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NANOENCAPSULATION TO CONTROL HERBICIDE RESIDUES AND RESISTANCES: A REVIEW

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Abstract – Weeds have the ability to interfere in the life cycle as well as the management practices involved with the crop plants. Their rigidity in adapting to any climate and soil condition has made them nearly impossible to eradicate from crop lands. Integrated weed management is considered the best option to limit their population, but when it comes to managing them at a large scale, herbicides are considered as the easiest method of weed management. But there are negative of herbicides even when their formulations or rotations are used to control weeds, of which their persistence is one. Their overuse has also made the weeds familiar to these chemicals, evolving out resistance in weeds. The use of nanotechnology in the field of agriculture has been developing. Weed management through the use of nanoencapsulated herbicide formulations can be a solution to the problem of weed menace. The article is a review of the potentiality of the nano-formulations in weed control.

Why Weed Management?

Weeds are the obnoxious plants that grow among the crop plants and interfere with their management practices. The growing menace of weeds in India has led to the loss in the yield of crops like groundnut, soybean etc, while economic losses have been reported high in cereals such as upland rice, wheat etc. Weed problem is common in agricultural lands throughout the world and it varies according to crop, soil type, cropping pattern and other management factors. The factor that can further aggravate this problem is the climate change as weeds have the ability to adapt themselves to any climatic condition faster than the crop plants. Adding to this problem, developing resistance to herbicides by the weeds due to their frequent overuse is making it harder to control them. Overuse of the herbicides is not only making the weeds familiar to these chemicals but also offending the principles of sustainable agriculture by leaving behind their residues which may remain persistent in soil for months or years.

Potential of Herbicides to Retain in Soil

The ease in controlling the weed menace has been possible due to the use of herbicidal formulations. The chemicals are produced as such to suit the particular crop and cropping systems for a given weather and soil conditions while targeting the weeds. The problem arises when these chemicals which are used to kill weeds start harming the crop plants and disturb the soil ecosystem. Ability of the herbicides to retain in soil for a longer period than desirable is not only potentially harmful to the crops but also lethal for the soil micro-environment. Some herbicides persist in soil for a longer time but are not available for plant uptake and therefore are not active as herbicides. The threshold limit beyond which a herbicide crosses its activity and comes to the potentially harmful phase is important to recognize when sustainability of soil is considered important. Residual activity of the herbicides is often described in terms of half- life. The herbicide half life may vary from days to years and it is primarily responsible for deciding the doses or extent to which we should use a herbicide formulation for a given soil condition. Table 1 shows the relative persistence of some of the herbicides in soil.

It has been observed that the differences in chemical characteristics among the herbicides is relatively small, and therefore, soil type and environment will have a greater impact on performance of herbicide than does the specific herbicide applied (Herzler, 2002). Whereas, literature reviewed by Bailey and White (1964) explains that adsorption and desorption of the herbicides are the sole factors responsible for the interactions of herbicides with soil and depends on the factors such as physio-chemical nature of the pesticide, nature of the saturating cation on the colloid exchange site and nature of formulations apart from soil reactions and soil types, whereas the physical properties of the soil as substrate and climate exert a more indirect influence. The ionic nature of the herbicides is responsible for their exchange on the soil clay complex and thus, retention for a longer period of time. Ionizability refers to the way a given herbicide ionizes in aqueous solution. It is of primary importance because positively charged (cationic) herbicides behave much differently than negatively charged (anionic) or uncharged (nonionic) herbicides. Cationic herbicides are ionically bound to both organic colloids (Best *et al.,* 1972) (Fig. 1) and to clay minerals (Weber and Weed, 1968) (Fig. 2) and their biological availability to plants and microorganisms are regulated by the geometry of binding (Scott and Weber, 1967; Summers, 1980; Weber and Scott, 1966; Weber and Weed, 1974). The herbicides that have non-ionic properties exist in the soil solution only in the molecular form and their reactivity with soils is dependent upon their water solubility the types of reactive functional groups present, and their volatility (Weber, 1966.) This shows that the chemistry of herbicides have an important role to play in their retention in soil.

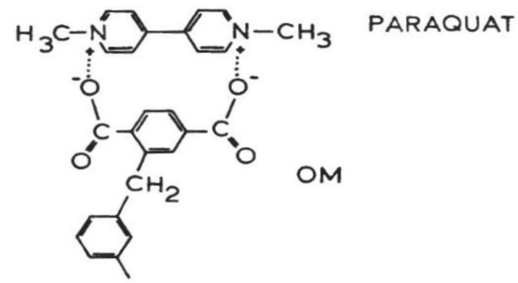

Fig. 1. Paraquat, a strong base herbicide ionically bound to soil organic matter (OM)

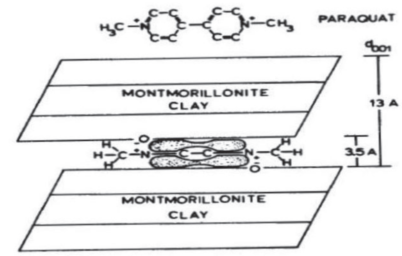

Fig. 2. Paraquat ionically bound on the interlayer spaces of smectite clay minerals

Herbicide Resistance

Herbicide resistance may be defined as the condition whereby a plant withstands the normal field dose of a herbicide, as a result of selection and genetic response to repeated exposure to herbicides with a similar mode of action. Weeds have evolved resistance to 23 of the 26 known herbicide sites of action and to 163 different herbicides (www.weedscience.org). Herbicidal resistance in weeds is most often due to an alteration of the site of action of the herbicide at the cellular level. It has become very essential to understand and manage the evolutionary responses of weeds to the

Table 1. Relative persistence of some of the herbicides in soil

< 1 month	1-3 months	3-6 months	>6 months
2,4 –D, Glyphosate, MCPA	Alachlor, Acetochlor, Ametryn, Amilofos, Bispyribac-sodium, Butachlor, Carfentrazone-ethyl, Dalapon, Halosulfuron, Metribuzin, Metamifop, Metsulfuron-methyl, Metolachlor, Oxyflourfen, Propachlor, Pyrazosulfuron-ethyl, Thiobencarb	Clomazone, Chlorimuron-ethyl, Diallate, Dithiopyr, Ethofumesate, Fluchlorine, Imazethapyr, Isoproturon, Metamitron, oxadiazon, Linuron, Pendimethilin, Pyrazon	Atrazine, Bromacil, Chlorsulfuron, Diuron, Diquat, Imazapyr, Methazde, Picloram, Simazine, Sulfometuron, Sulfentrazone, Trifluralin, Paraquat

herbicides to sustain the human and soil life. Characteristics of herbicide use that would increase selection pressure and the probability of the evolution of herbicide resistance, include herbicides with a single target and specific mode of action, increased activity and effectiveness in killing a wide range of weed species, long soil residual and season-long control of germinating weeds, and those which are applied frequently, over several growing seasons of the weed population without rotating, alternating, or combining with other types of herbicides. In reviewing the character of evolved herbicide resistance, we come across with the terms target-site versus non-target-site resistance. Evolved target-site resistance exists when herbicide(s) reach the target site at a lethal dose but there are changes at the target site that limit herbicide impact (see section below on Target-Site Herbicide Resistance). Evolved non-target-site resistance involves the mechanisms that minimize the amount of active herbicide reaching the target site (see section below on Non-Target-Site Herbicide Resistance). After expertly reviewing the literature, on traizine target site resistance, Artntzen *et al.* (1982) and Grondwald (1994) had concluded that how a single resistance mutation was able to evolve globally in 68 weed species. The resistance was due to the malfunctioning of PS(II) which inhibited the NADPH and ATP formation and carbon reduction cycle leading to carbohydrate starvation and oxidative stress (Fig. 3).

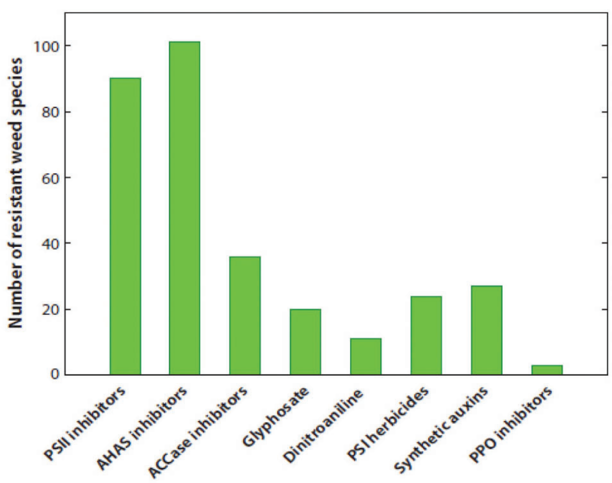

Fig. 3. Number of weed species that have evolved resistance to major herbicide modes of action (Heap IM, 2009)

Fundamental understanding of mechanism involved in evolution of herbicide resistance is yet to be discovered more at the genetic level. Together with the integrated management approaches, the molecular basis of the target and non- target site resistance have been studied in the past. Among the cultural practices, the use of herbicide mixtures and rotations was found to be more promising. Computer models (Fig. 4) have also been utilized as a tool for integrating the knowledge and hypothesis regarding the different factors and different processes that influence in the evolution of resistance. Inspite of the various management practices and overexploiting herbicide uses, it has been impossible to control the weeds from evolving herbicide resistances till date.

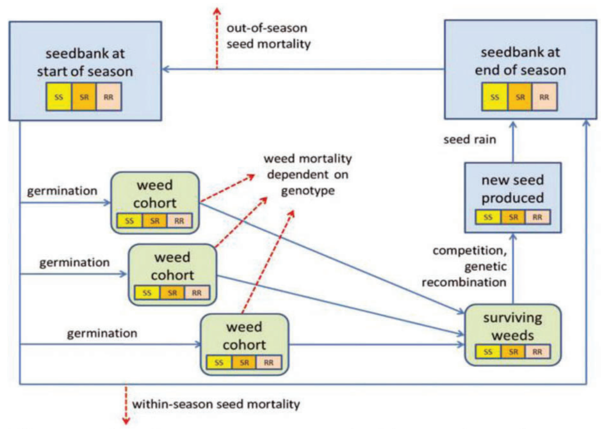

Fig. 4. Conceptual model illustrating the typical dynamics simulated in a model of herbicide resistance evolution in an annual weed.

The boxes usually represent the frequencies of different genotypes in the various weeds and seedbank subpopulations and cohorts; sizes or densities of the subpopulations may also be represented (here, three genotypes are represented: a homozygous susceptible SS, a homozygous resistant RR and a heterozygote SR, under the assumption of a single gene, but any number is possible). A number of steps occurring within a year are simulated, resulting in transitions between the various weeds and seed bank subpopulations and cohorts (solid arrows) or mortality (dashed arrows). At the start of the growing season, there exists a dormant seedbank consisting of seeds of different genotypes. One or more germination events occur, each resulting in a proportion of the seeds becoming a weed cohort (here, three germination events and cohorts are represented, but any number is possible). Herbicide application results in weed mortality, with a different mortality for different genotypes. Surviving weeds produce new seed, with the number of new seeds influenced by competition between weed cohorts and the crop, and the genotype of the new seeds determined by genetic recombination between the surviving seeds. This seed is added to the remaining dormant seedbank that has survived within-season seed mortality to form the end-of-season seedbank. Seed that survives out-of-season seed mortality then becomes the starting seedbank for the next season, and thus the simulation continues, with all these within-year processes simulated repeatedly to represent the population and evolutionary dynamics over several years. The process of evolution is driven by the differential survival of weeds under herbicide treatment, so that over a number of years the proportions and numbers of different genotypes will vary. Non-chemical weed treatments can be included in the model at almost any place; for example, harvest seed control techniques would result in a proportion of the seed produced being removed and not combined with the dormant seedbank, or higher crop sowing density might result in higher competition on the surviving weeds and thus lower seed production. Herbicide and non-chemical management can be assumed to be the same each year, or to vary from year to year Variable environmental conditions may affect any of the processes in the model (proportion of germination for each cohort, competition, seed mortality, herbicide efficacy, etc.), or an 'average season' can be assumed in each year. Typically, the dynamics are simulated over several years, and the changing genotype frequencies and population densities are tracked until either a set number of years is reached or some other stopping criterion (such as weed population reaching a critical density threshold) is reached (Renton *el al.,* 2014).

Current Weed Management Approaches

Weeds pose a great threat to the crop pants, hampering their natural growing conditions and reducing their biomass to sub-optimum level. Integrated management approach is considered the best for weed control as it lay equal emphasis on cultural, mechanical, biological or chemical control practices. But still, there are instances where weeds have managed to sustain all these practices and develop resistances against them. Of all the management approaches, chemical control has been proved to be more reliable, when we talk about production of food to meet the global demands. Herbicides are the maximum applied pesticides of all, throughout the world. Herbicide formulations are produced by the companies so that the weeds are controlled without imparting any lethal reactions to the associated crop plants. In spite of spraying excellent formulations of systemic herbicides available in the market till date, it has been impossible to eradicate the perennial weeds from the crop field and terminate their life span.

What if we Allow Nanotechnology to Intervene?

Nanotechnology is a novel, innovative, interdisciplinary scientific approach that involves designing, development and application of materials & devices at molecular level in nanometre scale i.e. at least one dimension ranges in size from 1 to 100 nanometres.

Nanotechnology is the tool that can be used to explore the hidden below surface organ systems of plants that is otherwise difficult to diagnose by man, especially when they are extended to deeper horizons. The focus towards nanotechnology has brought into light the use of nanoherbicides that can control weeds in an eco-friendly manner without leaving any residues in soil and environment. Having nanodimensions, these can be utilized for smart delivery of herbicides, thus preventing the weed growths that have become resistant to conventional herbicides. Due to the incredibly small proportions of nano-scale herbicides, they can easily blend with soil and attack seeds that are buried below the reach of tillers and conventional herbicides and thus killing them even when they remain viable and the get most favorable condition to germinate.

Nanoencapsulaion of herbicides

By now, it is clear that conventional herbicides have not been fully successful in eradicating the weeds. The deep root systems, i.e. rhizomes, tubers etc, manage to survive even after the continuous use of

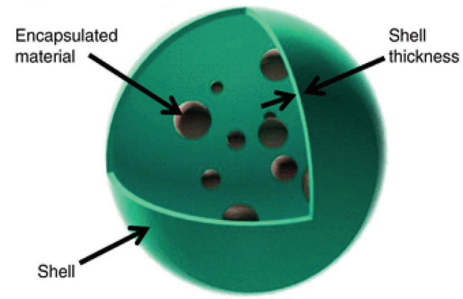

Fig. 5. Nanoencapsulated particles

systemic herbicides, thus failing the management approaches by farmers. Encapsulating herbicides with nanoparticles is a method that can boost the efficiency of herbicides by aiming at the specific receptor of the specific weed after entering into the root system and inhibiting glycolyis is thus starving them to death.

Nanoencapsulation is defined as the technology of packaging nanoparticles of solid, liquid, or gas, also known as the core or active, within a secondary material, named as the matrix or shell, to form nanocapsules.

Nanoencapsulation of herbicides is done with the purpose of its slow release, so that the active ingredients are released and available in concentration and with a duration that is just right to get an intended response from the weeds without getting any negative response from the crop plants. Nanoencapsulation is a membrane controlled system in which the herbicides are coated with any semi-permeable membrane that may be, organic or inorganic polymer, so that they are dissolved by the water and the active ingredients are released as a result of diffusion, osmotic pressure, ion exchange or degradation of matrices (Fig. 5). Rate of release of herbicidal suspension through diffusion is a membrane controlled system. Likewise, there are several systems developed for the controlled release of active ingredients after encapsulation of pesticides according to their properties. Fig 6 shows that how the release of herbicides from the coated materials, that is similar to the theory of nonoencapsulation of herbicides, are efficient in controlling the weeds as compared to the conventional herbicides application (Sopena *et al.,* 2009).

Wilkins (1990) first classified the materials used in encapsulation according to their degree of biodegradation: (i) Starch and systems based on

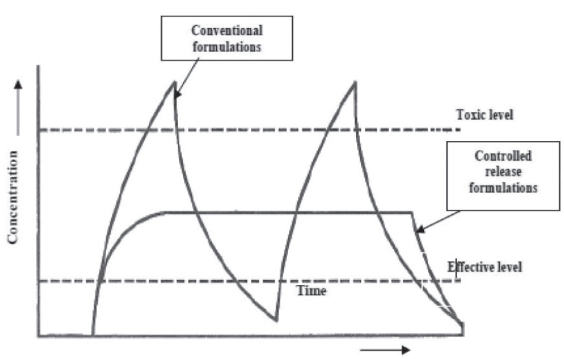

Fig. 6. Herbicide concentrations resulting from conventional and controlled release formulations.

amylose. (ii). Other polysaccharides (cellulose and derivatives, chitin, chitosan, dextran, alginate). (iii). Proteins (casein, albumin, gelatin).(iv). Lipophilic materials (rubbers and waxes). (v). Synthetic polymers (polyvinyl alcohol, polylactato, polyglycolato, other polyesthers, polyamines, polyamide-type acids, polyacryl amide). (vi). Miscellaneous (polyhydroxybutirato, tannins, polyhydroxyvalerato) lignins, resins and biopolymers modified by substitution, “crosslinking” or “grafting”. Of all these, the natural biodegradable polymers are mainly used due to their reduced environmental hazards.

Weed control by nanoencapsulated herbicides

Herbicides have long been considered a boon for the weed management in crop production. But when the treatments for one problem, becomes a problem for other components of the crop biosphere, it should be considered important to find out ways that would give better response. Nanoparticles encapsulated herbicides have been proven to release the active ingredients of herbicides slowly without imparting any toxic impact to the soil or soil biota. This technology can help to achieve weed control with long term sustainability of soil.

A review published by Green and Beestman (2007) on formulations of patents’ and commercialized agrochemicals, pointed out that of all the controlled chemical release products, the products made by the technology of nano or micro–encapsulation was most reliable for controlled and timely release of the chemicals.

Thus the nanoencapsulated herbicides can be trusted upon for their slow and adequate release of active ingredients, and this would reduce the quanity of active ingredients used for weed control as compared to the conventional herbicide spray.

The persistant activity is observed in many herbicides, one of which is atrazine which is used as a pre and a post herbicide. It can retain into the soil for months due to its slow degradation properties and have been responsible for contaminating soil and water. According to Oliveira *et al.,* (2015), the phytotoxicity of atrazine can be reduced when nanoparticles are used as the delivery or carrier system of the herbicide. The experiment performed by him included the formation of polymeric nanocapsules with the use of poly(epsilon-caprolictone) (PCL) as a carrier of atrazine after encapsulating. This had a magnificient reduction in the phytotoxic accumulation of atrazine in soil with increased herbicide activity because the mobiltity of atrazine was reduced (Pereira *et al.*, 2014). The same nanoncapsulated herbicide was found to decrease the photosystem II activity, root and shoot growth and biomass of the weeds *Amarathus viridis* and *Bidens pilosa* respectively, after 3 days of application (Sousa *et al.,* 2018) Figure 7 explains the reduction in photosystem II in case of application of encapsulated atrazine.

In another case studied by Cea *et al*., 2010, atrazine was incorporated into ethyl cellulose controlled release formulations (CRFs) by solvent evaporation and then nano-clays was added to modify the matrix. It was observed that the atrazine activity increased with reduction in leaching losses. The seedling death of the weed was more especially when nano-clay was added to the formulations.

Thus, the prolonged efficacy can give longer application intervals, minimizing the environmental impact. Using herbicides in lower concentrations and fewer amounts are desirable for the sustainable agriculture and less toxicity to environment. In this line, Maruyama *et al.,* (2016) have done an experiment to check the encapsulation efficiency of the two herbicides, imazapic and imazapyr, from the nanostructured polymer called chitostan that acted as carrier of the herbicides to the target site. This would minimize the dose of herbicide as well as increase the efficiency of the two herbicides. The result had shown that on encapsulation, the herbicide efficiency of both the herbicides was increased to 60% which were released at about 20-30% lower rate than the uncoated herbicides. Hence, the herbicide activity was increased and less phytotoxicity was observed in the soil. Herbicide toxicity causing an alteration in microbial population is a common problem where the persistence of herbicides in soil is for a longer duration. The residues are potential in altering the soil physo-chemical reactions that probably alters the microbial populations by directly interfering with their enzymatic activities. Some reactions are so lethal to even barren their population. Achieving smart delivery through the encapsulation of herbicides can battle with this problem by avoiding any interference with the microbes. Protection of herbicides inside the nano-capsules and their slow-release directly in the weed root zone is potential in causing minimum damage to the soil. This was explained again by Mayurama *et al.,* (2016), through his experiment in which the bacterial community attained minimum interference in their nitrogenous activity after the addition of encapsulated herbicides and also their population diversity and size also increased after 30 days of application as compared to the free herbicides.

Encapsulation efficiency is highly reliant on the chemical bonding between the herbicide and the nano-polymer used for encapsulating. The chemistry between them is still needed to be studied more for using this technology for a larger number of herbicides in future. Nanoformulations consisting of the biosensors can directly be driven to the receptors of the weeds without even the interaction with the main plants (Chinnamuthu and Kokiladevei, 2007) (Figure 8).

Nanoherbicides and herbicide resistance

Continuous use of the same herbicides is one of the

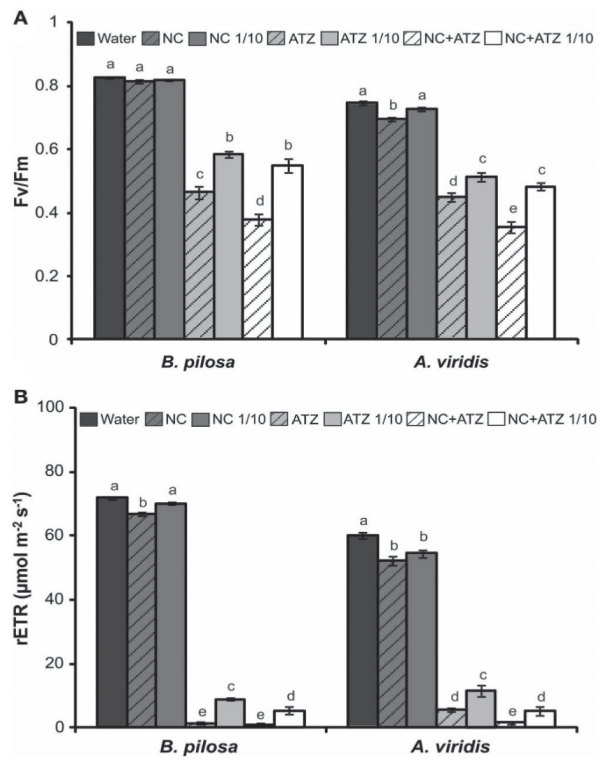

Fig.7. (A) Maximum quantum yield of photosystem II (Fv/Fm) and (B) relative electron transport rate of photosystem II (rETR) of Bidens pilosa and Amaranthus viridis leaves measured 3 days after the treatment with water (Water), concentrated (NC) or 10-fold diluted (NC 1/10) nanocapsules without atrazine, commercial atrazine at 2,000 g ha^{-1} (ATZ) or 200 g ha^{-1} (ATZ 1/10) and nanocapsules containing atrazine at 2,000 g ha^{-1} (NC+ATZ) or 200 g ha^{-1} (NC+ATZ 1/10). Data represent mean ± SE (n = 5). Different letters above the columns indicate significantly different values according to ANOVA followed by Scott-Knott's test ($P < 0.05$).

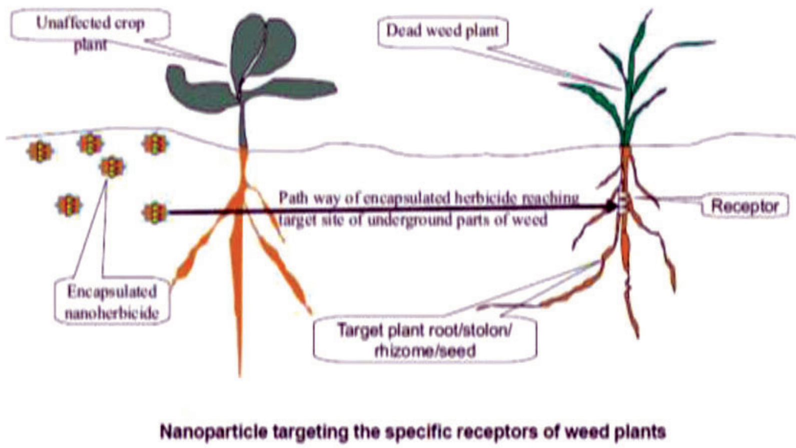

Fig. 8. Nanoparticles targeting the specific receptors of weed plants.

reasons of development of resistance by weeds. In order to avoid this, herbicide rotations or use of mixture of herbicides is advised to minimize the use of similar class of herbicides over and over again. Integrated weed management including cultural approaches such as crop rotation can also benefit. Nanotechnology on the other hand is one step ahead in avoiding the resistances build up in weeds. The use of nanoencapsulated herbicides or nano particles as carriers of herbicides are efficient in delivering the active ingredient directly to the target site of the weed plants, avoiding the residue build up in soil. The penetration ability of the nano-formulations is made so efficient that the herbicides would be carried directly into the plant metabolic system, where it could malfunction the targeted molecules or molecular pathways. The rational time –bound and smart release of the active ingredients avoids their residues to delocalize and metabolizes it before the resistance would develop. The nanocarriers required for preparing nanoherbicides provide short- and long-residual herbicides based on the need by averting the lethal dose at which the plant could develop herbicide resistance. Thus the nano formulations can be a boon in achieving the goal of sustainable and economic agriculture.

CONCLUSION

Weeds the problematic plants that create menace when not taken care of. In order to meet the global productivity demand, use of chemicals to manage pests are the easiest method. Herbicides have eased the problem of weed control but yet there are problems such as herbicide persistence in soil that have been declining the quality of soil. Apart from that, the trends among the weeds of developing resistance to the herbicides have been a serious issue. Nanotechnology with the unique way of herbicides release can give the promising results. The encapsulated herbicides can help in the easy delivery of herbicides to the weed plant that can avoid the residual accumulation in soil. The target specific release is also helpful in killing the weeds without even interacting with the crop plants. Nanotechnology is thus a boon that can further be developed with regard to the target site inhibition of the bio-chemical reactions of weed. The nano technology science is still in its nascent phase. Therefore, development of systems that would improve the release profile of herbicides without altering their characteristics and novel carriers with enriched activity without significant environmental damage is the focus areas that require further investigations.

FUTURE PROSPECTS

Nanotechnology is still in its growing phase. Its unique properties have made its use popular in various science and technology backgrounds. Although with the advantages, a scientific approach towards the knowledge of its potential hazards must also be achieved. The use of nano particles for weed management can be a miraculous achievement, but will its continuous use prove reliable in future? The potential hazard associated with the continuous use of these tiny particles is still unknown completely. We need a proof based scientific knowledge to rigid our faith on these nanoparticles for creating herbicide formulations so that the soil sustainability is not hampered. Further, apart from other field where this technology is used, a sound research and development is still required to sharp out this technology and make the best use of it in the field of agriculture also.

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