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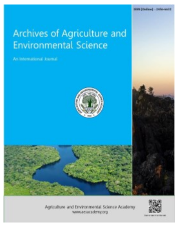

REVIEW ARTICLE

Recent advances in pesticide formulations for eco-friendly and sustainable vegetable pest management: A review

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ABSTRACT

In order to reduce the loss and maintain the quality of vegetables harvest, pesticides are used together with other pest management techniques during cropping to destroy pests and prevent diseases. However, the use of pesticides during production often leads to the presence of pesticide residues in vegetables after harvest. Higher doses and repeated applications of conventional formulations lead to accumulate pesticide residues in vegetable commodities along with environmental pollution. With the increasing awareness of toxic effects of conventional formulations, there is a significant trend towards switching over from such pesticide formulations using petroleum and organic solvent based constituents to user and environment friendly water based pesticide formulations. The developed world has progressed substantially in this regard to develop eco-friendly formulations which are safer to vegetable and the environment. These formulations would not only replace toxic, non-degradable ingredients/adjuvants of the conventional formulations but also increase the bio-efficacy of the products through incorporating latest technologies including size reduction (Wettable Powder to Suspension Concentrate, Soluble Concentrate to Microemulsion), increased coverage of applied surface area (EC to ME/Nano-formulations), reduced wastage (Dust/WP to Controlled Release Formulations) and dose rates of applied same pesticides to improve food quality with minimum pesticide residues.

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INTRODUCTION

Vegetables are important components of the human diet since they provide essential nutrients that are required for most of the reactions occurring in the body. Like other crops, vegetables are attacked by pests and diseases during production and storage leading to damages that reduce the quality and the yield (Oerke, 2006). In order to reduce the loss and maintain the quality of vegetables harvest, pesticides are used together with other pest management techniques during cropping to destroy pests and prevent diseases (Sarwar, 2013) However, the use of pesticides during production often leads to the presence of pesticide residues in vegetables after harvest (Chloe *et al.,* 2015). Pesticides in developing countries in Asia and Pacific region are mainly available as dust, wettable powder, emulsifiable concentrates, solutions, etc for vegetable pest management. These types of formulations are regarded now as in 'conventional', 'old technology' or 'classical' or 'traditional' because of their increased dose rate or repeated applications to get desired bio efficacy. These higher doses and repeated applications lead to accumulate pesticide residues in vegetable commodities along with environmental pollution (Koirala *et al.,* 2009). Conventional formulations, because of their characteristics i.e. dustiness and use of volatile organic solvents (VOCs) in their preparation maximize several problems like pesticide residues in fruit and vegetable products etc. With the increasing awareness of toxic effects of conventional formulations, there is a significant trend towards switching over from such pesticide formulations using petroleum and organic solvent based constituents to user and environment friendly water based pesticide formulations (Knowles, 2008).The developed world has progressed substantially in this regard to develop eco-friendly formulations which are safer to vegetable and the environment (Green *et al.,* 2007). These formulations would not only replace toxic, non-degradable ingredients/adjuvants of the conventional formulations but also increase the bio-efficacy of the products through incorporating latest technologies including size reduction (Wettable Powder to Suspension Concentrate, Soluble Concentrate to Microemulsion), increased coverage of applied surface area (EC to ME/Nano-formulations), reduced wastage (Dust/WP to Controlled Release Formulations) and dose rates of applied same pesticides to improve food quality with minimum pesticide residues (Beestman, 2003). Suspension Concentrates, Water Dispersible Granules, Emulsion in Water, Micro-emulsion, Combination Formulations, Effervescent Tablets, Floating Tablets, seed treatment formulations etc. are some of the formulation types that come under this category of safer formulations for the production of safe and clean vegetables.

Formulation selection considerations: The importance of formulation type is generally over looked. A well-considered decision to use the most appropriate formulation for vegetable pest management requires detailed analysis of the following factors (Copping, 2004).

Applicator safety: Different formulations present various degrees of hazard to the applicator. Some products are easily inhaled, while others can penetrate skin or cause injury when splashed in the eyes.

Environmental concerns: Special precautions need to be taken with formulations that are prone to drift in air or move off target into water. Wildlife can also be affected to varying degrees by different formulations. Birds may be attracted by granules, and fish or aquatic invertebrates can prove especially sensitive to specific pesticide formulations.

Pest biology: The growth habits and survival strategies of a pest generally determine which formulation provides optimum contact between the active ingredient and the pest.

Available equipment: Some pesticide formulations require specialized handling equipment. This includes application equipment, safety equipment, and spill control equipment.

Surfaces to be protected: Applicators must be aware that certain formulations can stain fabrics, discolor linoleum, dissolve plastic, or burn foliage.

Cost: Product prices may vary substantially, based on the ingredients used and the complexity of delivering active ingredients in specific formulations.

Formulation types of agrochemicals: Different types of formulations of agrochemicals can be identified depending on the application, customer acceptability and regional market requirements. At present, most agrochemical companies attempt to formulate a product in a form that can be accepted globally (Mulqueen, 2003). This presents a challenge to the formulation scientists who not only needs to understand the basic and fundamental principles in such formulation types, but also should be able to produce formulations that can be applied worldwide.

DRAWBACKS OF CONVENTIONAL FORMULATION TECHNOLOGIES

Granules (GR): Granular pesticide formulations are distinguished from powder formulations according to mesh size. It is generally accepted that a granular formulation is a product with a size range from 16-60 British Standard BS mesh (250-1,000 microns) with at least 90% of the granules within the specified mesh size range. Granules are, therefore, the largest of the solid pesticide formulations (apart from tablets) and their large size virtually eliminates drift leading to much less loss of pesticide than with powder and liquid formulations (Gilden *et al.,* 2010). Granular formulations are often used as pre-emergence herbicides or as soil insecticides for direct broadcasting to the field. They are also applied for "in-furrow" use, especially for insecticides.

Wettable powders (WP): Wettable powders are finely-divided solid pesticide formulations which are applied after dilution and as a suspension in water. They have been used for many years and are second only to emulsifyable concentrates in terms of the total volume of products produced globally. These particles are larger than the droplets produced by emulsifyable concentrate formulations. It is this factor, coupled with the lack of solvent, which gives WP's lower biological activity than most liquid formulations. However, this also makes them less likely to cause phytotoxicity to crops (Gupta, 2004).

Disadvantages: Difficult to mix in spray tanks; Poor compatibility with other formulations; Tank mix wetter may be needed: Dust hazard during manufacture: Dust hazard during application.

Emulsifiable concentrates (EC): Emulsifiable concentrates are popular for active ingredients which are very soluble in non-polar solvents. They are formulated by dissolving the active ingredient with emulsifying surfactants in an organic solvent. EC formulations are easy to use and, when diluted in water, should give a stable "milky" emulsion with very little creaming and no oil separation. EC formulations must also be compatible with spray tank water covering a range of water hardness from very soft water up to about 1,000 ppm of hardness.

Disadvantages: Emulsion stability problems may arise after dilution; Sometimes phytotoxic to vegetable crops; May increase dermal toxicity of active ingredient; Possible fire hazard; Solvents may affect plastics and rubbers in spray applicators.

Soluble concentrates (SL): A soluble concentrate is a clear solution to be applied as a solution after dilution in water. Soluble concentrates are based on either water or a solvent mixture which is completely miscible in water. Solution concentrates are the simplest of all the formulation types and merely require dilution into water in the spray tank (Zabkiewicz, 2000). However, the number of pesticides which can be formulated in this way is limited by two factors, the solubility and hydrolytic stability of the active ingredient in water. Water based solution concentrate formulations are hydrophilic after spraying onto crops and, therefore, often contain a surfactant to assist wetting onto the leaf surface.

Disadvantages: Often requires surfactant wetters for good wetting/spreading on vegetable leaves; Poor low temperature stability; May hydrolyze active ingredients; Corrosive to metals.

Trends towards safer formulation technologies: However, there has been a dramatic shift from WP formulations to WG, from EC to EW. SCs have also increased in popularity due to their environmental advantages, being water based, and their ease of application (spontaneous dispersion on dilution into water) (Mathur, 1999). In all the above formulations, considerable attention has been paid in recent years to achieve a number of objectives Smith *et al.* 2008):

Broader formulation inerts; Solvent reduction and safer solvent selection; Safer surfactant components with low toxicity, low skin irritation and enhanced biodegradability (Fantke *et al.,* 2012); Longer term physical and chemical stability; Enhancement of bio efficacy by incorporation of adjuvants; Controlled and sustained release formulations; Compatibility of various formulations in tank mixes.

These challenges require good knowledge of colloid and surface science as well as the key factors involved in formulating complex systems (Mulqueen, 2003). In this review, some of the recent advances in agrochemical formulation technology will be discussed in the four main areas.

Water based dispersion technology; Improved dry product (WDG) technology; Controlled release technologies for improved product performance; Combined/mixed formulation technology; Nanotechnology-based pesticides due to size and surface characteristics.

WATER BASED DISPERSION TECHNOLOGY

Suspension concentrates (SC): Suspension concentrate technology has been increasingly applied to the formulation of many solid crystalline pesticides since the early 1970's. Pesticide particles maybe suspended in an oil phase, but it is much more usual for suspension concentrates to be dispersions in water (Mulqueen, 2003). Considerable attention has been given in recent years to the production of aqueous suspension concentrates by a high energy wet grinding processes such as bead milling. The use of surfactants as wetting and dispersing agents has also led to a great deal of research on the colloidal and surface chemistry aspects of dispersion and stabilization of solid/liquid dispersions (Green *et al.,* 2007). Water-based suspension concentrate formulations offer many advantages such as:

High concentration of insoluble active ingredients; Ease of handling and application; Safety to the operator and environment; Relatively low cost; Enable water-soluble adjuvants to be built-in for enhanced biological activity.

Farmers generally prefer suspension concentrates to wettable powders because they are non-dusty and easy to measure and pour into the spray tank. However, there are some disadvantages, notably the need to produce formulations which do not separate badly on storage, and also to protect the product from freezing which may cause aggregation of the particles.

Example: Fipronil 5 SC, Sulphur 52 SC, Hexaconazole 10 SC, Carbendazim 50 SC etc.

O/W emulsions (EW): Oil-in-water emulsions are now receiving considerable attention because of the need to reduce or eliminate volatile organic solvents (VOCs) for safer handling (Ware, 2004). Because they are water based, oil-in-water emulsions can have significant advantages over emulsifiable concentrates in terms of cost and safety in manufacture, transportation and use. Key is that the active ingredient must have very low water solubility to avoid crystallization issues (Tadros, 1995). A solid active may be dissolved in a water–immiscible solvent.

Example: Butachlor 50 EW, Cfluthrin 5 EW, Tricontanol 0.1 EW etc.

Suspo-emulsions (SE): Mixed combination formulations are becoming more popular because of their convenience, they ensure that the farmer applies the correct amount of each component pesticide and overcome problems of tank mix incompatibility. Suspo-emulsions can, therefore, is considered to be mixtures of suspension concentrates and oil-in-water emulsions with added surfactants to prevent flocculation and thickeners to prevent separation of the dispersed phases (Tadros, 1995). Surfactants used as dispersing agents for the solid phase are similar to those already mentioned for suspension concentrates. Careful selection of the appropriate dispersing and emulsifying agents is necessary to overcome the problem of hetero-flocculation between the solid particles and the oil droplets and extensive storage testing of these formulations is necessary.

Example: Fenpropimorph 24.5 + Epoxiconazole 8.2 SE (Not registered in India)

Microemulsions (ME): Microemulsions are thermodynamically stable transparent dispersions of two immiscible liquids and are stable over a wide temperature range (Hiromoto, 2007). They have a very fine droplet size of less than 0.05 microns (50 nanometers). The total concentration of surfactants for a microemulsion can be as high as 10–30% or more, compared with about 5% for a typical o/w emulsion. Microemulsions have relatively low active ingredient concentrations, but the high surfactant content and solubilisation of the active ingredient may give rise to enhanced biological activity.

Example: Neemazal 30 MEC, Pyrithiobac Na 5.4 + Quizalofop-P-Ethyl 10.6 MEetc

Oil dispersion formulations: One of the latest formulation types are oil dispersions (ODs). This technology allows very efficient and environmentally friendly agrochemical formulations. In ODs the solid active ingredient is dispersed in the oil phase, making it especially suitable for water-sensitive or non-soluble active ingredients (Llácer *et al.,* 2012). When the oil dispersion comes into contact with water the formulation can either form an emulsion or a suspo emulsion. The oil-phase can comprise different oils such as mineral oils, vegetable oils or esters of vegetable oils. Special attention is needed with the auxiliaries in ODs: suitable oil-compatible dispersing agents and emulsifiers adjusted to the type of oil which forms a stable emulsion after dilution with water.

Aqueous flowables (AF): Aqueous flowables are concentrated 40% to 70% w/w suspensions of micronized insoluble active pesticide in water. Prior to spraying on target areas, aqueous flowables are diluted with water in a spray tank to achieve the minimum effective pesticide concentration. AFs must be formulated for low viscosity and good fluidity, so that transfer to the spray tank is easy and complete. This requires an effective wetting agent and an efficient dispersing agent to ensure adequate dispersion of the pesticide in the water (Castro *et al.,* 1998). Since the active ingredients in AFs are insoluble, good suspension stability is essential. If the suspension settles and leaves sediment at the bottom of the container, the application of the pesticide may be too weak to be effective (Dipak, 2015). Further, disposal of the residue in the container becomes a problem. A combination of smectite clay (aka bentonite) and xanthan gum works synergistically to provide excellent long term suspension stability at low viscosity and at low cost.

Seed treatment formulations: As a kind of pesticide preparation with film-forming characteristics used for coating of plants and other plant seeds, seed coating agent is generally prepared by technical material, dispersant, wetting agent, film former, pH regulator, antifreeze, defoamer, other auxiliaries and water (Dayer *et al.,* 2007). It can be directly coated on the seed surface after dilution to form a protective film with certain strength and permeability, so it is named as seed coating agent. Seed coating agent and seed treatment agent are two different concepts. Seed treatment agent is divided into seed dressing agent, multi-seed agent and seed coating agent. Seed dressing agent and seed soaking agent belong to a method of field pesticide application, but not a kind of pesticide formulation. Seed coating agent is not required within nearly 45 days at bud stage and seeding stage due to its characteristics of coating of seeds, and the dosage is only about 1/50 of field pesticide application. Therefore, it is called the new pesticide formulation saving the most pesticide (Dipak, 2015). Seed coating agent can, according to pesticide formulation, be divided water flowable seed coating agent (FS), water-emulsion seed coating agent (EWS), suspended emulsion seed coating agent (SES), microcapsule seed coating agent (CS), dry flowable seed coating agent (DFS), water dispersible granule type seed coating agent (WGS), etc (El-Mohamedy *et al.,* 2008). As a kind of seed coating formulation most widely applied in the maximum volume at home and abroad, FS is a special SC which super crushes solid pesticides and other auxiliary components into less than 4um.

NEW DRY PRODUCT TECHNOLOGY

Water dispersible granules (WG): Water dispersible granules, or dry flowables as they are sometimes known, are a relatively new type of formulation and are being developed as safer and more commercially attractive alternatives to wettable powders and suspension concentrates (Kim *et al.,* 2003). They are becoming more popular because of their convenience in packaging and use, being non-dusty, free-flowing granules which should disperse quickly when added to water in the spray tank (Marcroft *et al.,* 2008). They therefore represent a technological improvement over wettable powders and imitate liquids in their handling characteristics. Extrusion granulation is one of the safest, most versatile and economical process and is probably the most favoured process used by agrochemical companies at the present time, followed closely by fluid bed spray granulation. The dispersion time in water is a very important property and to ensure that no problems occur in the spray tank it is necessary for all the granules to disperse completely within two minutes in varying degrees of water temperature and hardness.

Example: Mancozeb 75 WG, Endosulfan 50 WG, Captan 83 WG, Cypermethrin 40 WG, Thiomethaxam 25 WG, Deltamethrin 25 WG and so on.

Dispersion concentrates (DC): These are formulations of active ingredient dissolved in a water-miscible, polar solvent together with a dispersing or emulsifying agent, designed to dilute in water giving stable, fine particle size dispersions (Nishiyama *et al.,* 2004). DC formulations are alternatives to SL, SC, EC and ME formulations, being suitable for active ingredients whose physical, chemical or biological properties preclude the use of these more conventional formulations. Choice of dispersing agent is critical for good dilution properties in water. It is important that fine particle size dispersion is obtained, stable for preferably at least 24 hours as a dilution to prevent possible spray equipment blockages and reduced bio-efficacy.

Advantages: Simple process equipment; Easy to use and clean down; Stable, solution-type formulation; Good bio-efficacy .

CONTROLLED RELEASE TECHNOLOGIES FOR IMPROVED PRODUCT PERFORMANCE

Microencapsulation/capsule suspensions (CS): The polymer membrane, or microencapsulation technique, has become popular in recent years (Beestman, 2003). A well-known method of microencapsulation uses the principle of interfacial polymerization. The rate of release of the active ingredient can be controlled by adjusting the droplet size, the thickness of the polymer membrane and the degree of cross-linking or porosity of the polymer. The rate of release of the pesticide is, there-fore, a diffusion controlled process. Further innovations are expected in microencapsulation technology over the next few years which may contribute to safer pesticide use (Fernández, 2007). Significant research is still being expended in the area of microencapsulation technology and there is likely to be further gains from this research.

Example: Lambda Cyhalothrin 10 CS, Lambda Cyhalothrin 25 CS etc.

Combined/mixed formulation technology: Our innovation is the development of a combined (mixed) ZW formulation in the field of agrochemicals for user & environment friendly application of synthetic agrochemicals. It is combination of capsule suspension of lambda cyhalothrin insecticide and concentrated emulsion of chlorpyriphos insecticide (Hazra *et al.,* 2013). In this unique formulation, two different active ingredients in such a way that one active ingredient i.e. chlorpyriphos will be quickly available/effective just after application on target pests for quick knock-down effect and on the other hand, the other pesticide i.e. lambda cyhalothrin will be efficacious slowly in a controlled manner for long term target pest management Takeshita *et al.,* 2001). As it is micro encapsulated in a polymer membrane, applicator can apply two pesticides simultaneously in a single application. The combination will have broad spectrum insecticidal activities and may be used for controlling insects on large number of crops

NANOTECHNOLOGY-BASED PESTICIDE FORMULATIONS

Nano emulsions: Nano-emulsions have a particle size of less than 200 nm, which makes the systems inherently transparent/translucent and kinetically stable (Nair *et al.,* 2010). Pesticides formulated with nano-emulsions having a lower surfactant concentration than micro-emulsions and surfactants are considerably more environmentally friendly and are cost effective and economically (Kuzma *et al.,* 2010). Low-energy emulsification methods are applied to produce nano-emulsions, and the energy store could promote smaller-sized nanoparticles of longer life (Zabkiewicz, 2000; (O'Sullivan *et al.,* 2010; Sarwar, 2014).

Conclusions

With the many pressures on product performance, formulation is becoming a key technology by which agrochemical companies can differentiate their products and add significant value. New product introduction is an important factor in brand refreshment and new formulation technology can impact this considerably. This article has described some of the changes occurring in formulation types employed and the further trends that are driving technologies such as examples of water-based dispersion formulation technology for oil-in-water emulsions, suspensions, micro-emulsions etc. as well as other formulation types such as gel and dry product formulations where new techniques of formulation, often combining polymers and surfactants in novel ways have resulted in a relatively safe and environment friendly product. Moving with a lustrous record of providing quality products to its customers for vegetable pest management since past many years, scientists is now shifting its focus towards 'nanotechnology', keeping in view the hazardous effects of highly toxic pesticides.

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