses (Hagstrum and Subramanyam, 2006). Quantitative losses in the harvested crop due to damage caused by these insects vary with the geographic area: 10% in temperate countries and 50% in the humid tropics (Wijayaratne *et al.*, 2018). These insect losses occur in different magnitudes during post-harvest practices. As cereal production is seasonal, harvested production must be stocked to meet demand during the off-season (Dowell and Dowell, 2017). About 20-40% of post-harvest losses occur during field and post-harvest operations; among these losses, 55% occur during storage. The worldwide damage to food grains from insect infestation is estimated to be 10-40% per year (de Souza *et al.*, 2016) (de Souza *et al.*, 2016). In India, storage losses for cereals were around

0.75-1.21%, while losses for pulses and oilseeds were 1.18-1.67% and 0.22-1.61%, respectively (Jha *et al.,* 2015). In Asia, about 6% of total post-harvest losses are due to inadequate storage facilities, of which insects and fungi account for half (3%) of storage losses (Sharon *et al.,* 2014).

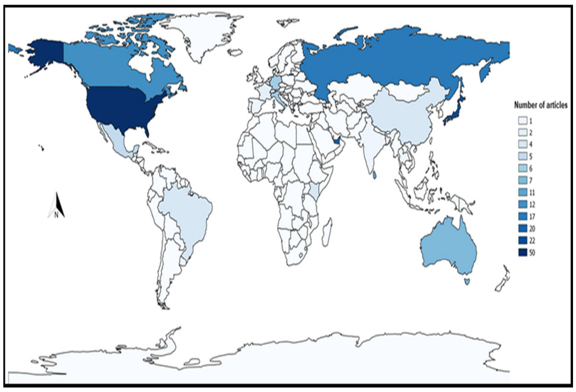


Fig. 6. Geographic distribution of research on pest management of stored grains.

In Sri Lanka, post-harvest losses of paddy rice amount to approximately 15%, including a storage loss of 4-6% (Palipane, 2000; Wijayaratne *et al.,* 2009). Surplus production of post-harvest rice in Sri Lanka is subject to severe insect infestations, necessitating appropriate pest management methods during grain storage (Dissanayaka *et al.,* 2018a). In Brazil, estimates of losses caused by insect attacks on the 35 leading crops vary between 2% and 43%. It is estimated that, on average, insects cause losses of 7.7% in these crops, causing significant annual damage to the Brazilian economy, despite the adoption of control measures (Oliveira *et al.,* 2014). Massive losses while storing durable agricultural commodities in different geographic regions are a critical challenge to global food security (Kumari *et al.,* 2020). Insect damage is not limited to weight and nutritional loss but includes monitoring and management costs. In a world scenario, some cultures are highlighted, but due to biotic factors, such as insect pests, the quality of a given product will not be the same. These insects are primarily polyphagous and can be essential pests for stored products and the culture itself. Contamination can come from the field of production. Some insect pests can attack about 69 different products worldwide (Hagstrum and Subramanyam, 2009); generally, the exempt ones acquired resistance to most of the used insecticides (Konemann *et al.,* 2017). This causes most affected countries to look for new methods to use in their control to promote the early detection of certain insect pest species in these products. One of the main grains of importance for the world scenario is the maize *Zea mays* L. (Poaceae). Over the last few decades, this cereal has become the largest agricultural crop in the world, in addition to being relevant in terms of food safety and human and animal nutrition. It is possible to use maize to produce many products, such as fuels, beverages and biodegradable polymers, including natural polymers such as chitosan, cellulose and collagen (Contini *et al.,* 2019; Tabasum *et al.,* 2019).

Maize and rice *Oryza sativa* L. (Poaceae) are important cereals with high nutritional value and potential for energy conversion (biofuels), thus promoting food security and clean energy production worldwide. Brazil is the largest corn exporter and the world's largest producer of soybeans, *Glycine max* (L.) Merr. (Fabaceae) and is responsible for 50% of the world market for this grain. Soybean is the most cultivated oilseed in the world, and production is expanding in Brazil (EMBRAPA, 2021). One of the leading agricultural products sold abroad is soy. Since the 1990s, its global demand has increased by 145% worldwide, and it is projected to increase by 70-80 million metric tons annually over the next ten years. Argentina, Brazil, and the United States are the main actors in global soy production; together, they accounted for 80.5% of world production between 2012 and 2018 and 86.2% of exports during that period (FAO, 2021). Massive losses that occur during the storage of agricultural products constitute a significant challenge for world food security. A common feature of the work carried out in each country is that they are related to beetles of the genus *Tribolium,* whose insects are considered the main pests of many food products. They have a devastating action, are voracious and attack different agricultural commodities, both raw and processed, stored after harvest and coexist in different habitats of the post-harvest supply chain (Kumari *et al.,* 2020).

Due to the growing environmental concerns caused by insecticides, many countries seek new monitoring and control methods (Wijayaratne *et al.,* 2018). Among these new methods, synthetic pheromones gained space and are now widely used in monitoring programs to determine these insects' presence and population density in stored products (Hagstrum *et al.,* 2012). Pheromone bait traps are the most used method in many countries (Campbell, 2012). There are many traps developed to monitor the presence of these insects, and one of the most used methods in the U.S. and other countries, which are always looking for the most convenient model for each situation, is the so-called dome trap (Phillips and Throne, 2010).

The dome trap is widely used for integrated pest management of meal beetles and other stored product insects in many countries' grain and food industries. This type of trap monitors both flying and non-flying insects simultaneously. The trap uses the pheromone produced by males of *T. castaneum*. The pheromone 4,8-dimethyldecanal (DMD) is released in an impregnated rubber septum and a food-based oil as attractants. The exact composition of the cooking oil sold with the trap is proprietary information, but it contains a grain-based cooking oil such as wheat germ oil. Food volatiles can be significant attractants. Many products have been evaluated as kairomones for stored-product insect pests, such as crude extracts or specific isolated compounds present in these foods (Collins *et al.,* 2007). The so-called wheat weevil *S. granarius* is by far the most studied stored product insect pest in terms of its response to kairomones. This species responds to whole or crushed seeds of rice, wheat, corn and oats (Wakefield *et al.,* 2004). Combining food odours with pheromones can increase the capture of insects belonging to Sitophilus, an effect observed in behavioural bioassays and traps (Wakefield *et al.,* 2004). Assessment of the attraction of *T. castaneum* to food volatiles has been more limited, although most commercial monitoring uses a combination of pheromone and kairomone.

However, not all cooking oils have a pleasing effect on stored product insects. Specific cooking oil may have different levels of attraction (and even repellence) for other species of these insects.

Several applications of pheromones and kairomones in managing *T. castaneum* have been extensively researched (Dissanayaka *et al.*, 2020a). The simultaneous use of pheromones with commercially available kairomone solution increases the percentage of the capture of *T. castaneum* (Doud *et al.,* 2021; Phillips and Doud, 2020). In addition, the combined use of 4.8 DMD with *Cocos nucifera L.* (Arecaceae) coconut oil and *Azadirachta indica* A. Juss neem oil (Meliaceae) increases the capture of *T. castaneum* (Dissanayaka *et al.,* 2018a); this effect was recently observed with other species, where the sex pheromone (*Z,E*)-9,12-tetradecadienyl acetate (ZETA) combined with neem oil increases the successful mating cycle of *Cadra cautella* (Walker, 1863) (Lepidoptera: Pyralidae).

Thus, the effect of fats, especially neem, has been shown to improve the response of adults of some adult insects, such as *T. castaneum*, when added to its aggregation pheromone 4.8 DMD, but this effect still needs further observation (Bandara, Dissanayaka, Wijayaratne, and Morrison, 2020: Sammani *et al.,* 2020b). With the evolution of insecticide resistance and concern about the risks posed by insecticides to human health and the environment (Bandara *et al.,* 2021), new research has been developed that aims to obtain more friendly methods and more innovative ways to control insects in the sector storage in several countries such as the United States, United Kingdom, Japan, Russia and Canada such as behaviour control. Considering all insect pest genera presented in the 22 countries studied, the species addressed in the articles in more significant numbers belong to the genus *Tribolium,* with 48.43% of the species. The beetle *T. castaneum* was the most studied insect, with 34.08% of the articles, followed by *R. dominica* and *P. interpuctella* with 9.50% (Table 3).

Table 3. Species observed in the analysed articles.

Family	Genera	Species	Articles	Grain or derivatives
Anobiidae	*Lasioderma*	*L. serricorne*	7	Peanut and Rice Wheat flour
	Stegobium	*S. paniceum (L., 1758)*	2	Wheat flour
Bostrichidae	*Prostephanus*	*P. truncatus* (Horn, 1878)	10	Maize
	Rhyzopertha	*R. dominica*	16	Wheat Rice
Chrysomelidae	*Acanthoscelides*	*A. obtectus* (*Say, 1831)*	1	Bean
	Calosobruchus	*C. chinensis* (*L., 1758)*	3	Bean
Cleridae	*Necrobia*	*N. rufipes* (*De Geer, 1775)*	1	Uninformed
Curculionidae	*Sitona*	*S. lineatus* (*L., 1758)*	1	Uninformed
	Sitophilus	*S. granaries*	5	Wheat and Rice
		S. oryzae	13	Rice Wheat Wheat flour and Yeast
		S. ssp.	1	Uninformed
		S. zeamais	12	Wheat Maize Rice
Dermestidae	*Anthrenus*	*A.* sp.	1	Uninformed
	Attagenus	*A. brunneus Faldermann, 1835*	1	Uninformed
	Trogoderma	*T. granarium Everts, 1898*	1	Rice and Wheat
		T. variabile Ballion, 1878	8	Wheat Rice Wheat flour
Gelechiidae	*Sitotroga*	*S. cerealella*	1	Maize
Histeridae	*Teretrius*	*T. nigrescens* (*Lewis, 1891)*	1	Maize
Laemophloeidae	*Cryptolestes*	*C. ferrugineus*	9	Wheat Silo grains
		C. pusillus (*Schénherr, 1817)*	2	Uninformed
Mycetophagidae	*Typhaea*	*T. stercorea* (*L., 1758).*	2	Peanut Wheat flour
Nitidulidae	*Carpophilus*	*C. hemipterus* (*L., 1758)*	1	Peanut
Pyralidae	*Cadra*	*C. cautella*	4	Peanut Almond Rice flour and Bean
	Ephestia	*E. cautella*	1	Wheat
		E. elutella (*Hübner, 1796)*	1	Uninformed
		E. kuehniella	3	Wheat
	Plodia	*P. interpunctella*	15	Wheat Peanut Rice
Silvanidae	*Ahasverus*	*A. advena* (*Walt, 1832)*	2	Oat Wheat flour
	Cathartus	*C. quadricollis* (*Guérin-Méneville, 1844)*	1	Uninformed
	Oryzaephilus	*O. mercator* (*Fauvel, 1889)*	5	Oat
Tenebrionidae	*Gnatocerus*	G. *cornutus* (F., 1798)	8	Wheat flour
	Tribolium	*T. castaneum*	42	Wheat flour
		T. confusum (*Duval, 1868)*	12	Wheat Wheat flour e Yeast
		T. destructor Uyttenboogaart, 1934	1	Wheat flour

Methodological approaches adopted in the analysed works

Pheromones have been increasingly important in pest control in agriculture; several works describe their use in monitoring and controlling stored grain pests. However, it is possible to determine the compounds' efficiencies through laboratory tests with insects (bioassays) and field tests (analysis of insect response at the storage site). Of most of the 109 articles, 70 were carried out in the laboratory, while 58 described field studies (Table 4).

To investigate the effects of pheromones on pest control, it is necessary to carry out bioassays; of the 109 articles analysed, 45 presented the type of behavioural bioassay indicated in Table 5.

Table 4. Total number and percentage of works that presented laboratory and field approaches.

Study location	Total Number	Percentage (%)
Laboratory	56	51.8
Field	50	46.2

For this system, two airflow sources merge at a decision point (Y tube) to form a blade flow down the corridor to an initial area where the insects or larvae are placed at the beginning of an experiment (Stevenson *et al.,* 2017). The Yolfactometer is the most used to test the bioactivity of the insect against the semiochemicals.

Table 5. Total number and percentage of bioassays performed in the analysed studies.

Bioassay in olfactometry	Total Number	(%)	Reference
Petri dish arena	1	0.92	Bloch Qazi *et al.* (1998)
Arena	10	9.2	Awater-Salendo *et al.* (2020); Athanassiou *et al.* (2006); Smith *et al.* (1999); Wakefield *et al.* (2004); Phillips *et al.* (1993); Dowdy *et al.* (1993); Trematerra and Girgenti, (1989); Lindgren *et al.* (1985); Hodges *et al.* (1983); Athanassiou *et al.* (2006) ; Jittanun and Chongrattanameteekul, (2014); Walgenbach *et al.* (1986)
Y-olfactometer	15	21.2	Pierce *et al.* (1987), Edde *et al.* (2005); Pierce *et al.* (1991a); Pierce *et al.* (1989); Gerken *et al.* (2018); Athanassiou *et al.* (2006); Faustini *et al.* (1981) McKay *et al.*(2017); Vuts *et al.* (2015); Tang *et al.*(2009); Smith *et al.* (1999) Phillips *et al.* (1993);Barak and Burkholder, (1985); Losey *et al.* (2019) Dissanayaka *et al.* (2018b)
semi-field-controlled climate	5	5.5	Phillips and Doud, (2020) Dissanayaka *et al.* (2018b); Gerken *et al.* (2018); Sambaraju and (Phillips, 2008); Wakefield *et al.* (2004)
Wind tunnel tests	3	5.5	Fadamiro *et al.* (1998); Gerken *et al.,* 2018); Campos and Phillips, (2013)
Electroantennography (EAG)	1	1.8	Chambers *et al.* (1990)

Most studies carried out the olfactometer test in the laboratory, which is quite common as it allows us to ascertain the "preference" of insects for one or more options (treatments and controls). Olfactometry is a technique used to study how a given semiochemical affects the behaviour or physiology of the insect. The primary function of olfactometry is to produce qualitative or quantitative data, depending on the question, to be answered quickly, efficiently and accurately. Most of this study uses olfactometers, which are commercially available and have the same operating principle and can and should be adapted to the insect tested (Eiras and Mafra-Neto, 2001). Olfactometers have the same elements: a chamber for inserting the tested insects, an area for inserting the chemical compound (or odour source) and a reading area (the section where insects fly or walk if attracted by the chemical compound). The data are generated through bioassays classified into (1) indiscriminate bioassays that evaluate the attraction or not of the insect to the odour source and (2) discriminant bioassays evaluating the entire behaviour of the insect to the odour source, observing behavioural changes even when the insect does not reach the olfactometer reading area (Eiras and Mafra-Neto, 2001). The development of different types of traps for sampling insects from stored products, along with progress in the identification and synthesis of pheromones and attractants for the main insect species, has been the subject of much research in the last two decades. In addition, the demand for products without insect contamination makes traps for the early detection of insects indispensable tools to keep grains and their products free from damage or loss (Barak *et al.,* 1990). Several traps are efficient at controlling and monitoring stored grain pests; of the 109 articles analysed, 81 showed the types of traps.

Stored product pest insect traps fall into three categories: aerial insect traps, including sticky and funnel traps, surface traps to capture insects while walking using pheromones or food attractants and lures used directly on the grain mass, such as the loss of the glue-type trap (Barak *et al.,* 1990). These categories become less distinct as traps are used for species other than those they were designed for. Two types of traps were used the most frequently, pitfall and dome traps. Pitfall traps are commonly used to estimate the relative abundance of terrestrial arthropods and have been used in many ecosystems. These traps are the most used method to capture invertebrates. Pitfall traps are popular because they are more affordable, cost-effective, and relatively simple to build and deploy. The fundamental design of the pitfall trap consists of a container buried in the ground with the top flush with the surface. The simplicity of this method attracts many wildlife researchers who want to investigate patterns of abundance and monitor arthropods.

Pest control and monitoring activities-approaches adopted in the studies

The management of stored grain pests is a set of monitoring and control techniques to maintain high-quality grains. (Fig. 7) and (Fig. 8) show the number and percentage of articles that controlled and monitored insect pests of stored grains in the analysed reports. There is a much greater prevalence of articles that showed the monitoring of stored grain pests as an object of study, a total of 93 articles. On the other hand, we observed a total of 12 articles that presented preventive control as an essential step towards the success of an integrated pest management program in stored grains.

To implement an effective integrated management program with reduced infestation potential, the management of the storage unit must be aware of the importance of the influence of ecological factors, such as temperature, grain moisture content, the relative humidity of the environment and the storage period involved in the system. In the same way, the choice of cultivar, the harvesting process, reception and cleaning, grain drying, aeration and refrigeration are also essential factors for the preventive control of stored grain pests. Several articles presented studies on pest control with different compound and grain types, as seen in Table 7.

Once stored, the grains must be monitored throughout their storage period. Tracking the evolution of pests that occur in the mass of stored grains is of fundamental importance, as it allows for detecting the beginning of infestations that may alter the final quality of the grain. This monitoring is based on an efficient pest sampling system, such as using fixed traps to capture insects or sieves with a mesh size of not less than 20 mm, and the measurement of variables such as grain temperature and humidity, which influence the conservation of the stored product. It allows researchers to record the beginning of the infestation and direct the decision-making by the storer to guarantee the quality of the grain (Lorini *et al.,* 2015). Several articles that monitored several pests, with their respective pheromones in different types of grains, are listed in Table 8.

Table 6. Total number and percentage of traps used.

Trap Type	Total Number	(%)	References
Pitfall traps	30	29.6	Bloch Qazi *et al.* (1998); Hodges *et al.,* 1998); Fadamiro *et al.* (1998); Tigar *et al.* (1994); Fargo *et al.* (1994); Perez *et al.* (2020); Stevens *et al.* (2019); McKay *et al.* (2019); McKay *et al.* (2017); Wong-Corral *et al.* (2001); Obeng-Ofori and Coaker,(1990a); Loschiaho *et al.* (1986); Dissanayaka *et al.* (2018b); Holloway *et al.* (2018); Rajan *et al.* (2018); Daglish *et al.* (2017); Ridley *et al.* (2016); Arthur *et al.* (2014); Carvalho *et al.* (2013); Likhayo and Hodges ,(2000); Athanassiou *et al.* (2006); Edde *et al.* (2006); Edde *et al.* (2005); Wakefield *et al.* (2004); Campbell and Arbogast, (2004); Birkinshaw *et al.* (2004); Wong-Corral et al. (2001) ,(2001); Gerken *et al.* (2018); Campos *et al.* (2013); Losey *et al.* (2019)

Trap Type	Total Number	(%)	References
Dome	21	20.3	Dissanayaka *et al.* (2018a); Smith *et al.* (1999); Wakefield *et al.* (2004); Jittanun and Chongrattanameteekul, (2014); Vuts *et al.* (2015); Tang *et al.* (2009); Omondi *et al.* (2011); Toews *et al.* (2009); Larson *et al.* (2008); Small, (2007); Fedina *et al.* (2007); Toews *et al.* (2006); Hawkin *et al.* (2011); (Campbell and Mullen, (2004) Pierce *et al.* (1989); Pierce *et al.* (1987); Tang *et al.* (2009); Smith *et al.* (1999); Phillips *et al.* (1993)
Adhesive traps	9	8.3	Campbell *et al.* (2002); Dowdy *et al.* (1998), Chambers *et al.* (1996); Ramírez-Martínez *et al.* (1994), Boake, (1985); Campos *et al.* (2014), Campos *et al.* (2013); Trematerra *et al.,* 2013), Buckman *et al.* (2013)
Bucket traps with funnels	4	1.8	Obeng-Ofori and Coaker (1990b); Sambaraju and Phillips, (2008), Fadamiro *et al.* (1998); Faustini *et al.* (1981)
Panel trap	1	0.92	Bloch Qazi *et al.* (1998)

Table 7. Pest control studies on stored grains using pheromones.

Grain	Family	Plague species	Compounds	References
Rice	Pyralidae	*Cadra cautela*	(*Z,E*)-9,12-tetradecadienylacetate (ZETA) e (*Z*)-9-tetradecadien-1-yl acetate (ZTA)	Sammani *et al.*. (2020a)
	Bostrichidae	*Rhyzopertha dominica*	(*S*) - (+) - 1-metilbutil- (*E*) -2-metil-2-pentenoato (DL-1) e (*S*) - (+) - 1-metilbutil- (*E*) -2,4-dimetil-2-pentenoato (DL-2)	Dissanayaka *et al.* (2020d)
	Tenebrionidae	*Tribolium castaneum*	4,8-dimethyldecanal	Dissanayaka *et al.* (2020a)
Oat	Silvanidae	*Oryzaephilus surinamensis* *Oryzaephilus mercator*	1-octen-3-ol	Pierce *et al.* (1989)
		Oryzaephilus surinamensis	(*R, S*)-(*Z,Z*)-3,6-dodecadien-11-olide (lactone II); (*Z,Z*)-3,6 dodecadienolide (lactone III) e (*R,S*)-(*Z,Z*)- 5,8-tetradecadien-13-olide (lactone IV)	White and Chambers, (1989),White, Chambers, *et al.* (1989)
Bean	Chrysomelidae	*Callosobruchus chinensis* (L., 1758)	2E-: 2Z-homofarnesal	Chiluwal *et al.* (2017)
Wheat	Tenebrionidae	*Tribolium castaneum*	Dimethyldecanal (DMD) e dominicalure 1 e 2	Stevenson *et al.* (2017)
	Bostrichidae	*Rhyzopertha dominica*	Dominicalure-1 ((*S*)-1-Methylbutyl (*E*)-2,4-dimethyl-2-pentenoate) Dominicalure-2 ((*S*)-1-Methylbutyl (*E*)-2-methyl-2-pentenoate)	Edde *et al.* (2005)
	Curculionidae	*Sitophilus granarius* *Sitophilus oryzae* *Sitophilus zeamais*	4*S*, 5*R*-sitophinone	Trematerra and Girgenti, (1989)

Table 8. Monitoring of stored grain pests with pheromones.

Grain Type	Family	Species Prague	Compound	Reference
Rice	Pteromalidae	*Lariophagus distinguendus* *Theocolax elegans*	Not specified	Tang *et al.* (2016)
	Curculionidae	*Sitophilus granarius*	Sitophyllate	Chambers *et al.* (1996)
Oat	Silvanidae	*Ahasverus advena* *Oryzaephilus surinamensis*	3-Octanol (racemico) and 3-octanona	Pierce, *et al.* (1991a)
	Silvanidae	*C. quadricollis*		
	Laemophoeidae	*Cryptolestes ferrugineus*		
Pea and Beans	Bostrichidae	*Rhyzopertha dominica*	Dominicalure-1 (*S*)-1-Methylbutyl (*E*)-2,4-dimethyl-2-pentenoate) Dominicalure-2 (S)-1-Methylbutyl (*E*)-2-methyl-2-pentenoate)	Edde *et al.* (2006); Selitskaya and Shamshev(1995)
	Curculionidae	*Sitona lineatus*	4-methyl-3,5-heptanedione	Smart *et al.* (1994)
Maize	Bostrichidae	Prostephanus *truncutus* (Horn, 1878)	Trun-call 1 (1-Methylethyl (*E*)-2-methyl-2-pentenoate) Trun-call 2 (2 1-Methylethyl (*E,E*)-2,4-dimethyl-2,4-heptadienoate)	Wong-Corral et al. (2001) Muatinte *et al.* (2018)
	Tenebrionidae	*Tribolium confusum*	4,8-dimethyldecanal	Gerken *et al.* (2018)
	Curculionidae	*Sitophilus oryzae*	Sitophilure	Likhayo and Hodges, (2000)
Wheat	Bostrichidae	*Rhyzopertha dominica*	Dominicalure-1 (*S*)-1-Methylbutyl (*E*)-2,4-dimethyl-2-pentenoate) Dominicalure-2 (*S*)-1-Methylbutyl (*E*)-2-methyl-2-pentenoate)	Mahroof *et al.* (2010)
	Mycetophagidae	*Typhaea stercorea*	4,8-dimethyldecanal	Phillips and Doud, (2020)
	Silvanidae	*Ahasverus advena*		
	Bostrichidae	*Oryzaephilus surinamensis*	Not specified	Arthur *et al.* (2014)
	Laemophloeidae	*Cryptolestes ferrugineus*	Cucujolide I e Cucujolide II	Losey *et al.* (2019)
	Anobiidae	*Lasioderma serricorne*	Not specified	Fardisi and Mason ,(2013)
	Dermestidae	*Trogoderma variable*	Not specified	Arthur *et al.* (2014)
	Pyralidae	*Cadra cautella*	(ZETA) *Z, E*)-9,12-tetradecadienyl acetate (ZTA) (*Z*)-9-tetradecadien-1-yl acetate	Trematerra *et al.* (2011)

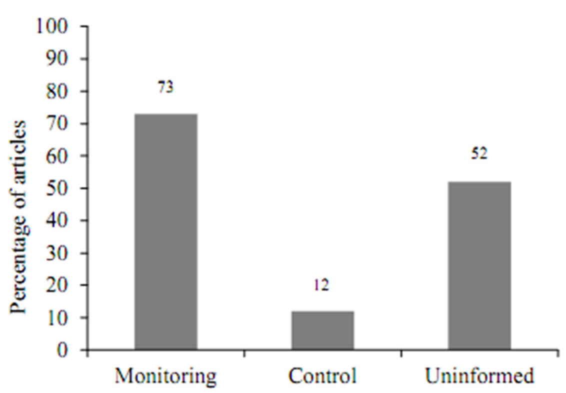


Fig. 7. Number of articles with monitoring and control studies of stored grain pests.

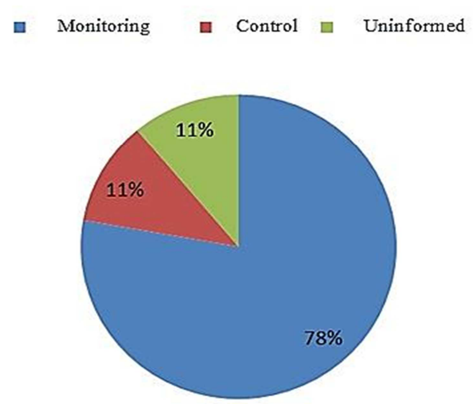


Fig. 8. Percentage of articles on monitoring and controlling stored grain pests.

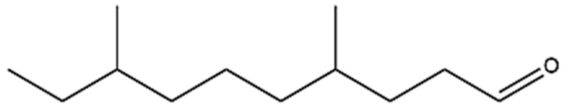
Synthesis of pheromones

Aggregation pheromone of T. confusum and T. castaneum

In the search for articles on the synthesis of stored grain pest pheromones, 15 references were found related to the synthesis of tribolure, an aggregation pheromone of *T. confusum* and *T. castaneum,* composed of a mixture of stereoisomers (4*R*,8*S*), (4*R*, 8*R*), (4*S*,8*S*) and (4*S*,8*R*) of 4,8-dimethyl decanal in a 4:4:1:1 ratio (Fig. 9). Here, we separate the synthetic routes into two groups. Group 1 includes the routes in which citronellic acid, or its derivatives, was used as starting material. Group 2 encompasses all the other routes; these departed from the most varied compounds. The two groups follow a chronological order of publication. These data are presented in Table 9.

Table 9. Pheromone syntheses of *T. confusum* and *T. castaneum* found in the literature from 1981 through 2015.

Pest insects	Products	Key step	Overall yield	Number of steps	Synthesis
		GROUP 1			
T. confusum e T. castaneum	4,8-dimethyldecanal	Wittig	4%*	5	Suzuki, (1981)
		Alkylation and Grignard reaction	54%*	3	Zarbin *et al.* (1998)
			58%*	3	Santangelo *et al.* (2006)
		Alkylation	25%	6	Akasaka *et al.* (2011)
	(4*R*,8*R*)-4,8-dimethyldecanal	Alkylation	(4*S*, 8*R*)-: 15% e (4*S*, 8*S*)-: 18%	Ten and 10	Mori and Takikawa, (1991)
		Alkylation and Grignard	58%*	3	Santangelo *et al.* (2006)
		Alkylation	25%	6	Akasaka *et al.* (2011)
T. castaneum	4,8-dimethyldecanal	Kolbe's electrolysis	(4*R*, 8*R*)-: 8% e (4*R*, 8*S*)-: 10%	5 and 5	Mori *et al.* (1985)
		Wittig and Alkylation	(4*S*, 8*S*)-:8%* e (4*R*, 8*R*/4*R*,8*S*)-:0,9%	5 and 5	Suzuki *et al.* (1983)
		Kolbe's electrolysis, Alkylation and Grignard	(4*S*, 8*R*)-: 11%, (4*S*, 8*S*)-: 28%, (4*R*, 8*R*)-: 7,2% e (4*R*, 8*S*)-: 5,4%	7, 8, 6 and 6	Mori *et al.* (1983)
		GROUP 2			
T. castaneum	(4*R*,8*R*)-4,8-dimethyldecanal	Grignard	5,9%*	9	Fuganti *et al.* (1988)
		Wittig	(4*R*,8*R*)-: 15% e (4*R*,8*S*)-: 19%	9 and 9	Cheskis *et al.* (1988)
		Grignard	34%	7	Odinokov *et al.* (1989)
		Alkylation	15,5%	7	Odinokov *et al.* (1991)
T. confusum e T. castaneum	4,8-dimethyldecanal	Ozonolysis	34%*	7	Ismuratov *et al.* (2003)
		Conjugate addition	7%	11	Kameda and Nagano, (2006)
		Grignard	(4*R*, 8R/*S*)-: 30% e (4*S*, 8R/*S*)-: 67%	6 and 3	Wang *et al.* (2015)



4,8-dimethyldecanal

Fig. 9. *T. confusum* and *T. castaneum* aggregation pheromone.

The synthetic routes for preparing tribolure components have generally improved over the years by increasing global yields, execution, and the number of steps. Furthermore, chain growth reactions using Grignard and Wittig are common in these routes and are present in most key steps. For scale preparation, overly complex and expensive reactions are only sometimes attractive. Routes like those of Zarbin *et al.* (1998) and Santangelo *et al.* (2006) are just three steps and of little complexity, resulting in products with 54% and 58% global yields, respectively, they are good candidates for this purpose. From an environmental point of view, emphasis should be given to the work of Wang *et al.* (2015) They used compost derived from industrial waste as a starting material and reached the products through routes of 3 and 5 steps with overall yields of 67% and 30%, respectively.

Synthesis of sitophilure, aggregation pheromone of S. oryzae and S. zeamais and sitophilate aggregation pheromone of S. granaries

Six publications concerning the synthesis of *Sitophilus* pheromones were found. Three of them deal with the synthesis of (4*R*,5*S*)-5-hydroxy-4-methyl-3-heptanone (sitophilure), an aggregation pheromone of *S. oryzae* and *S. zeamais* (Fig. 10). The others deal with the synthesis of 1-ethylpropyl (2*S*,3*R*)-3-hydroxy-2-methyl pentanoate, known as sitophilate, an aggregation pheromone of *S. granarius* (Fig. 10). All were published between 1988 and 2013 and Table 10 summarises the syntheses of each pheromone in chronological order of publication.

For both sitophilure and sitophilate, routes that use chemoenzymatic steps are of great importance from a scalable point of view. Kalaitzakis *et al.* (2006) used this approach and in just two steps, obtained the highest yield described so far in the synthesis of sitophilure. In the same group, Kalaitzakis *et al.* (2007) prepared the sitophilate with a high yield in just four steps. Emphasis should be given to Ravía *et al.* (2013), who, in addition to using enzymes, also used microwave irradiation under solvent-free conditions in two of their five steps and obtained a high overall yield.

Table 10. Synthesis of *S. oryzae*, *S. zeamais* and *S. granarius* pheromones.

Pest insects	Products	Key step	Overall yield	Number of steps	Synthesis
Sitophilure					
S. oryzae e *S. zeamais*	(4*S*,5*R*)-5-hydroxy-4-methyl-3-heptanone	Grignard	16%	7	Mori *et al.* (1988)
		Microbiological reduction (*Saccharomyces cerevisiae*)	18%	12	Pilli and Riatto, (1999)
		Stereoselective enzymatic reduction and alkylation	81%	2	Kalaitzakis *et al.* (2006)
Sitophilato					
S. granarius	(2*S*,3*R*)- e (2*S*, 3*S*)-3-hydroxy-2-methylpentanoate	Beilis-Hillman condensation	35%	2	Cheskis *et al.* (1990a)
	(2*S*,3*R*)-3-hydroxy-2-methylpentanoate	Stereoselective enzymatic reduction and enzymatic hydrolysis	63%	4	Kalaitzakis *et al.* (2007)
		Alkylation; stereoselective enzyme reduction and Mitsunobu inversion,	50%	5	Ravía *et al.* (2013)

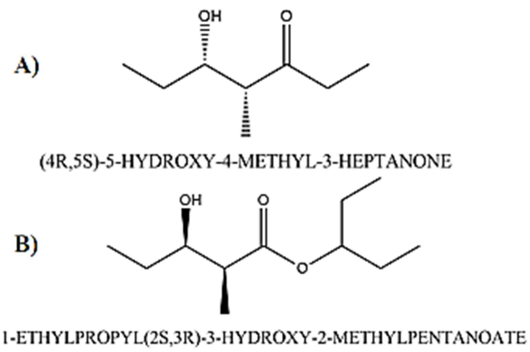


Fig. 10. A) Aggregation pheromone from *S. oryzae* and *S. zeamais*. B) *S. granaries* aggregation pheromone.

Synthesis of methyl (R)-(-)-(E)-2,4,5-tetradecatrienoate and methyl (2E,4Z)-2,4-decadienoate, aggregation pheromone from A. obtectus

For the aggregation pheromone of *A. obtectus*, eight articles were found that approached the acquirement of the molecule (*R*)-(-)-(*E*)-2,4,5- methyl tetradecatrienoate (Fig. 11) between the years 1981 and 2018. Investigations continued over the years, which led to the discovery of another attractive compound, methyl (2*E*,4*Z*)-2,4-decadienoate (Mori, 2012) (Mori, 2012). Two articles were published referring to its synthesis, presented in Table 11.

Table 11. Synthesis of *A. obtectus* pheromones found in the literature.

Pest insects	Products	Key step	Overall yield	Number of steps	Synthesis
Acanthoscelides obtectus	(*R*)-(-) -(*E*)-2,4,5-Methyl tetradecatrienoate	Claisen rearrangement of ortho ester	1%	7	Mori *et al.* (1981)
		Horner-Wadsworth-Emmons reaction	29%	3	Franck-Neumann *et al.* (1998)
		Horner-Wadsworth-Emmons reaction	35%	5	Satoh *et al.* (2002)
		Asymmetric catalytic reaction with palladium	uninformed o	6	Ogasawara *et al.* (2005)
		Claisen rearrangement	22%	8	Mori, (2012)
	(±) -(*E*)- Methyl tetradeca-2,4,5-trienoate	Wittig Olefination	35%	3	Sakhautdinov *et al.* (2018)
		Alkylation and lactonisation	10%	5	Melikyan *et al.* (1990)
		Wittig and Schlosser reactions	uninformed o	uninformed o	Badanyan *et al.* (2001)
	(2*E*,4*Z*)- Methyl 2,4-decadienoate	Thermal rearrangement of allenic 3,4-esters	40%	2	Mori, (2012)
		Wittig Olefination	42%	3	Shakhmaev *et al.* (2017)

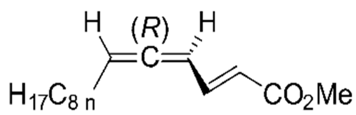


Fig. 11. (*R*)-(-)-(*E*)-2,4,5-methyl tetradecatrienoate trienoate.

From the analysis of the articles, we observed that the most used reactions for the synthesis of methyl (*R*)-(-)-(*E*)-2,4,5-tetradecatrienoate are Wittig, Horner-Wadsworth-Emmons and Claisen rearrangements. The progress in routes over the years regarding yields, reaction conditions, route shortening, and economy is also visible. The routes described by most authors are asymmetrical because of the complexity of the reaction and difficulties in elaborating the reactions. The proposal of the racemic synthesis was published in 2018 in three stages with a yield of 35%, thus having a low number of steps, the economy of reagents, mild conditions and satisfactory yield is a promising route for large-scale pheromone production (Sakhautdinov *et al.,* 2018). For the methyl (2*E*,4*Z*)-2,4-decadienoate molecule, the best synthesis is the one described by Shakhmaev *et al.* (2017) which occurs in three steps with an overall yield of 42%.

Synthesis of cucujolides aggregation pheromones of C. ferrugineus and O. surinamensis

Macrolide lactones, the "cucujolides" (Fig. 12), are a class of aggregation pheromones (Hötling *et al.,* 2014).

In this review, 15 articles referred to synthesising these compounds from *C. ferrugineus* and *O. surinamensis*. For *C. ferrugineus,* there are two macrolides, (4*E*,8*E*)-4,8-dimethyl-4,8-decadien-10-olide (ferrulactone I or cucujolide I) and (3*Z*,11*S*)-3-dodecen-11-olide (ferrolactone II or cucujolide II) (Hötling *et al.,* 2015). For *O. surinamensis* there are five macrolides, (3*Z*,6*Z*)-dodeca-3,6-dien-11-olide (cucujolide IV), (5*Z*,8*Z*,13*R*)-tetradeca-5,8-dien-13-olide (cucujolide V), (3*Z*,6*Z*)-dodeca-3,6-dien-12-olide (cucujolide IX), (5*Z*, 8*Z*,12*R*)-tetradeca-5,8-dien-12-olide (cucujolide X) and (9*Z*,12*Z*,15*R*))-octadeca-9,12-dien-15-olide (cucujolide XI) (Hötling *et al.,* 2014). Table 12 shows the articles that synthesized these compounds in chronological order of publication.

In general, the articles described obtaining ferrulactone starting from geraniol; lactonisation is the common step among them. The most exciting commercial synthesis is described by Cheskis *et al.* (1990b), a six-step synthesis with a 28% yield. For ferrulactone II, its production converges as a whole to form hydroxy acids that are subsequently lactonised. The routes presented have low yields. However, studies on obtaining the pheromone via fatty acids are promising for shortening the route and higher yields (Odinokov, Ishmuratov, Botsman, Vakhidov, Khametova, *et al.,* 1992; Vanderwel *et al.,* 1992). Finally, Cucujolide X and XI are synthesized with satisfactory yields of 29% and 37%, respectively.

Table 12. Synthesis of *A. obtectus* pheromones found in the literature.

Insect pest	Products	Key step	Overall yield	Number of steps	Synthesis
Cryptolestes ferrugineus	Ferrulactone I	Intramolecular alkylation	11%	5	Oehlschlager *et al.* (1983)
		Macro lactonisation by Corey's method	10%	7	Sakai and Mori, (1986)
		Lactonization of α,ω-hydroxy acid	28%	6	Cheskis *et al.* (1990b)
		Oxidative cleavage	uniformed	2	Vanderwel *et al.* (1992)
		Cycling with palladium	17%	5	Kukovinets *et al.* (1996)
	Ferrulactone II	Grignard reaction and lactonisation.	1.7%	17	Sakai and Mori, (1986)
		Wittig reaction and enantioselective reduction	17.5%	5	Keinan *et al.* (1991)
		Hydroxy acid cyclization	uniformed	3	Vanderwel *et al.* (1992)
		Ozonolysis and hydroxy acid cyclization	uniformed	uniformed	Odinokov *et al.* (1992)
		Wittig reaction	19%	8	Czeskis *et al.* (1993)
			20%	8	Cheskis *et al.* (1993)
		Cadiot - Chodkiewicz cross coupling	10%	10	Mavrov and Serebryakov, (1993)
		Horner-Emmons reaction	9%	5	Vasil'ev *et al.* (1996)
Oryzaephilus Surinamensis	Cucujolide X	Wittig reaction and Ring-closing alkyne metathesis (RCAM)	29%	7	Hötling *et al.* (2014)
	Cucujolide XI	Wittig reaction and carbodiimide esterification	37%	3	Hötling *et al.* (2015)

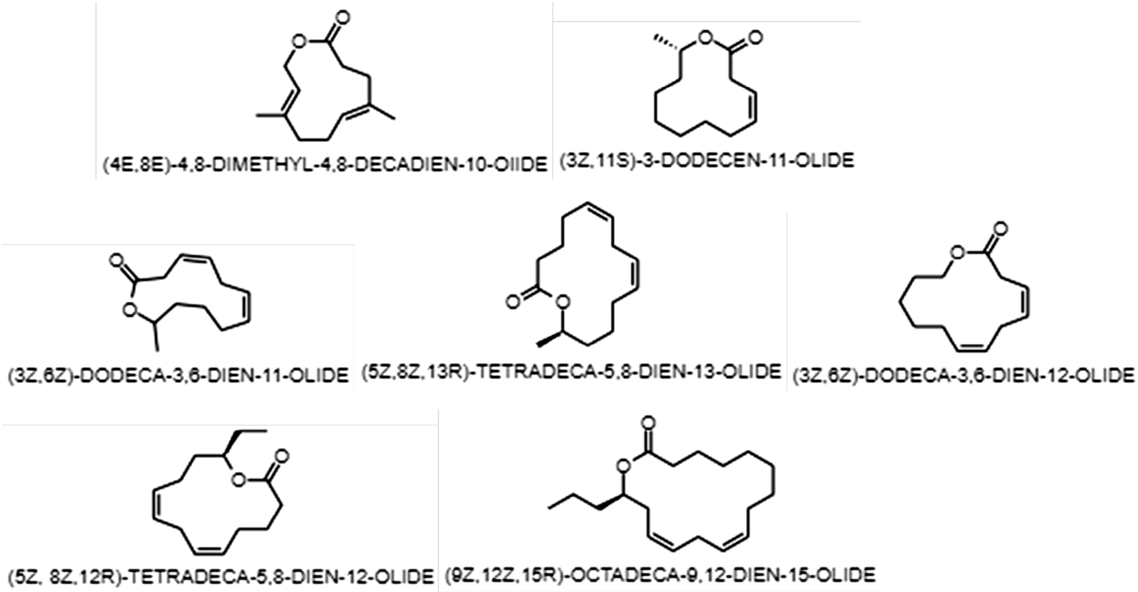


Fig. 12. Macrolide lactones

Conclusion

To our knowledge, 17 distinct compounds are used to control and monitor stored grain pests as pheromone baits, including ten aggregations and seven sexual. The compounds used as pheromones belong to different chemical classes, four alcohols, four aldehydes, three ketones, fifteen esters and two hydrocarbons. This highlights a low chemical diversity among the chemical constituents of the pheromones. The number of publications was higher in 1990, showing a decrease in 2000, an increase in 2010 and a sharp reduction in 2021, demonstrating a lack of interest in this topic for some countries. A more significant number of research studies on the use of stored grain pest pheromones have been carried out in the North American continent, demonstrating that countries like the U.S.A. have invested massively in this area of research. Despite being the largest corn exporter and the largest soybean producer in the world, Brazil does not have the most scientific studies in this area. This fact highlights the need for more significant investments in this area of research by South American countries, mainly Brazil. Considering the main pests of many food products, beetles of the genus *Tribolium* were the most addressed in the articles. The beetle *T. castaneum* is being studied, followed by *R. dominica* and *P. interpuctella*. Most of the articles in the present review monitored stored grain pests, totalling 93 articles. This is probably because this approach prevents the overuse of insecticides to control these pests. Finally, most of the studies were carried out in the laboratory, and little has been studied about the performance of these products in the field, which opens a critical window for evaluating this approach in an environment closer to reality. Since factors such as climate and location can influence the response of the pest to the pheromone, it is necessary to evaluate each pheromone compound in the context of each region, thus avoiding the wrong execution of strategies and frustration with programs that integrate different control and monitoring strategies.

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