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Review

REVIEW ON PESTICIDES FORMULATION IN THE WORLD FROM OLD TO NEW, WITH ITS STATUS IN IRAN

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Abstract: *Pesticide toxicology has made great progress in recent years. Formulation is a set of operations that improve transport, use, and efficacy of a chemical compound. In addition to high risks to humans and non-target organisms, old pesticide formulations had a long persistence in agricultural products and also nature where their consumption was associated with high risks to consumers. Carcinogenicity, teratogenicity, and genetic mutations were only part of the risks of old pesticides to humans, many of which caused high mortality in underdeveloped countries. In recent years, the world's leading pesticide companies have reconsidered their approach to changing formulations and have taken steps to produce formulations with shorter persistence but with better efficacy on target pests. In this way, some new pesticides can be made from combination of old pesticides; but, they are offered in new formulations that are completely different from their original form. This article examines new pesticide formulations and compares them with old and conventional pesticides, which are notable in various aspects.*

Keywords: *Pesticide, formulation, technical material, active ingredient.*

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1. Literature used

In this research, valuable toxicology references in Iran and the other countries of the world, which are often in the form of main textbooks, were used. Instructions from pesticide manufacturing companies were also considered.

2. Introduction

2.1. Chemical control of pests

With the world's population growing rapidly in recent decades, the need for agricultural products and food is increasing. Given the increasing of demand for food, production of pesticide chemicals in order to preserve food products needed by humans was also intensified. This is reflected increasing attention of countries around the world, especially populous countries to this science [1, 2].

Every year, large quantities of crops and fruits obtained from farmers' labor in the field or gardens are attacked by harmful insects, pathogens, and plant weeds. Therefore, protecting cultivated plants from damage of these destructive factors is becoming increasingly important. Chemical control is one of the methods of pest control in which the chemical pesticides are used to control pests. This method, which gained great development after World War II, initially showed decisive and rapid results compared to other control methods; but, it was not long before its harmful and destructive effects on humanity became apparent. For this reason, attempts were made to reconsider its use. Chemical control is now the latest tactic that mankind is using in the new method of pest control [3, 4].

2.2. Trade in Pesticides Formulation

Toxicology is a branch of pharmacology. In parallel with progress in pharmaceuticals; the pesticides production industry has also made significant advancement during the last decade [5]. The world major pesticides production companies are pioneers in this field. Some of them are active in both fields (drugs and pesticides production). Asian companies especially Japan, India, and China have made good progress in this case. The global value of the pesticide production market has grown significantly in recent years which grew at rate of 4.2% from 2015 to 2019, was close to $84.5 billion in 2019. Also, according to Statista (2022), the value of crop protection market reached 65.21 billion U.S. dollars in 2021 which indicates very high turnover of this industry [3]. In Iran, approximately 30,000 tons of agricultural pesticides are annually consumed, of which 20 to 25% is imported and 75% is domestically formulated. In addition to monitoring imported pesticides, the process of producing domestic pesticides is carefully monitored. Therefore, most domestically produced pesticides are of good quality. The price of imported materials is always dependent on the exchange rate. Due to exchange rate fluctuations, the market for chemicals used in agriculture in Iran has been facing turbulence. On the other hand, due to foreign sanctions, it is not possible to import high-quality technical materials. For this, we are forced to import lower-quality technical materials from some Asian producing countries.

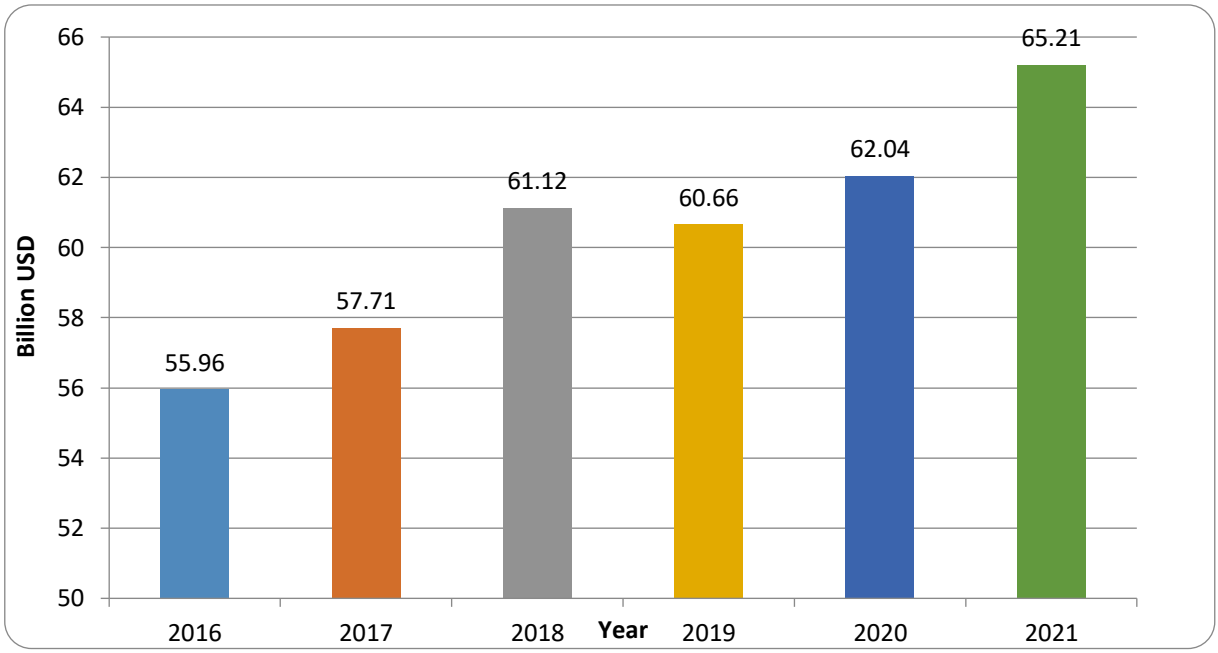


Figure 1. Value of global market for crop protection 2016-2021 (in billion US dollars [5]. (**Source:** Adapted from [6])

2.3. Steps in preparing a pesticide

Preparing a chemical compound in form of a new formulation has various steps as follows: synthesis of a compound in the laboratory, selection in greenhouse, determination its biological activity, testing on a limited scale in field, long-term field testing for three to four years, and launching it on the market based on a license or protected use right. The introduction of a new pesticide takes 17 to 20 years and out of every 100,000 new compounds, one compound has a chance of being introduced [6-8].

2.4. Technical and Active Ingredients

Technical Ingredient (TC): Every pesticide is manufactured in factory as a relatively pure chemical (Figure 2) which called Technical Material. The technical material is relatively pure and contains 90 to 95% of active ingredient (AI) [7, 8].

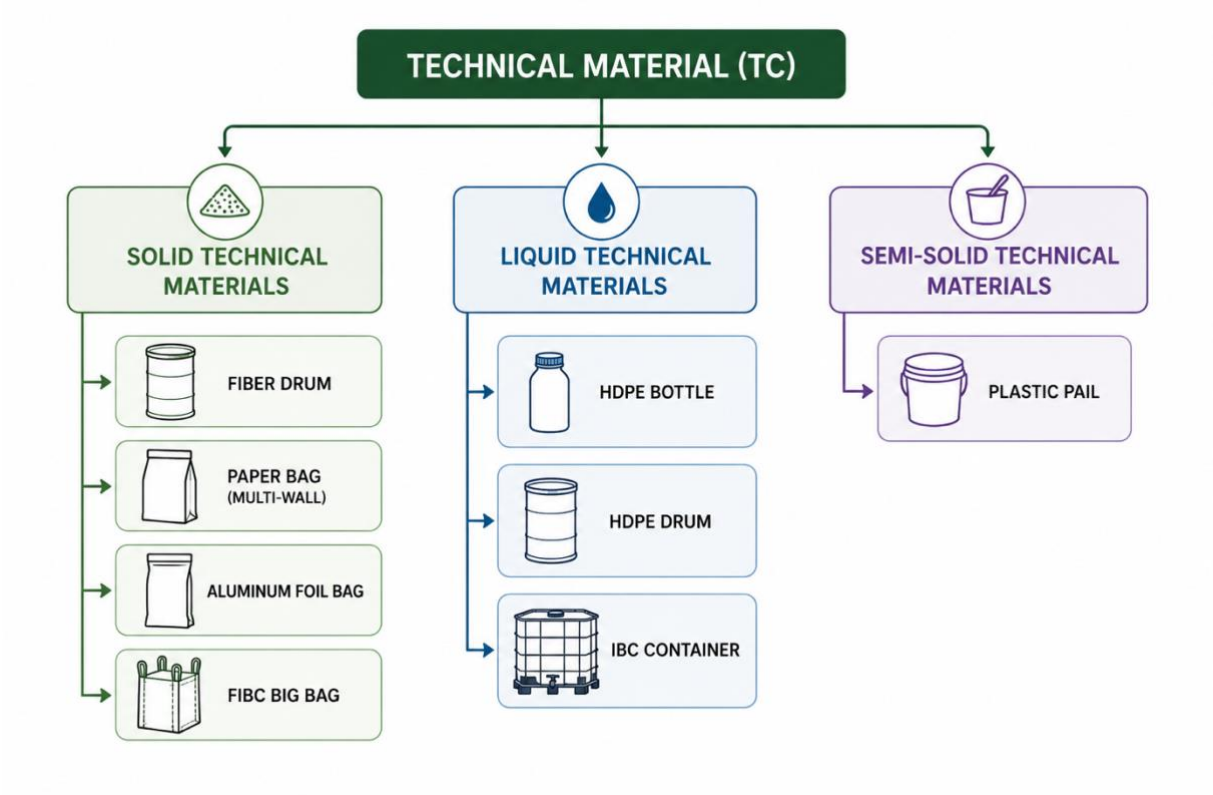


Figure 2. Packaging for technical materials used in agriculture.

Active Ingredient (AI): The chemical compound which is directly responsible for pesticidal effects. It cannot be used directly against pests (Figure 3). In order to use, operations must be performed on it. These operations are called “pesticide formulation” [9-11].



Figure 3. A sample of an active ingredient from pesticide [12].

3. Pesticide Formulation

Pesticide chemicals in their raw or unformulated form are not suitable for pest control. These chemicals are concentrated and their active ingredients may not dissolve well in water, or they may not be chemically stable. Also, they may not be easy to store or transport. For these reasons, pesticide formulators add substances such as clay and solvents to raw chemicals to increase their effectiveness, safety, ease of application, storage, and persistence. These additives do not have pesticidal activity and are combined with active ingredient such as a carrier. The percentage of these substances in the formulation is listed on the pesticide label as "other ingredients" (Figure 4). The combination of active chemicals and other ingredients is called "pesticide trade formulation". Pesticides are usually composed of combination of several chemicals and are used to prevent or eliminate insects, fungi, weeds, nematodes, rodents, and any other type of pest. The last stage of pesticide preparation in the factory is called "formulation". In general, formulation is a set of operations that are performed on a technical substance to improve its properties in terms of storage, transportation, effectiveness, penetration, stability, and hygiene [9-12].



Figure 4. Schematic design of the ingredients in formulation of a pesticide [12].

3.1. Ingredients used in pesticide formulation

Emulsifiers: They cause tiny oil particles to remain suspended in water such as soaps.

Stabilizers: They make the toxic solution more stable by reducing bonding between particles.

Wetting agents-spreaders: Substances that increase contact surface between pesticide droplets and leaf surface.

These three groups have common physicochemical properties. They have a hydrophilic polar part and an insoluble hydrophobic part that are soluble in water and oil (Figure 5). The other ingredients are as follows:

Sticking agents: These cause pesticide particles to stick sprayed surface.

Defoaming agents: They are substances that prevent foam forming in spray tank, such as dimethylpolysiloxane compounds.

Acidifiers: Most pesticides work best in slightly acidic conditions. Therefore, these compounds decrease pH and improve the effectiveness of toxicant.

Attractants: These are substances such as sugar and molasses that attract insects to sprayed area.

Thickeners: Reduce fine droplets and are very effective in reducing pesticides drift, such as polyacrylamide.

Humectants: Increase time between spraying and the evaporation of water droplets from the sprayed surface. Therefore, they are used in cases where penetration of pesticide into leaf requires more time.

Diluents: Substances that are used with water and concentrated insecticides, such as water and some refined oils [13-16].

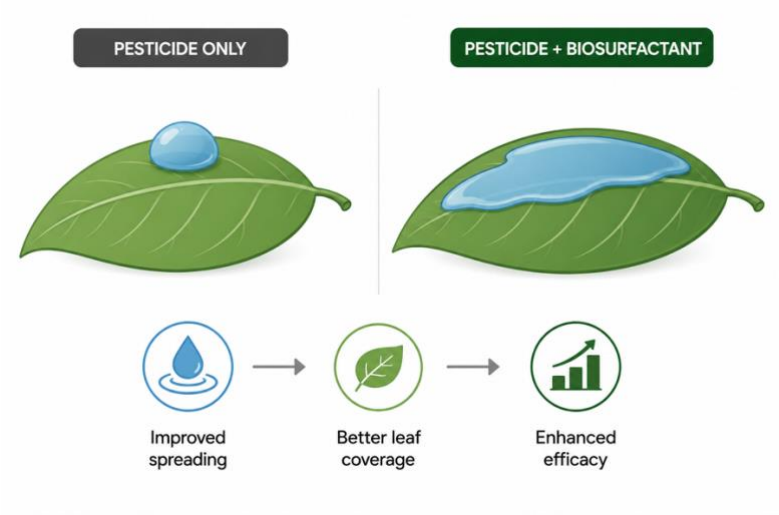


Figure 5. The effect of co-ingredients in improving the efficacy of pesticides [14].

3.2. Old or conventional formulations

Suspension Formulation or Flowable Concentration (SC, FL): This type of formulation is thick and creamy. The active ingredient is suspended in a liquid. It is mixed with water at time of use. This type of formulation is easy to work and does not clog nozzles [17].

Invert Suspension (IS): Is a formulation to reduce pesticides drift. It is mostly used in areas with constant winds. The active ingredient is enclosed in capsules of the same size whose shell is positively charged. This formulation, while preventing evaporation of the active ingredient, allows more droplets to be placed on the target and creates better coverage [18].

Liquid, Solution or Soluble Liquid (L, S, SL): These types of formulations are soluble in water. Their active ingredient is dissolved in water or alcohol. This formulation is similar to an emulsion. However, it does not become milky when mixed with water like an emulsion [19].

Emulsifiable Concentrate (EC): It is a concentrated oil solution containing active ingredient of pesticide that mixes easily with water. It turns into oil particles containing active ingredient and suspended in water. Xylene is a cyclic organic solvent used in this formulation. If the emulsion is well

prepared and should remain suspended in water for several days. If the liquid turns into two phases, it is said that the emulsion has broken. Studies have shown that the type and ratio of the active ingredient, solvent, emulsifier, and method of formulation are effective in the stability of the emulsion in an aqueous solution. A substance that helps oil droplets to remain suspended in water is called an "Emulsifier". About 60% of pesticides consumed in Iran are supplied with this formulation [20].

Oil Solution: This type of formulation is mostly prepared for home use. It consists of a solution of the active ingredient in an oily medium such as odorless petroleum. A pesticide that was produced by an oil company years ago and was widely used in residential spraying had this formulation [9].

Powder (P) or Dust (D): Dust is the oldest type of pesticide formulation that is easy to use. Sulfur is a good example for this formulation. Powder formulations are usually less effective than other formulations. The pesticide particles do not stick to the aerial parts of plant and therefore are not very stable. In addition, it should be applied in calm weather [9].

Wettable Powder (WP): A wettable powder is essentially a ball containing active ingredient along with a wetting agent. If this powder is added to water, it becomes suspended. Usually, the active ingredient is added to inert materials such as talcum powder or clay. After adding the wetting agent, it is ground. The suspension of the powder in water is not very stable. A sprayer with an agitator must be used to apply this formulation [20].

Soluble Powder (SP): This formulation contains the active ingredient in form of a fine powder with a small amount of wetting agent. This powder dissolves easily in water. It forms a true clear solution. This solution does not require constant stirring unlike powder and tablets, because it does not precipitate [20].

Ultra Low Volume (ULV): This formulation is used for agricultural, sanitary, and forest spraying purposes. It can also be used in difficult-to-reach areas where it is not possible to carry a lot of water for spraying. In this formulation, either the active ingredient is a liquid that is used after synthesis; or if it is a solid that it is dissolved in the smallest volume of solvent and used without excessive dilution. This formulation should be applied by special aerial or ground sprayers that can adjust the spraying rate between 0.6 and 4.7 liter/hectare [21].

Granule (G): Granules are small grains with a size of 0.1 to 2.5 mm that contain a toxic compound. Central core of the granule consists of clay, sand, softened walnut shells or ground cob wood on which the active compound is sprinkled and absorbed [22].

Dry Flowable or Water Dispersible Granule (DF, WG, WDG): This formulation is similar to a powder or tablet. The difference is that active ingredient is absorbed into the granules. This formulation mixes easily with water [23].

Pellet (P): Like granules, it is composed of small grains. The size of the pellets is larger than granules [24].

Fumigants: In this formulation, the active ingredient is volatile and fills the environment. One of the advantages of this type of formulation is that the gas molecules are dispersed throughout environment and control the pest. After ventilation, it does not leave much residue in the environment. This formulation should be used in a closed place [25].

Aerosols: Are sealed and pressurized cans. By pressing a button on them, the poison particles are released into the air. To make aerosols, active ingredient is dissolved in a volatile medium such as ether and packaged with a pressurized gas such as fluorocarbon or carbon dioxide [25].

Paste: Used to combat pests that live in the interior of branches and trunks of trees or plants. The paste contains a volatile active ingredient. It usually kills the pest through the fumigant phase [9].

Poisonous bait: It is prepared by mixing active ingredient of pesticide with a compound that attracts the pest, which may also be its food. It is applied as a spot in the area where the pest is active [20].

Poisonous tape: In order to apply pesticides locally and on tree trunks, poison-containing tapes are used. Grease-impregnated tapes are also used to trap insects that climb tree trunks. Prominent examples of this type of formulation include Apistan which is used against the Varroa mite (*Varroa destructor*), a honey bee parasite. Sheltox tape consists of a poison and an attractant and Nexalot tape also is used against storage pests and its active ingredient is Gamexan. There are also poison tapes impregnated with Dichlorvos which are used against household insects [20].

Slow-Release Formulations: The first form of these formulations was introduced on 1963. It was a polychloroprene resin tape impregnated with the insecticide dichlorvos. It slowly releases the toxic compound and kills nearby insects. It is mainly used in food warehouses and silos. Phostoxin and aluminum hydroxide are examples of this formulation [9].

Water-Soluble Packets (WSP) or Ready To Use (RTU): Applying of this type of formulation reduces the risks of mixing and preparing some highly toxic pesticides at time of spraying. Certain weights of powder and tablet or soluble powder are packaged in water-soluble plastic bags. When this package is dropped into the spray tank, it dissolves in water and its contents enter the water [25].

3.3. New formulations

The main approaches for producing of new formulations are reducing environmental risks and hazards to human health. Also, reducing the impact on non-target organisms and eliminating hazardous solvents are important in this filed Furthermore, decreasing the occurrence of chemical incompatibilities, improving the effectiveness of pesticides and half-life in the product are the other important aspects. Finally, reducing hazardous residues in products and decrease pre-harvest interval (PHI) are the other aspects [26, 27].

The New Formulations are as follows:

Oil Dispersion (OD): In the oil dispersible formulation, the solid active ingredient is dispersed in oil. The oil can be a variety of paraffinic and aromatic solvents, vegetable oils, or methylated oils. Ideally, the active ingredient is uniformly suspended in the oil phase [28].

Special Formulation for Seed Disinfection or Flowable Concentration for the Seed Treatment (FS): The FS formulation is a suspension concentrate (SC) with supplementary additives for adhesion to seed surface. It uses dyes as safety indicators to show that a seed has been treated with this product [28].

Capsule Suspension (CS): Capsule suspensions are water-based and slow-release formulation. It contains the active ingredient encapsulated within polymer microcapsules. After application, the pesticide is slowly released from the microcapsules. This formulation is very useful for providing a long-lasting effect in controlling target pests [29].

Zeon Technology or Zeon Concentrate (ZC): Zeon technology is the result of years of research by scientists at the Switzerland Company known as “Syngenta”. In this formulation, active ingredient particles between 1 and 4 microns are surrounded by microcapsules. The number of microcapsules in one liter of this formulation is more than 53 trillion. About, 40 microcapsules stacked on top of each other have a diameter equal to a human hair. Only 17 microcapsules are needed to kill a sucking insect such as an aphid. After spraying, the capsules containing the active ingredient adhere firmly to leaf surface. The capsules dry quickly, burst, and release the active ingredient into an area 40% larger than the diameter of a drop of pesticide. First, the large capsules and then gradually the small capsules are released [30].

Emulsion in Water (EW): It produces smaller particle sizes than EC formulation. This results in less drift and greater exposure of active ingredients to leaf surface. It has better efficacy compared to EC formulation. It has also shown lower environmental risks compared to EC formulation [31].

MicroEmulsion (ME): Microemulsion formulation is isotropic mixtures of at least one hydrophilic, hydrophobic, and amphiphilic compound. Thermodynamic stability and nanostructure are two important characteristics of this formulation. Distinguishing them from conventional emulsion which are thermodynamically unstable [32, 33].

SuspoEmulsion (SE): This formulation is made by combining two active ingredients with different physical properties in one formulation. It is a combination of suspension concentrate (SC) formulation and water-based emulsion (EW). One of the advantages is that it is possible to formulate several active ingredients together. It expands their spectrum of action and eliminates the disadvantages of incompatibility from the mixture in tank [34].

A general comparison of the new and old formulations is listed in Table 1.

Table 1. General characteristics of old and new pesticide formulations.

Formulation	Effectiveness	Toxicity	Cost	Application method
SC, FL	+++	++	++	Easy
IS	++	++	++	Easy
EC	+++	+++	+++	Easy
Oil Solution	+++	+++	+++	Easy
L, S, SL	++	++	++	Easy
Powder (P) or Dust (D)	+	++	+	Hard
WP	++	++	++	Easy
SP	+	++	++	Easy
ULV	++	+	++	Hard
G	+	+	+	Easy
DF, WG, WDG	+++	++	+	Easy
Pellet (P)	+	+	+	Easy
Fumigants	++	+++	++	Hard
Aerosols	++	+++	++	Hard
Paste	+	+	+	Easy
Poisonous bait	+	+	+	Easy
Poisonous tape	+	+	+	Easy
Slow-Release Formulations	+++	++	+	Easy
WSP or RTU	++	++	+	Easy
OD	+++	+++	++	Easy
FS	+++	+	+	Easy
CS	+++	+	+++	Easy
ZC	+++	+	+++	Easy
EW	+++	+	++	Easy
ME	+++	+	++	Easy
SE	+++	+	++	Easy

+: Low, ++: Medium, +++: High

4. Pre-harvest interval (PHI) and Maximum Residue Limits (MRL)

The pre-harvest interval is minimum time between the last application of a pesticide and harvest of that crop. This time can be found on the label of each pesticide and is a legal requirement. PHI provides farmers with the information they need to ensure that residues in treated produce do not exceed the Maximum Residue Limits (MRL). The MRL represents the maximum amount of pesticide residue that is expected to remain on a food product when the pesticide is used according to label instructions. The MRL is legally established and regulated under the pest control products. It is important to respect PHI so the MRL for a given crop is not exceeded. Residues found in excess of the MRL on food would constitute a violation of the food and drug act and could also pose a risk to consumer health. In such situations, the harvested crop could be seized, destroyed, or forbidden for export. Use pesticides only for the crops and pests listed on the product's label and make sure to follow the application rates, number

of applications, and PHI stated on the label. The PHI can typically be found under the “directions for use” section of the pesticide label. Always read the label to verify PHI instructions [35, 36].

5. Conclusion

Pesticide formulations made significant progress in recent years and various formulations have been introduced in the world. These formulations have received much attention in improving pest control and reducing their persistence in agricultural products. For this reason, they are more expensive than conventional formulations. OD, FS, EW, Microemulsion (ME), and SuspoEmulsion (SE) are examples of them, each with unique properties. It is hoped that in the future we will see the introduction of newer formulations that will benefit plant science and pest management.

6. Future prospects

More formulations will be introduced in future. Reducing environmental risks is one of the main approaches in the future. By using new formulations, the adverse effects of pesticides on humans and non-target organisms will be reduced. Occurrence of chemical incompatibilities caused by consumers' failure to comply with mixing principles, often do not have sufficient knowledge of the mixing principles of pesticides, will be eliminated. Overall, the effectiveness of pesticides can be increased as best as possible and the durability of pesticides should be improved for long-term pest control. In addition, hazardous residues in products are minimized. Reducing the PHI of pesticides and eliminating toxic residues is one of the main approaches for future pesticide formulations.

Ethical statement

As this study is a literature review based solely on previously published studies, ethical approval was not required

Conflict of interest

The authors haven’t any conflicts of interest.

Authors’ Contributions

Parivash Rezaei: Preparing of manuscript
Parisa Ghezelbash, Editing the manuscript
Oruj Valizadegan: Final Approval

Generative AI statement

Generative AI tools were used only for the preparation of figures and graphical illustrations in this study. No generative AI tools were used for writing, editing, interpreting, or generating the scientific content of the manuscript.

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