oxyacetic acid (MCP), are used today in the form of the sodium salt or an amine salt. In general, the information provided on amine salt formulations of 2,4-D holds true for 4-chloro-*o*-toloxyacetic acid and the current practice is much the same as that discussed under 2,4-D amines.

Dinitro herbicides, those containing compounds similar to the dinitro-*o*-*sec*-butylphenol (DNOSBP), are formulated both as amine solutions and as emulsifiable oil solutions.

The amine concentrate solutions of dinitro-*o*-*sec*-butyl phenol are pretty much restricted to alkanolamine salts, owing to the relative insolubility of the ammonium and alkyl amine salts. The less soluble ammonium dinitro-*o*-*sec*-butyl phenol can be used in the lower concentration selective-type formulation, while the alkanolamine salts need to be used in the higher concentration preemergent type formulation.

In formulations of salts of dinitro-*o*-*sec*-butyl phenol and related compounds it is necessary to think in terms of hard water protection where usage is in low volume equipment, again because of the insolubility of calcium and magnesium salts of the phenol at these concentrations.

Pentachlorophenol formulations are used in weed control generally in the form of oil solutions. Emulsifiable formulations can also be prepared. Solvents useful with pentachlorophenol are similar to those useful with 2,4-D esters, except that some of the heavier aromatic oils of dark color can be used.

Formulation Problems

The problems of the formulation chemist are many and varied and have only briefly been touched upon. A few of the things that the chemist needs to know about the intended use of the product before a formulation is devised are:

1. How will it be used—i.e., what amount per acre?
2. What are the physical characteristics of the chemical, including solubilities, toxicology, etc?
3. What are the corrosion problems connected with the use of the chemical?
4. How much money can be afforded in the formulation?
5. Where will the formulation be used?

Given the above information, it is possible to do a good job of formulating a herbicide. Trouble comes, for example, when it is thought that a chemical will be applied at say 1 pound in 100 gallons of water per acre and then equipment is developed to the point where people are applying the same chemical at rates of 1 pound in 5 gallons of water per acre. Such was the case with the amine salts of 2,4-D. Almost overnight a good product became useless as users began to apply it at rates for which it had not been designed. Now the formulation chemist finds that he must continually keep an eye on the developments in equipment and techniques used in the application of his products. Only by so doing can he be sure that his product is performing at peak efficiency.

Application equipment is important to the formulator in yet another way, in that any product put on the market must be tested so that undue wear or corrosion to the application equipment will become apparent and the adequacy of protective measures evaluated.

Such testing generally starts in the laboratory with carefully controlled tests on individual metals at the time field work begins. If a corrosion problem exists, steps are then taken to remedy it. Subsequent field testing in standard application equipment corroborates the laboratory findings. Considerable time and effort are put forth to ensure that the herbicides do not harm the application equipment.

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HERBICIDES

Residues in Soil

RICHARD J. ALDRICH

Division of Weed Investigations, Bureau of Plant Industry, Soils and Agricultural Engineering, U. S. Department of Agriculture, New Brunswick, N. J.

There is little direct evidence of a hazard associated with residues of any particular herbicide, but ample indication of the need for intensive studies of herbicides in soil. Critical factors are the effect on soil microorganisms and soil properties and removal of herbicides from soil. 2,4-D is relatively nontoxic to most soil organisms at concentrations ordinarily used for weed control. Certain other herbicides appear to be very inhibitory to microbial activity. The significance of inhibition of microorganisms for growth of subsequent crops has not been established. Herbicides or effects of herbicides are removed from soil by leaching, retention by the soil colloids, decomposition, and volatilization. Aqueous solubility affects leaching, but possible differential fixation of formulations and the carrier used may also have an influence. Chemical changes following application of a herbicide to soil complicate a comparison of formulations of different solubilities. Soil microorganisms decompose many herbicides; soil moisture, soil temperature, and other factors most conducive to microbial activity are conducive to herbicide decomposition. Volatility may be an important factor in the removal of a herbicide from soil.

HERBICIDE RESIDUES in soils have received relatively much less consideration than the effects of direct applications to plants. This placement of emphasis can be attributed to the very obvious effects of herbicide applications on plant growth, whereas the effects of herbicide residues in soil may not be readily apparent. Studies of herbicides in soils have been primarily concerned with the persistence of the herbicide as it may affect subsequent crops and with the effect of herbicides on microorganisms. No reports were found in the literature dealing with the direct influence of herbicides in the soil on soil properties.

Because injurious effects of the persistence of a herbicide in concentrations toxic to germinating seeds and young seedlings are obvious, this phase of residues in soils is not considered here.

Microorganisms and Soil Properties

Microorganisms The possible effects of 2,4-D on soil microorganisms have been investigated extensively. In general, a review of these investigations indicates that 2,4-D is relatively nontoxic to most microorganisms at concentrations ordinarily used in the field for weed control (*24*). On the other hand, there are several reports of inhibitory effects on organisms by 2,4-D at concentrations not greatly higher than those normally used for weed control (*12*, *18*, *23*, *24*, *31*, *39*, *40*). All microorganisms are not affected to the same degree by a particular herbicide, just as all higher plants do not react the same (*9*, *12*, *18*, *23*, *27*, *31*, *39*, *44*, *45*, *48*).

The selective action of 2,4-D on microorganisms suggests that the composition of the microorganism population may change with repeated herbicide applications. Careful studies of the rate of 2,4-D decomposition in soil indicate that there is a period of little or no decomposition immediately after treatment, followed by a period of rapid decomposition (*8*, *34*). Presumably the lag period is necessary for build-up of organisms capable of breaking down 2,4-D, which suggests that the number of such organisms is limited. Work with 2,4-D and 2-methyl-4-chlorophenoxyacetic acid (MCP) indicates that pretreatment with these compounds results in more rapid decomposition of subsequent applications of the compounds (*8*, *34*). This suggests an accumulation of organisms capable of using such compounds as a source of carbon. However, Audus (*8*) was not able to culture the organism responsible for 2,4-D decomposition in the absence of 2,4-D, which led him to postulate that 2,4-D is the only source of carbon utilizable by these organisms. Decomposition, it is suggested, is the result of either the selective proliferation of a mutant having suitable enzyme system or the adaptive development of such enzymes in normal soil. This work suggests that for 2,4-D and 2-methyl-4-chlorophenoxyacetic acid it is improbable that organisms other than those which utilize these compounds will be affected by repeated applications. Nevertheless, as suggested by Norman and Newman (*35*), the build-up of organisms capable of decomposing a particular herbicide may reduce the effectiveness of subsequent soil applications of the herbicide.

Little is known concerning the effects of herbicides other than 2,4-D on specific microorganisms and on the possible shift in microorganism populations which might be brought about by repeated herbicide applications. Kratochvil (*25*) found that sodium trichloroacetate, sodium pentachlorophenate, isopropyl-*N*-phenylcarbamate, and dichloral urea significantly reduced microbial activity; sodium 2,4-dichlorophenoxyethyl sulfate resulted in a significant increase in microbial activity. Microbial activity was reduced with rates of sodium pentachlorophenate and sodium trichloroacetate within the range of rates recommended for weed control with these herbicides. Harvey and Crafts's (*22*) observation that pentachlorophenol compounds did not decompose appreciably in soil stored under conditions conducive to microbial activity substantiates the finding of Kratochvil. Smith *et al.* (*40*) found that nitrate-forming organisms were extremely inhibited by ammonium thiocyanate and sodium chlorate at rates comparable to field applications for weed control. The writer has observed inhibitory effects of 4,6-dinitro-*o*-*sec*-butylphenol (DNOSBP) and its ammonium and amine salts on damping-off organisms in soil. The Pythium and Fusariam species were inhibited in soil which had been treated 6 weeks previously with 10 pounds per acre of the different dinitro formulations.

These results, although they are encouraging concerning the use of 2,4-D, suggest that the effect of new herbicides on microorganisms in the soil must be given serious consideration prior to the large scale use of the herbicides.

Soil Properties There are no reports of direct effects of herbicides on soil properties. A review of the effects of fumigants on soil properties and of certain phenomena associated with the removal of herbicides from soil gives a clue to the possibilities in this connection. Aldrich and Martin (*2*) found that the quantity of soluble calcium, and to a lesser extent magnesium and potassium, in saturation extracts in treated soil was increased immediately after fumigation with carbon disulfide, chloropicrin, dichloropropane, and dichloropropylene mixture. As microbial assays indicated that the fumigants had virtually eliminated microbial activity, these workers theorized that the pronounced chemical changes produced by the treatments must be attributed to a direct chemical reaction of the partial sterilizing agent with the soil and not to microbial activity. Robbins *et al.* (*38*) reported a close relationship between soil fertility and chlorate toxicity and also found that certain of the soil sterilants were fixed on the soil colloids. Subsequent investigations have shown that 2,4-D, 2-methyl,4-chlorophenoxyacetic acid, and dinitrophenol and -cresol compounds are held in the soil by binding forces of varying degree (*3*, *13*, *14*, *36*). These results suggest that the composition of a soil may be altered by a herbicide, if the fixation is effected through the exchange complex, by the replacement of an ion adsorbed on the soil colloid with a herbicide ion.

Obviously, any pronounced shift in the microorganism population which may accompany herbicide applications will affect soil properties.

Factors in Removal from Soil

Herbicides, or the effect of herbicides, are removed from soil by one or more of the following means: (1) leaching, (2) fixation by the soil colloids, (3) decomposition, and (4) volatilization. Conditions that affect these means of removal determine the persistence of a herbicide in soil.

Leaching The primary factors that affect leaching are the water solubility of the herbicide, permeability of the soil, soil texture, and the amount and intensity of rainfall.

There are few reports comparing the leachability of different herbicide formulations. With 2,4-D there are a large number of possible formulations ranging in solubility from esters, which are relatively insoluble, to alkanolamine salts, which are soluble to the extent of 300 grams per liter. In laboratory studies Aldrich (*3*) observed greater leaching of sodium and triethanolamine salts of 2,4-D than was observed with a butyl ester formulation. There was no difference observed in the leachability of the two salts. Stein and Staniforth (*41*, *42*) studied the leachability of a triethanolamine salt, butyl ester, acetanilide, and acetamide formulation of 2,4-D. The formulations ranked in the order of acetanilide, acetamide, ester, and amine in the order of increasing leachability. Although results of both of these investigators agree with the concept that leachability will decrease directly in proportion to aqueous solubility, it remains to be proved if the differences between formulations can be ascribed only to the degree of solubility. As shown by Aldrich, 2,4-D formulations vary in the tenacity with which they are held by the soil colloids. The differences observed in leaching of the formulations may be due, at least in part, to differences in adsorption or absorption by the soil colloids. With ester formulations of 2,4-D it is probable that the oil carrier itself restricts leaching.

Although there can be no question concerning the great differences in solubility of different 2,4-D formulations, Minarik (*30*) has pointed out that chemical reactions in the soil following a 2,4-D application will very quickly transform the applied formulation to new formulations determined by the cations present in the soil solution and on the soil colloids. In comparing the toxicity of pentachlorophenol and its sodium salt in soil, Harvey and Crafts (*22*) theorized that the alkaline reaction of sodium pentachlorophenate was reduced by the soil buffer, resulting in precipitation in a finely divided state. This is essentially the state of the parent pentachlorophenol applied directly.

Soil characteristics may influence leaching of a herbicide through their physical effects on water movement and through their chemical composition. Water moves more rapidly through sandy soils than through soils high in organic matter or in clay. It would be expected, then, that herbicides would move more rapidly from a sandy soil than from soils high in organic matter or clay. Again using 2,4-D as an example, it is known that heavy metal salts such as iron and copper are even less soluble than the acid, which suggests that the concentration of these heavy metal ions in soil will affect the leachability of 2,4-D. It might be expected that leachability of readily soluble 2,4-D salts would be less in alkaline soils as the result of the formation of less soluble calcium and magnesium salts, but reports on the effect of pH are inconsistent (*1*, *5*, *15*, *20*, *26*). It is possible that factors other than pH are responsible for the inconsistencies.

Fixation

In percolation tests Crafts observed that arsenic compounds (*38*), dinitro-*o*-cresol and its sodium and ammonium salts (*13*), the ammonium salt of dinitro-*o*-*sec*-butylphenol (*15*), and 2,4-D (*14*) were apparently held in the soil by the soil colloids, as these compounds moved less freely with percolating water in heavy soils than in light soils. The early work of Nutman *et al.* (*36*) suggested that 2-methyl-4-chlorophenoxyacetic acid was held in the soil. Weaver (*46*) found that appreciable quantities of 2,4-D were adsorbed on synthetic ion exchangers.

Aldrich (*3*) found that 2,4-D formulations varied in the extent to which they were held by a naturally occurring montmorillonitic type of clay. A butyl ester formulation was held to a much greater extent than a triethanolamine salt, sodium salt, and acid, although appreciable quantities of the latter three formulations were held by the clay. In addition, the ester retained by the soil colloid, unlike the remaining three formulations, was not readily removed by subsequent washing. This suggests that the mechanism of 2,4-D retention with the esters is completely different from that for the salt and acid. As the active part of the triethanolamine molecule, which ionizes in solution, is an anion, it is possible that fixation is effected through an anion exchange reaction with the clay mineral.

Present information on anion exchange reactions on clay minerals does not adequately explain the mechanism involved, although it is generally accepted that phosphate and certain other anions are adsorbed. It has been postulated that some of the hydroxyl groups in the hydrated clay mineral can be exchanged with phosphate ions by reactions analogous to the cation exchange reactions, which are for the most part equilibrium reactions (*19*). Considering results obtained with the triethanolamine salt of 2,4-D, decreased amounts removed with each subsequent washing suggest that an equilibrium reaction is involved. The ester formulations of 2,4-D, on the other hand, are nonpolar, undissociated molecules. There is no clear explanation for the possible fixation of such molecules.

Crafts has shown that substituted dinitrophenols (*15*) and cresols (*13*) and arsenic compounds (*38*) are fixed in the soil in a form available to plants. Weaver (*46*) and Nutman *et al.* (*36*), on the other hand, observed that adsorbed 2,4-D was not readily available to plants.

Decomposition

It would appear that decomposition of herbicides in soil is accomplished largely through the action of soil organisms. Numerous investigations (*6*, *7*, *10*, *11*, *16*, *17*, *26*, *28*, *32*, *33*, *37*, *43*, *49*) have shown that soil moisture, soil temperature, organic matter content, and other factors most conducive to microbial activity are also most conducive to herbicide decomposition.

There has not been sufficient work on decomposition of herbicides by microorganisms to provide any generalities concerning the effect of molecular configuration on breakdown. In a review of the action of microorganisms on hydrocarbons, ZoBell (*50*) states, "in general, aliphatic hydrocarbons are oxidized more readily than aromatic or naphthenic compounds. Within certain limits long-chain hydrocarbons are attacked more readily than similar compounds of small molecular weight. The addition of aliphatic side chains increases the susceptibility of cyclic compounds to microbial attack." Happold (*21*) reviewed the chemistry involved in the oxidation of aromatic rings by microorganisms. These two reviews on decomposition of compounds basic to many of the newer herbicides suggest some interesting leads for research.

The reports of decomposition of herbicides by microorganisms indicate that small differences in chemical structure have a pronounced effect on decomposition by organisms in the soil. Isopropyl-*N*-(3-chlorophenyl)-carbamate is readily attacked by microorganisms in soil (*32*, *37*, *43*), whereas 3-*p*-chlorophenyl-1,1-dimethylurea apparently is extremely resistant to attack. Audus (*8*) found that the organism capable of breaking down 2,4-D was also able to break down 2-methyl-4-chlorophenoxyacetic acid but at a somewhat lower rate. However, the organism involved in 2,4-D decomposition did not effect decomposition of 2,4,5-T (2,4,5-trichlorophenoxyacetic acid). If, however, the soil was first subjected to enrichment by the 2,4-D decomposing organism followed by enrichment by organisms from samples treated with 2-methyl-4-chlorophenoxyacetic acid, subsequent 2,4,5-T applications were rapidly broken down. The writer has observed that a triethanolamine salt of 2,4-D is decomposed more rapidly than an isopropyl ester formulation of 2,4-D. It is possible that the carrier in which the herbicide is applied may have an influence on decomposition by microorganisms.

Evaporation

Recent studies by Anderson *et al.* (*4*) suggest that rate of evaporation may have a pronounced effect on removal of a herbicide from soil. In a preliminary test isopropyl-*N*-phenylcarbamate lost 55% of its original weight when stored for 2 weeks at temperatures of 70° to 85° F. In a follow-up comparison of evaporation of isopropyl-*N*-phenylcarbamate and isopropyl-*N*-(3-chlorophenyl)carbamate it appeared that isopropyl-*N*-phenylcarbamate was relatively more volatile than isopropyl-*N*-(3-chlorophenyl)carbamate. Isopropyl-*N*-phenylcarbamate lost 2, 21, and 25% of its original weight through evaporation when stored for 2 weeks at temperatures of 60°, 70°, and 85° F., whereas no measurable amount was lost under storage temperatures of 16°, 32°, 40°, and 50° F. Samples of isopropyl-*N*-(3-chlorophenyl)carbamate stored in a similar manner lost an average of 12% of their original weight at 85° F., but measurable amounts did not evaporate at other temperatures.

In an experiment designed to measure the rate of evaporation of isopropyl-*N*-(3-chlorophenyl)carbamate when distributed evenly over a large surface, a piece of tinfoil was dipped in a solution containing isopropyl-*N*-(3-chlorophenyl) carbamate. The amount of isopropyl-*N*-(3-chlorophenyl) carbamate retained on the tinfoil was approximately equivalent to 1.5 pounds of the chemical per acre of surface. When the coated tinfoil was exposed to air at 83° to 85° F., most of the isopropyl-*N*-(3-chlorophenyl) carbamate evaporated by the end of the first 24-hour period. In a similar experiment there was no appreciable loss of weight of (4-hydroxy-5-isopropyl-2-methylphenyl) trimethylammonium chloride, 1-piperidine carboxylate.

These results indicate that for some herbicidal formulations evaporation may be a very important factor in the ultimate removal of the herbicide from soil. It has been demonstrated that formulations of 2,4-D vary in volatility (*29*, *47*). Wolf *et al.* (*47*) state that, in general, volatility of ester formulations of 2,4-D decreases as the length of the side chain increases.

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