# 3. Seed Coatings, Pelleting and other Innovative Techniques I

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TECHNICAL AND COMMERCIAL ASPECTS OF SEED PELLETING AND FILM-COATING

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## ABSTRACT

Coating techniques are described which are used to apply repeatably high and even pesticide loadings onto seeds. Pelleting is now well established for this purpose for sugar beet, and some vegetable seeds. Film-coating is a versatile new commercial technology for the treatment of a range of seed species and quantities with active ingredients formulated with liquid-based polymeric adhesives. Advantages include: bulk loadings which are reliably close to the target dose; more uniform seed-to-seed distribution (cv  $\sim$  20%); and durable, even coverage of material over the seed surface, eliminating loss of material during the handling or drilling of treated seed. Film-coating can apply relatively large quantities of material to seed, making possible the design of novel multi-pesticide seed treatments as part of broad-spectrum pest and disease control programmes. Film-coating also offers the potential to modulate pesticide release, and to change the nature of the seed surface affecting drilling efficiency, and imbibition kinetics.

#### INTRODUCTION

The treatment of seed with chemical additives is a longestablished practice to protect crops, especially from diseases and pests that affect establishment. The advantages are well recognized. Pesticides, nutrients and other materials - provided they can be loaded securely onto seed in adequate amounts, and with sufficient accuracy are ideally placed to be effective in the plant or the soil. Seed treatment is also an economical method of treating crops: considerably smaller amounts of material are needed to be effective compared to field application methods, where only a very small proportion of the active ingredient reaches its target.

In recent years, pesticides have been introduced for seed treatment which offer the possibility to widen the range of controllable organisms, and extend the protection phase further into the life of the crop. At the same time, public concern continues to mount about the perceived environmental hazards arising from the wasteful use of large quantities of pesticides, and increasingly stringent regulations to control their use are being implemented by Authorities. Partly in response to these trends and developments, there has been increased interest within the agrochemical and seed industries and research institutes in developing seed treatment "packages" as a part of integrated pest and disease control programmes.

In technical terms, any method of seed treatment should be able to reliably achieve target doses of pesticides, and distribute them uniformly from seed to seed. Materials should also be securely attached to the seed coat structures, so that active ingredients are not dislodged before planting, depriving the seed of full protection and risking blockage in the seed drill. The development of new seed treatment equipment and pesticide formulations, discussed earlier in this Conference, continue to improve conventional seed treatment technology. There comes a limit, however, where seed needs to be treated with more material than it can naturally carry. It is here that coating techniques - most notably pelleting and film-coating - have an increasingly important role to play. The aim of this paper is to review the present commercial seed coating scene, dealing with methodology (as far as is possible in what is a highly secretive trade) and performance characteristics in respect to pesticide application, and to consider areas where future developments may be made.

It is generally accepted that individual seeds should carry the same loadings to give the same protection to each plant. Erratic loading and seed-to-seed distribution of pesticides can have complex effects on pest and disease control, depending upon the nature of the active ingredient involved, its mode of action and the soil type (recently reviewed by Griffiths, 1986). Because there is not always a wide safety margin between the dose of pesticide that controls the pathogen or predator and the dose that harms the plant, poor pesticide recovery and distribution in a treated seed batch may impair the growth of some plants and give inadequate protection to others. Also research trials aimed at assessing protective and phytotoxic properties, and establishing optimum dose rates, may give misleading or uncertain results. Dusting-off of material, apart from loss of active ingredient, can both interrupt the flow of seed and cause blockages in drills, particularly in pneumatic ones (Kohsiek & Jeffs, 1986). There is also a health risk for those who handle the treated seed.

Analytical recovery analysis, e.g. using glc and hplc, provides an important tool for assessing the effectiveness of seed treatment equipment, in terms of both bulk chemical loading and individual seed-to-seed distribution of material. Old studies (reviewed by Graham-Bryce, 1973) revealed that the overall loadings and seed-to-seed distributions of insecticide on cereal grains at that time were very variable, and on average less than 50% of target. There is however very little published information in the succeeding literature on the performance of seed treating equipment. Griffiths (1986) in his recent review states, but does not document, that target doses are still seldom achieved in practice. Distribution on seeds can still be correspondingly poor. For example, cereal grains treated with insecticide using four types of seed treaters, including revolving drum and a misting chamber equipment, had coefficients of variation of individual seed doses in the range 35% to 50% (calculated by the author from Figs. 3.10 and 3.11 in Jeffs and Tuppen 1986), with disproportionate overloading of some seed (i.e. positively skewed distributions).

Loading problems also arise with treatments involving combinations of fungicides and insecticides, where it becomes difficult to apply the relatively large quantities of material needed, stably, accurately and evenly. Seeds with smooth waxy coats, like the testa of legumes and brassicas or the husks of barley (<u>Hordeum vulgare</u>) and grass species, are particularly difficult to treat. For example, in one analytical study the retention of thiram applied at similar rates of active ingredient to pea seeds was 38% for a dust formulation, and 80% for a slurry (Maude <u>et al</u>. 1986). The difficulty is increased where seed dust or wettable powder formulations must be used, which tend to lower seed loading limits by virtue of the large amounts of adjuvants they contain.

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Two examples of formulation loadings will suffice: (1) the treatment of onion seed with iodofenphos at the rate of 10g/kg seed to provide partial protection against first generation onion fly (<u>Delia antigua</u>), plus benomyl at 1g/kg seed for protection against neck rot (<u>Botrytis allii</u>), (Scopes & Ledieu, 1983); and (2), the treatment of Brassica seed with gamma-HCH at 45g/kg to protect against moderate attack by flea beetles (<u>Phyllotreta spp.</u>) plus iprodione or fenpropimorph at 5g/kg seed to eradicate leaf spot and canker (seed-borne <u>Alternaria brassicicola</u> and <u>Phoma lingam</u> - Maude <u>et al</u>. 1984). In both cases a fungicide at 1.5 to 5g/kg seed is also needed to protect against the soil-borne damping-off complex of diseases (<u>Pythium</u>, <u>Rhizoctonia</u> and <u>Phytophthora spp</u>.). To some extent, formulation of actives as suspension concentrates can help the loading problem by reducing the amounts of inert carrier material that needs to be loaded, but this is not always technically possible, e.g. if actives are not stable in aqueous conditions.

## By what criteria should one attempt to distribute active ingredients throughout a batch of seeds in order to achieve optimal biological benefits? Should one aim to load pesticides in equal doses

biological benefits? Should one aim to load pesticides in equal doses on individual seeds, or in doses proportional to the weight of each seed, or on some other basis? Equal dosing per seed would appear to be preferable for materials whose action depends on developing a critical concentration in the soil, where each plant station needs to receive the same dose regardless of the size of the seed placed there. This is particularly pertinent in crops sown at wide spacings, where "sharing" of chemicals between adjacent seed stations is not possible: indeed, for sugar beet seed it is common practice in many countries for pesticide application rates to be expressed on a "per unit" (10<sup>5</sup> seed) basis. However, it can be argued that current seed treatment practices are logically more in line with the second option - equal individual loadings normalised on a single seed weight basis - because recommended doses of materials are conventionally expressed on a weight per batch-seed-weight basis . Such a distribution pattern may indeed be preferable for materials which have a systemic mode of action, since lighter seeds tend to give smaller seedlings which may therefore need to take up less chemical (Longden, 1975). On the other hand, the smaller seedlings may produce less vigorous plants which perhaps need higher doses. There is clearly a need for more research on this subject.

#### PELLETING OF SEED

From its commercial introduction in the US in the late-1940s and in Europe in the mid-1960s, pelleting (Pillierung, enrobage, confettatura) has become a well-established commercial seed processing treatment. Its main purpose is to build-up small or irregularly-shaped seeds, which are difficult to singulate, into spherical, or near-spherical capsules. Pellets are used to sow a variety of crops which require precision drilling to achieve optimal plant spacings in the field or under cover, instead of the highly costly alternative of oversowing raw seed and subsequent thinning of established plants. Sugar beet (Beta vulgaris)

<sup>&</sup>lt;sup>1</sup>Thus in practice individual seeds in a batch of low mean seed weight currently receive proportionally smaller doses than do seeds in a batch of high mean seed weight. For consistency, it should follow that within any <u>single batch</u> of seeds the lighter ones should be intended to receive proportionally smaller doses than the heavier ones.

is the major seed species pelleted worldwide, and is sown extensively in this form, particularly in Western European countries (Durrant <u>et al</u>. 1986). Other crops of which a proportion of seed is pelleted include: red and fodder beet (<u>Beta vulgaris</u>), carrot (<u>Daucus carota</u>), celery (<u>Apium graveolens</u>), eggplant (<u>Solanum melongena</u>), endive (<u>Cichorium endivia</u>), leek (<u>Allium porrum</u>), lettuce (<u>Lactuca sativa</u>), onion (<u>Allium cepa</u>), parsnip (<u>Pastinaca sativa</u>), radish (<u>Raphanus sativus</u>), sweetcorn (<u>Zea mays</u>), tomato (<u>Solanum lycopersicum</u>), pepper (<u>Capsicum annuum</u>) and horticultural and fodder brassica spp. (<u>Brassica oleracea, B. rapa, B.</u> rutabaga), as well as smaller quantities of a range of flower species. Commercially, pelleting is available combined with various physiological seed preconditioning treatments, e.g. for celery, eggplant, lettuce, pepper and tomato seed. Seed encrustment - a variation on pelleting available in the UK which does not aim to produce a fully rounded shape - is used to treat onion and leek seed with pesticides.

Commercial pelleting is performed in specialised production plants, which are operated either by seed producers or as independent concerns. Companies currently operating include, alphabetically: Asgrow (USA), Cermer (France), Germain's (USA, UK, and Eire), Hilleshog (Sweden), Kleinwanzleben (E. Germany), Maribo (Denmark), Qualisel (USA), Sarea (Austria), Societe Europeenne de Semences s.a. (Belgium, Spain), Seed Dynamics (USA), Sluis (Holland, USA) and SuET (W. Germany).

## Pelleting techniques

The principle of seed pelleting is to use rotating mills,

cylindrical drums or pans, conveyor belts or other means to continuously roll the seed mass, whilst gradually adding water along with a powdered blend of coating material to build up incremental layers around the seed. The process is followed or accompanied by drying and grading to achieve the desired pellet size. Commonly, seed is pelleted in batches of up to 100kg at a time, though some systems for sugar beet use a continuous flow-through process, and the process has a relatively slow throughput rate overall. Typical weight-increase ratios range from 2:1 (for sugar beet and sweet corn) to about 30:1 (for onion and lettuce) and 150:1 for the tiny-seeded <u>Petunia</u> and <u>Lobelia</u>, but vary depending on species, pelleting system, and seed and pellet size.

#### Pelleting materials

Pelleting materials are selected for their adhesive and moulding properties during the wet milling stage (without causing seed doubling), for their strength when dry to permit handling during shipping and drilling, for not restricting germination, and for their chemical compatibility with required additives. Patent and research literature contains references to the use of:

filler materials such as cellulose powder, chalk, diatomaceous earth, limestone, non-ionic synthetic polymers, peat, perlite, sand, talc, quartz flour and vermiculite, and adhesives or binders such as calcium sulphate, clay, cellulose derivatives, polyvinyl polymers and starch.

However the precise compositions of the materials used in commercial processes are trade secrets. Recent discussions of these aspect of seed pelleting can be found in Longden (1975), Durrant et al. (1986). Also, Tonkin (1979, 1984) has briefly reviewed the wider literature.

#### Peroxide additives

Seed pelleting with calcium peroxide is commercially available in Japan and the USA for rice (Oryza sativa) cultivation, to increase

oxygen availability in submerged paddy conditions. Peroxides are also said to be used as additives in some coating processes for vegetables and sugar beet. Though not in commercial use, they have also been advocated for coating wheat (Triticum aestivum), barley (Hordeum vulgare), maize (Zea mays), soybean (Glycine max), ryegrass (Lolium perenne) to give similar physiological benefits after sowing in cold wet soils, or for their biocidal properties (Leaver & Roberts, 1984; Ollerenshaw, 1985; Langan et al. 1986).

## Pesticide treatments

Pelleting allows a degree of flexibility in the placement of additives. Pesticides can be mixed throughout the coating material: alternatively, they can be added in discrete layers - next to the seed, at an intermediate position, or in the outermost part of the pellet - to suit the location of target organisms, to separate chemically incompatible additives, or to minimise mammalian toxicity.

## Sugar beet

Fungicides are applied during pelleting to at least a portion of pelleted sugar beet seed in most countries. Major uses include hymexazol or maneb directed against soil-borne Pythium and Aphanomyces spp, and organo-mercurials or, chiefly, thiram for protection against seed-borne Phoma betae. A thiram steeping treatment, carried out just prior to pelleting, will be in commercial use in 1989 in the UK. Insecticides now in commercial use include methiocarb, carbofuran, carbosulfan and furathiocarb, for the partial control of soil-inhabiting seedling pests, such as wireworm (Agriotes spp.). (Durrant et al. 1986).

#### Vegetable seeds

Fungicide treatments are in some cases administered to the raw seed by conventional treatment methods, or by steeping, before pelleting is carried out (Maude, 1986). Alternatively, fungicides and insecticides are added during the pelleting process itself, in particular to treat onion, leek, brassica and carrot seed.



Fig. 1. Seed-to-seed distribution of insecticides applied to sugar beet within Germain's EB pelleting process: (A) methiocarb added during pelleting (cv = 23%), and (B) furathiocarb added to complete pellets using film-coating (cv = 14%). Data from hplc analyses of 120 pellets (3.50-4.75mm).

## Loading and distribution characteristics of pesticides

In principle, pelleting provides high recoveries of active ingredients, since loss by dusting off is essentially eliminated. Fig. 1A shows an example of the seed-to-seed distribution of methiocarb added during the EB process of Germain's: in this case, the coefficient of variation was 23%, and the bulk recovery was 98%.

There is very little published analytical information available on recoveries or seed-to-seed distributions of pesticide or other pelleting additives. Recoveries of the insecticide chlorfenvinphos from pelleted carrot seed reportedly ranged from 78% to 100% of the target dose (Thompson <u>et al</u>. 1982). Likewise, in a survey of the incorporation of the insecticide carbofuran in four commercial pelleting systems, Huijbrechts (1986) reported that recoveries ranged from 83% to 97% of the target dose over a range of 5.0g to 30.0g a.i./unit pellets (1 unit =  $10^5$ ). However, the coefficients of variation of individual seed dosages ranged from 14% to 62% in the four processes tested, and were considerably higher in one process. One commercial pelleting system is known to use film-coating to apply carbofuran to sugar beet pellets (Horner, 1985; Grimm, 1986).

## FILM COATING OF SEED

Film-coating (Inkrustierung, "Micropelleting") is a relatively new seed treatment technology, which is still emerging commercially. In its most developed form, additives are dissolved or dispersed in a liquid adhesive, usually a dyed solution of a polymer, into or with which seeds are dipped or sprayed before drying. Similar effects, which can also produce a film-coated finish, can be obtained by the addition of powder additives in a separate step after seeds have been treated with adhesive. The formulation can be changed at intervals to produce a multi-layered film-coat. After coating, additives are fixed on the seed surface embedded in an even layer, unlike conventional treatment with powders where material tends to be deposited in clumps on different parts of the seed. In contrast to pelleting, the increase in seed weight is small - typically 1% to 10% depending on seed size and the amount of additives involved - and the seed retains its natural shape. Characteristically, there is no dusting-off to cause loss of active ingredients during handling, or blockage in the seed drill. Seed flow is improved in comparision with seeds treated with formulations applied by conventional means, and the "bridging" problems associated with drilling rough-coated seeds, like carrot, can also be overcome.

Companies offering seed film-coating systems, in what is a fast-developing and competitive commercial arena, include at the time of writing: Canadian Seed Coaters, Conder Coatings (UK), Gustafson (USA), Heid (Austria), Hilleshog (Sweden), Nickerson RPB (UK), Sarea (Austria), Seedcote Systems (UK) and SuET (W. Germany), and their affiliates.

## Film-coating techniques

Film-coating depends on efficient exposure of seed to liquid to ensure an even coating. Also seeds must be kept separate during drying to prevent them sticking together, but not so vigorously as to cause mechanical damage. Various methods are available to do this.

## Conventional seed treating equipment

When amounts of material, and hence liquid, to be added are small and there is no need for immediate extensive drying to prevent seed clumping, it is possible to use conventional seed treating equipment, employing one of the variety of established methods to move or stir the seed mass (augers, revolving drums, seed falling in a curtain, rotation in a toroidal vortex) and to apply formulations to it (e.g., by dribbling, spraying, wiping, or using atomising discs) (Jeffs & Tuppen, 1986). Adhesives can be applied either in liquid pesticide formulations or to seed immediately before the addition of powder formulations, in appropriate equipment. Residual surface tackiness can be removed by dusting with absorbent powders, and any necessary drying can follow in a separate stage.

#### Fluidised beds

Where larger amounts of liquid are involved, fluidised-bed techniques using the Wurster process (Wurster, 1959; Hall & Pondell, 1980) have proved successful (e.g. Schreiber & LaCroix, 1967; Yada, 1983a,b; Maude & Suett, 1986a,b). The Wurster Process is also the basis of the commercial "SHR" process - "Spraying, Homogenization, Redrying" - used and licensed by SuET in West Germany (Horner, 1985; Grimm, 1986).



Fig. 2. Schematic diagram of (A) the Wurster fluidised bed process, and (B) the pharmaceutical-type coating drum, for film-coating of seeds.

In the Wurster process, seed contained in a vertical cylindrically-shaped chamber is fluidised and continuously circulated in an upward moving column of warmed air (Fig. 2A), into which the adhesive coating formulation is sprayed so that it dries almost immediately onto the seed surfaces, until the required amount of material has been added. The properties of the coating process have been described in detail for

seeds (Yada,1983a,b).

The Wurster process is versatile in terms of the sizes and quantity of seed that can be handled. Seed as small as Timothy grass (<u>Phleum</u> <u>pratense</u>) or as large as broad beans (<u>Phaseolus vulgaris</u>) has been successfully film-coated on an experimental basis in the author's laboratory, and amounts of seed from less than 0.5 kg to more than 50 kg of various species in a single batch. Processing time is determined by the liquid quantity and application rates: limited at one extreme by the relatively small surface area of large seeds, and at the other by the drying capacity of the low air flow rates needed to fluidise small seeds. The process has also been adapted to semi-continuous production design in the "SHR" system, where the seed is moved along a production line, receiving different coating layers at each successive fluidising station. This system is being used commercially to film-coat seed and pellets of sugar and fodder beet, and vegetable crops, with fungicide and insecticide (Horner, 1985; Grimm, 1986).

## Confectionery or pharmaceutical-type coating drums

In this system, liquid is sprayed onto seed (or other small objects) rotating in a perforated drum, through which warmed drying air

is drawn continuously (Fig. 2B). Equipment based on this principle is used for coating sweet or drug tablets with sugar or film-forming materials (e.g. cellulose derivatives). Though the use of this technique for seeds has not been documented in the literature, several companies are developing its potential, including Seedcote Systems for whom it has become the basis of their "Polycota" process. Quantities of up to 100 kg, or more, of small-seeded vegetable and grass species can be film-coated in single batches (Dennis & Newby, personal communication).

Because both the fluidised-bed and coating-drum film-coating processes are carried out in enclosed chambers, from which air is filtered before being expelled, those working in or around the treatment plant are not exposed to a health hazard from seed or chemical dust in the atmosphere.

#### Film-coating adhesives

As in pelleting, the film-coat layer must be made both durable and permeable to water and gases. Where drying is not carried out immediately during coating, the degree of adhesion must not be great enough to cause clumping or "tacking-off" from already coated seed. Reading of the literature suggests a variety of polymeric adhesives that can be considered for film-coating applications. Adhesives used for conventional seed treatment include: methyl cellulose, dextran, gum arabic, or vegetable or paraffin oils (Jeffs, 1973; van de Sandt, 1982; Scopes & Ledieu, 1983). The film-forming ability of water-soluble or water-dispersable polysaccharides and their derivatives (e.g. alginates, starch, galactomannans and cellulose) and of synthetic polymers (e.g. polyethylene oxide, polyvinyl alcohol and polyvinylpyrrolidone and their copolymers and related polymers) are exploited in a variety of processes, including pharmaceutical products industrial coating (Davidson, 1980).

Few reports have been published concerning the practicability of using such polymers for coating seeds, or importantly their possible phytotoxic or germination-inhibitory effects. Sauve and Shiel (1980) reported success using a water-miscible polyvinylacetate emulsion for treatment of vegetable seeds with the fungicide iprodione: there was low toxicity to turnip, carrot and cabbage seed, but onion seed germination was reduced. Strona & Dindorogo (1983) and Karpenko (1984) coated seeds of cereals, sunflower (<u>Helianthus annuus</u>), soybean and sugar beet using polyvinyl alcohol; Zinin & Imamaliev (1984) used polyvinylpyrrolidone to coat cotton (<u>Gossypium</u>) seeds; and West <u>et al</u>. (1985) tested methyl cellulose, polyvinyl alcohol, polyethylene oxide, and polyvinylidene chloride for film-coating of soybean. Also of possible relevance is the use of salts to harden aqueous gels of various polysaccharides, as has been devised for the encapsulation of somatic embryos (Redenbaugh et al.

## 1986).

For several years SuET have marketed a water-dispersable adhesive formulation for their "SHR" process. Similarly, Sarea have developed an encrusting agent, "Sacrust", to be mixed with the chosen additives, and an application process for it (Wieser, 1982; Bruckner, 1984) which has been used in Austria and West Germany. "Sacrust" needs to be dissolved in an organic solvent (e.g. methylene chloride), and applied in specialist machinery, for reasons of safety. More recently, in the UK specifically, commercial seed treatments based upon water-based formulations of fungicides and insecticides with polymeric binders have been made available in the "Polycota" process of Seedcote Systems, for the film-coating of carrots, onions, peas, oilseed rape and horticultural brassica seeds, using the special application equipment described above (Dennis & Newby, personal communication). Nickerson RPB, in conjunction with Shell Research, have also developed a water-based film-coating system for high-volume crops, such as wheat, barley and oil-seed rape (Bacon & Clayton, 1986). Film-coated rape is available from Conder Coatings, which is linked to Canadian Seed Coaters (Anon. 1984). The nature of the adhesives used in these commercial products are trade secrets.

## Loading and distribution characteristics of film-coating

Published recovery rates with film-coating are reportedly high. Up to 90% of the target dose has been recovered in commercial low-dose applications of fungicides (involving up to 5g a.i./kg seed), recoveries in the range 86% to 108% of target dose have also been found in experimental work involving applications of up to 16g a.i./kg seed of a fungicide/insecticide mixture to peas (Suett et al. 1985; Maude & Suett, 1986a; Salter & Smith, 1986). In trials using a prototype Wurster-type film-coating apparatus, Maude & Suett (1986b) found that loadings were closely repeatable, with high correlation values between target and achieved dose in two tests ( $r^2=0.99$ , n=10), compared to slurry and dust application methods, which gave erratic results ( $r^2=0.77$  to 0.96).

Seed-to-seed distribution characteristics are also narrow. Fig. 1B shows the distribution of an insecticide film-coated onto sugar beet pellets using the "Polycota" process, with a coefficient of variation of 14%. Distribution is to a certain extent dictated by seed surface area, as would be expected. The data in Fig. 1B relates to a sample of pelleted seed that ranged in diameter from 3.50 to 4.75mm: analysis of each of the five successive 0.25mm size increment fractions revealed smaller coefficients of variation, with a mean of 9.5%. Similar data relating to the film-coating of carbofuran insecticide on sugar beet pellets using the "SHR" process has been presented (Horner, 1985; Grimm, 1986). Likewise, in experimental trials on film-coated brassica seed, Maude & Suett (1986a) have found coefficients of variation in the range 16% to 20%, a proportion of which is accounted for by the variation in seed weight.

It is evident that there will be a need to revise seed application rates of pesticides where film-coating is to be used. Since the technique obtains repeatably high and even loading efficiencies of pesticides on seeds, one should question on at least two counts the advisability of using quantities previously established for use in conventional seed treatment equipment. Current recommended rates presumably tend towards initial overdosing, because allowance is in effect being made for the considerable amount of material that is lost from seed before or during sowing. Moreover, the increased degree of retention of materials trapped in a film is highly likely to modulate the biological effectiveness of pesticides in its own right (e.g. see Griffiths, 1986).

## Controlled release of additives

In some situations, the retentive capacity of film-coating may be developed as a positive attribute: to control the availability of materials, and thus modify their biological properties, either by using controlled release formulations in the spraying mixture, by modifying the polymer used, or by applying barrier layers. Data has been presented showing how the rate of release of carbofuran from film-coated sugar beet pellets can be modulated in the commercial "SHR" system (Horner 1985).

## TABLE 1.

Control of seed-borne <u>Ascochyta pisi</u> in combining peas, cv. Progreta, with fungicides applied by conventional seed treatment or film coating.

	Plants infec (% suppr	Yield (t/ha)	
Untreated	32.3	(0%)	1.8
Film-coat polymer alone	27.8	(14%)	
Conventional treatment A	5.8	(82%)	2.0
Film-coat treatment A	5.1	(84%)	2.1
Film-coat treatment B	0.16	(95%)	2.3

Treatments and doses (g a.i./kg seed): (A) Metalaxyl (68.5), Thiabendazole (37); (B) Metalaxyl (68.5), Thiabendazole (37), Thiram (30). Seed was infected with <u>Ascochyta pisi</u> (41%) and <u>Mycosphaerella</u> pinodes (3%). Extracted from Salter & Smith (1986).

#### Manipulation of seed imbibition

Film-coating has been used experimentally to apply a

water-impermeable plastic layer around seeds, in order to permit autumn sowing in situations where winters are severe and spring conditions for drilling are not dependably, contriving an artificial seed coat-imposed dormancy, which relies on weathering to crack the layer and allow germination as soon as conditions are suitable in the spring (Shreiber & LaCroix,1967; Nikol'skaya & Svirskaya,1984). In a similar vein, Tashkov & Furdzhev (1984) have coated maize seed with polysterol dissolved in chloroform to delay imbibition and prevent the initial leakage of solutes from damaged seed. Karpenko (1984), Krylov & Reshetnikov (1985) and Priestley & Leopold (1986) have coated maize and soybeans with the same purpose in mind. West <u>et al</u>. (1985) showed that a polymer film-coat using polyvinylidene-chloride can prevent deterioration and fungal penetration in soybean seed stored in conditions of high humidity, and somewhat surprisingly can promote imbibition.

## Broad spectrum seed treatments

Perhaps the major commercial potential of film-coating lies in the facility it offers to apply high chemical loadings of combined fungicides and insecticides, in order to achieve integrated pest and disease control treatments which cannot be applied reliably by other means. This review will be concluded with an illustration of successful film-coated pea seed treatments (Baughan <u>et al</u>. 1985; Salter & Smith, 1986) consisting of the following pesticides:

- Captan or metalaxyl to control seedling damping-off diseases (primarily Pythium ultimum, Rhizoctonia spp., Phytophthora spp.),
- Metalaxyl or fosetyl aluminium to control primary downy mildew (Peronospora viciae),
- Thiabendazole to control leaf and pod spot (seed-borne Ascochyta pisi, Mycosphaerella pinodes), (Table 1),
- Bendiocarb or furathiocarb for control of pea and bean weevils (Sitona lineatus) and thrips (Thrips angusticeps), (Table 2). Similarly, Baughan & Toms (1984) report that seed-applied bendiocarb increased pea yields by 5% and 18% compared to untreated peas drilled in soil incorporated with phorate.

Moreover, it has been found possible to control the late season foot rot complex (Phoma medicaginis var. pinodella, Fusarium solani et spp.), which hitherto has not been controllable by chemical means (Table 3).

## TABLE 2.

Control of Field Thrips (<u>Thrips angusticeps</u>) and of leaf notching caused by Pea weevil (<u>Sitona</u>) in combining peas, cv. Progreta, film-coated with fungicides and insecticides.

	Emergence (Plants per 3m)	Field Thrips Insects per 10 (% supp	Pea weevil Notches plants ression)	Yield (t/ha)
Untreated Film-coat treatment A	27.9 28.5 29.6	38 (0%) 1.5 (96%) 0.4 (99%)	155 (0%) 65 (58%) 0.5 (95%)	2.9 3.4 3.5
LSD	nsd	10 (27%)	33 (21%)	

Treatments and doses (g a.i./kg Seed): (A) Fosetyl aluminium (154), Thiabendazole (37), Captan (30), Bendiocarb (200); (B) Metalaxyl (68.5), Thiabendazole (37), Furathiocarb (400). Extracted from Salter & Smith (1986).

By using a film-coating approach, the amount of chemicals needed to protect the crop can be drastically reduced. Again, in the case of peas in particular, the seed coating treatment can in effect reduce insecticide usage from soil applied granules at the rate of 2.24 kg a.i./ha phorate to seed treatment at the equivalent of 0.32 kg a.i./ha bendiocarb (Baughan & Toms 1984; Biddle, 1986).

There is also an indication that lower amounts of chemicals can be placed more effectively on seeds by film-coating. Maude & Suett, (1986a) reported that the degree of eradication of <u>Alternaria brassicae</u> on infected cabbage seed by film-coating with iprodione was significantly better over a range of achieved doses, compared to slurry or dust applications of the fungicide. There was no adverse effect of the polymer coating system itself on germination, and interestingly the system was toxic in its own right to seed-borne A. brassicicola.

## TABLE 3.

Suppression of post-emergence foot rots (Phoma medicaginis, Fusarium spp.) in combining peas, cv. Progreta, film-coated with fungicides.

	Plants infecte (% suppress	Yield (t/ha)	
	TRIAL 1	TRIAL 2	TRIAL 2
Untreated	97 (0%)	30.3 (0%)	2.45
Film-coat polymer alone	_	32.0 (0%)	2.52
Conventional treatment A	28.5 (29%)	23.8 (21%)	2.99
Film-coat treatment A	29.6 (48%)	24.0 (21%)	3.16
Film-coat treatment B	29.6 (57%)	-	
		nsd	nsd

Treatments and doses (g a.i./kg Seed): (A) Metalaxyl (68.5), Thiabendazole (37); (B) Metalaxyl (68.5), Thiabendazole (37), Thiram Extracted from Salter & Smith (1986) and Salter (personal (30).communication).

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Zinin, T.; Imamaliev, A. (1984) (The use of pelleted cotton seed.) Khlopkovodstvo 1, 11-12. SEED COATING TECHNIQUES

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## ABSTRACT

After defining the term coated seed and the objectives in coating, the mechanism of coating-layer build-up is Three machines are described, together discussed. their variations which provide a range of with techniques for coating. Three types of drum coater batch methods for applying powdered or provide suspended solids. The need for adhesives is also highlighted in order to give strength to the coating. This method is the best for moulding the shape of Fluidised bed techniques provide a coated seeds. uniform gentle approach with good drying facility but with no opportunity to mould the shape of the seed. Spouted beds are also gentle with the seed, give good drying potential and will treat a wider range of seeds that the fluidised bed.

## INTRODUCTION

It is important initially to define the term coated seed in the context of this paper. It is considered to be a seed with a thin layer of material bonded to its surface, which does not substantially change the shape of the seed. The material may be polymeric or powdery, continuous or patchy, thick or thin relative to the size of the seed. Coated seed, therefore, may be considered to fit in between dust- or slurry-treated seed and pelleted seed in terms of added material thickness.

The quality of coated seeds is somewehat harder to define except in terms of the end objectives which, although they may vary between different growers, generally aim to achieve 100% germination and maximum yields. Quality of coated seeds may, therefore, be interpreted as uniformity of shape and size, pest and disease resistance, physical strength and good water absorption properties. This paper is addressed largely to the shape, size and strength aspects. These three physical properties are functions of the coating materials, the technique or skill of the operator and the particular process employed.

A further important marketing quality is, of course, appearance, which must appeal to the customer. Another objective, especially for fine flower and vegetable seeds, is simply to increase their size to improve handling. Begonia and tobacco, for example, are very tiny otherwise.

## MECHANISM OF COATING

The process or mechanism of coating is usually one of steady build-up of layers onto a seed nucleus. The seed must present an adhesive surface to the coating material. If a dry powder were added to a dry seed only a mono-layer of particles would adhere. The adhesive is commonly used to bond the seed to the coating material and this may be applied alternatively with the adhesive first or together as with a The seed mass must be kept mobile or else painted surface. the seeds will stick together and it must be able to dry off in readiness for the next layer to be added. If the cycle time is short then the amount added should be very small. Typically, a thin layer of clay using water as the adhesive might take 20s to dry between layers. Alternatively, a surplus of clay may be added to absorb all the excess water and prepare the seed immediately for the next layer, in which case the seed should be dried carefully after the full coating process.

If the seed is to emerge from the process already dried then the cycle time should be as long as necessary to provide a stable surface ready for the next layer to be added. Furthermore, a warm blast of air should be provided to dry the seed as quickly as possible. The rate of layer build-up is, therefore, clearly related to the rate of drying. The total process may well take from 10 - 50 minutes to complete.

The resultant seed shape depends to a small extent on the rate of particle growth, but only in as much as increased residence time tends to wear the edges of the coating and chip holes in it.

#### COATING MACHINES

#### Drum coater

The drum is essentially a mixer, whose axis may be horizontal, vertical or sloping (Figure 1).



Fig. 1: Three types of drum mixer (a) horizontal axis (b) vertical (c) sloping axis.

Variations occur in the design of each type. Baffles or lifting bars may be incorporated, an air purge may be used, the base may rotate with stationary walls and so forth. Essentially, however, the material is metered into the drum with the seed and thoroughly mixed.

The crucial features of the drum are its ability to give an overall mixing pattern without high shear zones and its size. The toroidal type, with vertical axis, rotating base and stationary walls, gives an excellent mixing regime with vertical and rotational movement and a uniform shear rate within the body of seed. Shear rate in rotational equipment of this type may be defined as:

#### Shear velocity Shear depth

This is a similar relationship to that in a rotational viscometer where shear velocity is the relative velocity between inner and outer layers of seed and the shear depth is the gap between these layers (Figure 2). Care has to be taken, therefore, when using equipment with paddles or flights so as to avoid high shear zones in seed layers between stationary and rotating parts of the coater.



Fig. 2: Section of seed layer in toroidal drum mixer.

Shear rate =  $\frac{V_2 - V_1}{d}$  where  $V_2$  = Velocity of outer layer of seed

 $V_1 = Velocity of inner layer of seed$ 

d = seed layer width.

If the drum axis is horizontal then the speed of rotation is limited by gravitational effects and care has to be taken to prevent centrifuging (Figure 3). The limiting rotational speed can be found by equating centrifugal to gravitational forces such that:



## Fig. 3: Horizontal drum mixer.

Lifters may be provided to prevent slipping of the seed in the base of the drum and may be angled to give end to end mixing. The size of drum is important in this case because a large drum carries a heavy mass of seed and coating which can crush a seed coat or mould it into shape to form a firm round pellet.

Angled drums are good mixers inherently whether flat pan or partly spherical (Figure 4). Hence, this type is used widely in the sweet and pharmaceutical industries for sugar coating and suchlike. The mixing pattern is very effective as shown (Figure 4).





(c)

Fig. 4: Angled drum mixer. (a) Spherical drum

- (b) Front view of flat pan
- (c) Side view of flat pan

If the drum is too small it cannot contain sufficient mass to mould the coating into a round pellet, although this can be compensated for by centrifugal forces. The toroidal machines can, for example, be speeded up to create adequate forces in the mass and thus start to change the shape of the coated seed. If the forces are too great, or if the adhesive is inadequate in the wet state the coating will disintegrate.

Drying air can be provided at the ends of the drum, but this is not very efficient. The dry strength can, however, be maintained steadily by slowly building up and drying the coating.

## Fluidised bed coater

Although a fluidised bed is mobile, especially in the more turbulent modes, it is not inherently a good mixer. A further means of mixing should be imposed upon it to give good transfer of materials across the whole bed. The coating is sprayed from above usually on to the fluidised bed of seed (Figure 5). One problem is that some seeds do not readily fluidise. Many vegetable seeds such as lettuce, peas, sugar beet, rape and brassicas fluidise well, but others such as carrot, onion and parsnip do not (Clarke, 1985). Cereals generally can be fluidised especially if agitated at the same time. Another problem of this technique is that the fluidising air is ascending while the spray material is descending which means that some of the coating is carried away upwards to waste filter or recovery.

The great advantage of the fluidised bed is the drying potential. It provides an ideal drying mechanism, which can reduce the cycle time considerably compared to drum techniques.



Fig. 5: Fluidised bed coater.

It is possible to calculate mean residence times and provide a continuous inflow and outflow of seed, but batch systems are easier to control especially with heavier coatings.

Fluidised beds are very gentle on the seed and cushion all contacts with a layer of air around each seed. This leads to good overall coverage of the seed although creases and cracks are often not coated and original shape of the seed is largely unaffectd. The energy requirements of a fluidised bed can be signifcantly higher than that of a drum by a factor of about 10 for a medium throughput. In principle, a fluidised bed would be used for a fairly quick uniform application of coating material where shape is not a significant factor.

Spouted Bed Coater



Fig. 6: Spouted bed coater.

Spouted beds were largely introduced through the work of Mathur and Epstein, 1974. They provide a fluidised bed type of application whereby the coating is sprayed on to the seed in a highly mobile zone just above the nozzle in the base of a cone (Figure 6). The seed is carried up into the fountain to descend back on the recirculating bed. It recycles back through the application zone every 1 to 30s depending on the bed depth and picks up another layer. It is important that the seed dries out sufficiently in the settling time so that it does not adhere to the vessel sides or to other seeds. Even so, due to the relatively short recycle time, the coating should be applied either at very low rates or intermittently. Further drying may be necessary afterwards. Total residence times in a spouted bed usually vary between 10 - 30 mins. If the spouting air is warmed the drying time and hence the residence time is minimised.

The physical abrasion in a spouted bed is slightly greater than in a fluidised bed, but this is not usually a significant factor. The original shape of the seed is largely retained, but with good overall coverage. In the spouted bed the seed shape does not affect the process so much as in the fluidised bed so that carrot, onion and cereal seeds can be coated easily. Care has to be taken with certain seeds such as peas which imbibe water very readily and swell so that when they are dried and shrink again the coating becomes detached and cracked.

Due to the upward current of air inherent in a spouted bed an air outlet must be provided at the top. Additives may escape if a suitable filter is not provided. The throughput of this type of machine is rather low compared to the drum method and a 300 mm diameter model will only coat a few kg per hour. This may, however, be adequate for many vegetable or flower seeds.

#### Other variables

The same range of adhesives and coating materials can be used in each of the machines. This paper is not so much concerned with this matter, but so long as an adhesive is soluble, usually, though not necessarily, in water, it can be used in all three machines. Similarly, the coating material can be applied as a slurry in all three but only as a dry powder in the drum machines.

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## EFFECTS OF FORMULATION ON THE PERFORMANCE OF FILM-COATED SEEDS

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## ABSTRACT

The development of the technique of film-coating has created a new tool with which to apply pesticides to seeds. The use of polymers and other additives can influence the performance of the applied chemicals and subsequent seed germination. Possible effects on germination are studied in detail but possible effects on the release of pesticides from coated seeds are seldom reported. This paper shows effects of formulation on the release of fungicides from vegetable seeds, and questions the desired mode of release for different groups of pesticides.

## INTRODUCTION

Film-coating of vegetable seeds is a technique which overcomes many of the limitations of seed treatments (Lord & Jeffs, 1971). By film-coating, desired doses of pesticide can be applied to any type of seed in a very uniform way, and stickers prevent loss of the pesticide during transport and handling.

The advantages of using seed as a carrier for pesticides have been illustrated by others (e.g. Furmidge, 1984) and are easy to recognize:

- dustfree products cause no problems in pneumatic sowing machines
- optimal protection of seedlings and plants by uniformly applied pesticides
- no phytotoxicity to the seeds and therefore no adverse influence on germination
- less environmental "load" by using less a.i. per hectare, compared to other ways of application

Zaadunie has been one of the first seed companies to appreciate the advantages of film-coating for growers and has dedicated an important part of their research to the development of this technique for all vegetable and flower seeds.

Research is concentrated in three areas:

- development of formulations for coatings and pills
- testing of treated seed for germination under laboratory and field conditions
- testing the efficacy of the applied pesticides (chemical and biological tests)

Potential risks of seed treatment, and maybe even more of filmcoating, are in germination reduction and in changed activity of the applied pesticides.

Germination, although of major importance, will not be discussed in this paper. Effects of coating on germination can easily be measured, and seed firms will not sell seed with reduced germination caused by the coating process. The effect of formulation on the activity of pesticides is much more complicated. In 1960 Bardner (Bardner, 1960) revealed the possibilities of modifying activity of insecticides by the use of polymers. Although no measurable difference was found by the use of different adhesives (Griffiths <u>et al</u>.1974), his results suggest that difference in distribution of pesticides after application with adhesives will influence their biological activity. Griffiths (1986) also noted some possible side effects of stickers on the

insecticidal action and concluded that the subject needs more study.

Research has been done with a new insecticide in sugar beet pelleting (Elmsheuser <u>et al</u>.1988). Release and efficacy of furathiocarb from pellets from 8 major pelleting firms in Europe was measured in different ways. Only one of the 8 pellets tested fulfilled all criteria for ideal performance.

As sugar beet pelleting has already been a subject of research for over 30 years, this illustrates the potential risks of pesticide use in formulations in such a little studied field as horticultural seed.

Effects of polymers on the release of a.i. has been studied in coating research at Zaadunie. The main effort has been concentrated on fungicides and some results are presented here.

#### MATERIALS AND METHODS

Effects of polymer concentrations for three main types of polymer have been studied with two fungicides (thiram and iprodione) on seed of three species (radish, carrot and witloof chicory). Seeds were film-coated with different amounts of polymer and standard amounts of fungicides per kg of seeds (radish, carrot and chicory each with 3g thiram (Tripomol 80%, Pennwalt) and 5g iprodione (Rovral aquaflo, 500g/1, Agriben). Polymers and fungicides were applied in one mixture. Fungicides were extracted from the seeds by soaking 10g of seed in 100ml water. Samples were taken at intervals in time, and after preparation analysed by hplc, with spectrophotometric detection at 212.8nm. The percentage of applied a.i. was calculated from the absorption values.

#### RESULTS

## Difference between seedtypes (Fig.1)

Using the same formulation and the same dose of iprodione, a difference was found between species. Release from chicory seed was faster than from carrot and radish.

## Effect of a.i. (Fig.2)

Two fungicides (iprodione and thiram) showed a different pattern of release from both carrot and chicory. Leaching of iprodione was faster than thiram.

This difference cannot be explained by the solubility in water (thiram, 30mg/1 and iprodione, 13mg/1).



Fig. 1 Release of iprodione from seed of carrot (o), radish ( $\blacktriangle$ ) and chicory ( $\bullet$ ).



Fig. 2 Release of iprodione (....), and thiram (-----), from seed of carrot (o) and chicoree (•).



Fig. 3 Release of iprodione from carrot seed coated with a) polymer A,b) polymer B or c) polymer C at concentrations of 0.0-20.0 g/kg



Fig. 3 Release of iprodione from chicory seed coated with d) polymer A, e) polymer B or f) polymer C at concentrations of 0.0-20.0 g/kg

## Effect of polymer concentration (Fig. 3a-3f)

Three polymers from different groups were used to evaluate effects on a.i. release. With carrot seed, release could be accelerated or delayed using different amounts of polymer A or B. With chicory release was always delayed if polymer A or B was used. The results for polymer C were inconclusive, but release seemed to be little influenced by polymer C. All results are given for iprodione.

## DISCUSSION

Interactions between seed coat and polymer depend on seed coat characteristics, polymer type and concentration and type of a.i. applied. The fibrous coat of carrot seed with greater pores might be more open to a viscous liquid compared to the much smoother chicory seed coat. At low polymer concentrations the a.i. possibly does not penetrate into the seed coat which facilitates release. At high concentrations the a.i. is bound in the polymer. Polymer concentration also effects viscocity and therefore the behaviour of the suspensions during spraying.

It is clear that polymers can be used to influence the effects of a.i. on film-coated seed. However this can only be used effectively if differences can be correlated with field observations. Controlled release of fungicides, herbicides and insecticides is well documented but to find relations between that research, and controlled release from filmcoated seed is difficult. Research to find the best release mode of a fungicide should be done in extensive field trials, because climatic factors and soil type are of major importance. As long as these relationships are not known, a pattern of release comparable to the standard treatment, which is known to be effective, is a safe aim.

Persistence of pesticides in soils is affected by four factors (Beynon,1973; Fisher & Robinson,1984): Leaching run-off, transfer to animals and plants, degradation and evaporation. The relative importance of these factors might be influenced by the use of filmcoating.

A fungicide to protect seed and seedlings against damping-off should become available directly after sowing, but insecticides to control pests which occur only some weeks after sowing should be released more slowly.

#### CONCLUSIONS

Companies using film-coating should be aware of possible side effects from the use of polymers. Sufficient research should be done to avoid failure of chemicals which are known to be effective. The development of formulations should always be related to the release pattern of the a.i. from the coating.

As the importance of film-coating in vegetable and flower seeds is increasing, a contribution from agrochemical companies, and from national research institutes to clarify relations between polymer type, mode of release and field performance seems necessary. REFERENCES

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## 1987 BCPC MONO. No. 39 APPLICATION TO SEEDS AND SOIL

## THE EFFECT OF POLYMER BINDER ON THE ACTIVITY OF INSECTICIDES APPLIED TO MAIZE SEEDS

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## ABSIRACT

Four insecticides were applied to maize seeds using two different polymer binders. A newly developed polymer system was compared to a standard polyvinylacetate/alcohol mixture. The insecticidal activity of these seed coatings was assayed in pot experiments using *Diabrotica balteata* larvae as the test organism. *D. balteata* feeds on maize roots and is often used as a model for the economically important *Diabrotica* species (corn rootworms). The new polymer system consistently improved performance in several soil types.

Insecticide residues on the seeds at the end of the experiment were analysed by hplc. The polymer binder which gave the lowest residues also gave the best control of *D. balteata* larvae. This result is discussed with respect to the controlled release of pesticides from seeds.

## INTRODUCTION

Polymers are at present used to improve the physical and mechanical properties of seed treatment formulations. Inclusion of a polymer binder may increase adhesion of the active ingredients to the seed and improve the uniformity of the treatment. The polymer may also make the surface of the treated seed smoother and thereby increase the flow rate through a seed drill (Bacon & Clayton, 1986, Toms & Blackett, 1983).

A further possibility which has been discussed is that controlled release of active ingredients applied as seed treatments may improve their biological efficacy (Baughan et al. 1985). It has been demonstrated that slow release of insecticides from seed treatments may increase systemic activity of the pesticide (Bardner, 1960). However, there is generally little evidence for the influence of controlled release on the efficacy of seed treatments. There is more data to indicate that slow release may alter the efficacy of insecticides applied to the soil in a granule formulation (Zeoli and Kydonieus, 1983). Slow release of active ingredient may give prolonged control from a lower concentration of the insecticide than would be required in a conventional granule formulation. However, the effect of slow release on the control of soil-living insects by insecticides applied to seeds may be different from the effect of slow-release in a granule formulation. This is because the distribution of seeds in the soil is quite different from the distribution of granules. In order to predict the effect of controlled release from seed systems, we need to understand the distribution of active ingredient from a series of uniformly spaced point sources. Our present knowledge does not allow us to make this prediction (Suett and Thompson, 1985).

In the present study, the effects of two polymer systems on the efficacy of several seed-applied insecticides were investigated in growth chamber experiments. The control of *Diabrotica balteata* larvae feeding on maize roots was taken as a model system. Damage to the plant, larval survival and insecticide residues on the seeds were measured during the course of the experiments.

## MATERIALS AND METHODS

## Seed treatment

Maize seed (Hybrid G4753) was treated with four insecticides (furathiocarb, terbufos, isofenphos, fonofos) at a dosage rate of 10 g active ingredient/kg seed. The maize seed was treated in 1 kg batches on a commercially available pelleting plate. Technical grade insecticide was applied in combination with sufficient inert absorbents and polymer stickers to produce a uniform coat. Two different polymers were used to bind the insecticides to the seed: either a standard polyvinylacetate-based product (PVAC) or a new polymer system (HYPAC) developed by CIBA-GEIGY (1986).

## Tests of biological activity

Three soil types were used in separate tests:

- A natural soil from Stein, Canton Aargau, Switzerland. This had been heat sterilised and sieved to give an agglomerate size of less than 5 mm. Its composition was clay 20.7 %, silt 13.7 %, sand 65.6 %, organic matter 3.2 %, pH 5.6. Moisture was adjusted to 20 % of dry weight.
- 2. An artificial mixture of fine quartz sand and milled neutralised peat (2 % of total by dry weight). The moisture was adjusted to 10 % of dry weight.
- An artificial mixture similar to (2) except that the neutralised peat comprised 6 % by dry weight. Moisture was adjusted to 20 % of the mixture.

Plastic pots, 2 litre volume, were filled with soil to within 3 cm of the top and one maize seed was sown 3 cm deep in the centre of each of 20 pots per treatment (i.e. per combination of insecticide and polymer). The pots were placed in a growth chamber with a relative humidity of 70 % in a 12 hour daylength, light intensity 30,000 lux, using 26°C day and 22°C night temperatures. Soil moisture was maintained at the original level by daily irrigation with a commercially available nutrient solution.

After two weeks of plant growth, 40 larvae of the banded cucumber beetle (*Diabrotica balteata*) were scattered on the surface of each of 10 pots per treatment. This insect species is commonly used as a laboratory model for the corn rootworm (*Diabrotica* spp.) which is a common soil insect pest of maize in the USA. For this test, second instar larvae were selected which had been reared on maize seedlings at 23°C for 13 days. The insects were allowed to feed on the plant roots for one week before the contents of each pot were separated into three components (roots, insects and soil) using a wet sieving technique. Living larvae were then counted and damage was assessed on crown roots growing from the stem-base using the 0-6 scale shown below:

## 0 = no damage

- 1 = slight feeding damage, no roots eaten through
- 2 = more than 3 places with feeding damage, no roots eaten through
- 3 = 1 2 roots eaten through
- 4 = more than 2 but less than half the roots eaten
- 5 = more than half the roots eaten, but at least 3 still connect stem and soil
- 6 = less than 3 roots connect stem to soil.

A further 10 pots per treatment were inoculated with larvae three weeks after planting. Insect damage was again assessed after one week of plant growth. In total, this then gave 2 observation times for each experiment at three and four weeks after planting.

## Chemical analysis of insecticides on seeds

Insecticides were extracted from single seeds by shaking for 10 min. with  $10 \text{ cm}^3$  of a 1:1 mixture of acetone and hexane. The concentration of insecticide was estimated by hplc using a filter-photometric detector. The stainless steel column (200 mm long x 4.6 mm internal diameter) was packed with Lichrosorb RP-18 (10  $\mu$ m). Flow rates, eluent systems and filter wavelengths were optimised in preliminary experiments using technical grade active ingredients of known purity (Burkhard 1986).

Ten seeds per treatment were analysed before the start of the experiment and then at the times of biological assessment. At these dates, seeds were carefully removed from the soil before wet-sieving and either analysed immediately or stored in glass phials at  $-20^{\circ}$ C until they could be analysed.

## RESULTS

## Test of biological efficacy

All insecticides reduced *D. balteata* damage, but the control level was influenced by soil type, formulation and the insecticide (Table 1). The use of the HYPAC binder gave less insect damage than the PVAC binder in 22 out of the 24 possible combinations of active ingredient, soil type and plant age. The effect occurred with all insecticides but was greatest where control by the PVAC system was poor (e.g. furathiocarb in the sand / peat mixtures, Table 1). There was a smaller effect of HYPAC where control by the PVAC system was good (e.g. terbufos in 98 % sand, Table 2).

Terbufos gave the largest reduction of root damage both in the HYPAC polymer system (range of scores 0.5 - 2.2) and in the PVAC system (range of scores 1.4 - 3.3). Isofenphos gave the least reduction of damage in both polymer systems (HYPAC scores 2.5 - 3.3, PVAC scores 4.0 - 5.3). There was no evidence that the ranking of insecticidal performance could be altered by the polymer binder.

Damage scores were little different at the two observation times. However, the plants had grown considerably during the week between observations and a high damage score on 4-week-old plants indicates more root feeding than the equivalent damage score on 3-week plants.

## Table 1

Insecticide	Polymer	Stein E Plant 3 3 w	arth Age 4 w	SOIL 98 % Plant 3 w	TYPE Sand Age 4 w	94 % Plant 3 w	Sand Age 4 w
Furathiocarb	PVAC	2.0	3.0	3.6	4.0	4.2	4.0
	HYPAC	2.6	1.4	1.0	1.2	2.4	2.6
Terbufos	PVAC	2.6	3.3	1.6	1.4	3.0	2.8
	HYPAC	1.8	2.2	0.8	0.5	1.4	1.2
Isofenphos	PVAC	5.3	4.0	4.0	4.6	5.0	4.0
	HYPAC	3.0	3.0	3.3	2.5	2.5	3.3
Fonofos	РVАС	3.6	3.8	3.2	2.5	4.3	2.8
	НҮРАС	3.0	2.6	2.0	1.0	1.4	3.2
Untreated check		5.5	5.0	5.3	4.4	6.0	4.3

Scores of damage caused by *D. balteata* larvae on 3- or 4-week-old maize plants grown from seed treated with insecticide/polymer combinations.

The effect of polymer binder on larval survival is less clear because survival in all treatments was often poor (Table 2). In the soil from Stein, in one test, larval survival was only 15 % in the untreated check. This may in part be due to the difficulty of finding larvae in this high clay soil type. Wherever a PVAC-containing treatment gave more than 3 % larval survival, then there was always a lower survival from the corresponding HYPAC treatment.

Owing to the generally good performance of terbufos, it was not possible to show a consistent improvement from the HYPAC binder. However, for isofenphos, where larval survival for the PVAC formulation was II - 43 %, then the corresponding HYPAC treatment always gave a lower survival (range 0 - 20 %).

## Table 2

Percent survival of *D. balteata* larvae on 3- or 4-week-old maize plants grown from seed treated with insecticide/polymer combinations.

Insecticide	Polymer	Stein Plant 3 w	Earth Age 4 w	SOIL 98 % Plan 3 w	TYPE Sand t Age 4 w	94 % Plan 3 w	5 Sand t Age 4 w
Furathiocarb	PVAC	1	5	1	24	8	55
	HYPAC	3	0	0	3	0	29
Terbufos	РVАС	1	3	1	1	0	5
	НҮРАС	2	2	0	2	0	4
Isofenphos	РVАС	11	16	11	36	13	43
	НУРАС	1	5	0	6	2	20
Fonofos	РVАС	6	5	1	2	3	18
	НҮРАС	3	0	0	4	2	12
Untreated check		15	48	69	61	59	71

## Chemical analyses of seeds

Seeds from the tests in Stein soil and 98 % sand were analysed (Table 2). In both soil types, at both observation times and for all insecticides, HYPAC formulations gave lower seed residues than the corresponding PVAC formulation. If results are averaged over all other factors, then residues on HYPAC treated seeds were 25 % of the initial dose, and 57 % on the PVAC treated seeds.

In the Stein soil, insecticide residues were higher on three-week-old than on four-week-old plants. In this soil type, averaged over all treatments, 52 % of the applied insecticide remained on the seed after three weeks and 35 % after four weeks. In the 98 % sand mixture, 39 % of the initial insecticide level was found on the seed at both observation times.

The initial loading of the seeds was variable: less insecticide was found on terbufos and fonofos treated seeds than on furathiocab and isofenphos treated seed. This loss of active ingredient is probably due to volatilisation of the insecticides during either the treatment process or storage.

Insecticide	Polymer	Before Test	Stein I Plant 3 w	SOIL Earth Age 4 w	98 % 5 98 % 5 Plant 3 3 w	and Age 4 w
Furathiocarb	PVAC	2.9	1.1	0.8	1.3	1.3
	HYPAC	3.0	0.4	0.2	0.4	0.6
Terbufos	PVAC	1.2	0.8	0.6	0.9	0.6
	HYPAC	1.0	0.4	0.3	0.2	0.1
Isofenphos	РVАС	2.9	3.3	2.2	2.0	2.6
	НҮРАС	2.8	1.5	0.8	0.9	0.5
Fonofos	РVАС	1.6	1.2	1.1	0.8	1.2
	НҮРАС	1.4	0.7	0.5	0.4	0.5

Amount (mg a.i./seed) of insecticides found on seeds before and after biological testing.

## DISCUSSION

Table 3

Seed treatments containing the HYPAC binder gave the best control of D. balteata. Smaller insecticide residues (compared to the PVAC polymer) were also found on seeds which had been treated with th HYPAC polymer. It can be concluded from these results that the best insect control was achieved by the system which released the most insecticide into the soil during the course of the experiment. Other studies have confirmed that the release rate of insecticides from HYPAC systems is higher than that from other film-forming polymers (Geissbühler *et al.* 1987).

*D. balteata* larvae do not usually feed on the seed itself, but rather on newly developing roots in the region of the stem base. Insecticide on the maize seed is therefore not directly active in controlling *D. balteata* larvae, although it may act as a reservoir or source from which the active ingredient must spread in order to control the insect. In this test system, the retention of the insecticide on the seed may be disadvantageous if the concentration of insecticide distant from the seed is thereby reduced. The insecticide must reach the location where the insect is feeding. In previous discussions of controlled release formulations as seed treatments, the potential of slow release has been emphasised (Bardner, 1960 Baughan *et al.* 1985), however, it would seem that the important issue is an appropriate distribution of the pesticide in the soil. The optimal rate of release will depend on the feeding behaviour of the insect, on the physical properties of the insecticide and on the soil environment.

This study was not sufficiently detailed to clarify the importance of these different factors. Subjects for further investigation in this test system should be the effect of soil type, soil moisture and physical properties of the insecticides. It is also necessary to investigate whether these differences in growth-chamber tests can be confirmed in the field.

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SOME ASPECTS OF FILM COATING AGROCHEMICALS ONTO SEED

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## ABSTRACT

Compared with conventional applications, film-coating chemicals onto seeds gave improvements in operator safety, seed-to-seed distribution of chemical and handling at drilling. However, where broad-spectrum fungicides were compared in conventional and coated applications, only one of the polymer coats, ie that physical lowest strength tested, with improved of the characteristics gave consistently good disease control of both seed borne and foliar pathogens. Coating was also the only effective high dose applying an practical method of treatment to pea seeds which gave insecticide/fungicide substantial yield benefits.

## INTRODUCTION

In recent years the technique of film-coating chemicals onto seeds has been adopted on a commercial scale by several vegetable and pea seed houses, oil seed rape merchants and by one cereal seed merchant, Nickerson Seed Specialists. Adoption has come through the advantages given by coatings eg improvements in both the target dose achieved and in the control of the seed borne pathogen Alternaria brassicae obtained when a standard oil seed rape seed treatment, iprodione, was applied by film-coating rather than conventionally by dust or slurry applications (Maude & Suett, 1986). Coatings have also extended the limits of seed treatment technology by allowing additional heavy doses of materials to be applied to the existing standard treatments, eg the addition of an insecticide to a fungicide pea seed treatment (Baughan & Toms, 1984 and Salter & Smith, 1986).

However, coatings can be formulated from several materials and applied at various strengths. This paper reports on how these factors affected the physical handling characteristics of treated cereal seed and the efficacy of the broad-spectrum fungicides(seed disinfectant and mildewicide) applied. The benefits of film-coating fungicidal and insecticidal pea seed treatments are also discussed.

## MATERIALS AND METHODS

## Chemicals and their applications to seeds

On cereals conventional dust applications of Baytan DS (fuberidazole 3%, triadimenol 25% at 1.5g product/kg) and Baytan IM (fuberidazole 3%, triadimenol 25%, imazalil 3.3% at 1.5g product/kg) and conventional slurry applications of Baytan Flowable (fuberidazole 2.25%, triadimenol

18.75% at 2ml product/kg) and Ferrax (ethirimol 40%, flutriafol 3%, thiabendazole 1% at 5ml product/kg) were compared with the same rates applied in seed film-coats (SFC). Film-coats were either fully soluble (SFCC) or insoluble based on a polymer formulation unique to Nickerson's used at two strengths, SFCA (high) and SFCB (low). Film-coats were applied at 25ml product/kg.

On peas a May and Baker insecticide carbosulfan 40% DB (dustless base) was applied at 1.87, 3.75 and 7.5g a.i./kg seed conventionally, as a slurry, and compared with the 3.75g a.i./kg rate film coated. All treatments were combined with the fungicide Aliette Extra (fosetyl aluminium 52.8%, captan 17.2%, thiabendazole 12.9%) applied at 2.9g/kg seed.

## Drill flow assessments

Tests were done in the laboratory using a Massey Ferguson 30 drill unit with the weight of cereal seed passed through after 25 turns being recorded. Five replicates were done for each treatment.

#### Seed-to-seed loading analyses

Seeds were taken from commercially treated batches and twenty seeds analysed from each using an ultraviolet spectrophotometric method of analysis. A standard seed treatment machine based on the rotary atomiser and falling curtain principle was used for the slurry application. Seed film coats were applied by the Nickerson seed film-coater.

## Small plot disease observation trials on cereals

The control of seed borne pathogens and mildew (Erysiphe graminis) were assessed in  $\ln^2$  plots. Leaf stripe (Pyrenophora graminea), smut (Ustilago nuda) and net blotch (Pyrenophora teres) were assessed by counting the number of infected tillers, smutted heads and diseased seedlings per plot respectively and Septoria (Leptosphaeria nodorum) by counting healthy and diseased emerged and non-emerged seedlings taken from one row in each plot. The leaf area affected by mildew was assessed on the Bayer scale of 0 - 9.

## Field trials for mildew assessments

Spring and winter barley trials were established at 4 sites. In all cases one site was located in Norfolk and the remainder in north and mid-Lincolnshire. Plots were  $1.2 \times 4.5m$  with three replicates per treatment. Mildew was assessed twice if possible, first when it became well established in the untreated plots and second about 7 - 10 days later. Light infections were assessed by counting the number of lesions per plant, or on larger plants the numbers on a standard leaf or tiller. Heavier infections were assessed on a standard leaf using the ADAS scale No 1.1.1 to determine the percentage of leaf area affected. Ten or five plants per plot were assessed on each occasion.

## Protein Peas

Insecticide safety and efficacy were assessed in 1986 by a) visual assessments for phytotoxicity made at intervals throughout the life of the crop (cv Progreta), b) emergence counts at growth stage (GS) 0.4 and 1.02 in three 0.5m quadrats per plot and c) counts of live and dead <u>Sitona</u> spp larvae found in five soil cores per replicate at GS 2.03. Whole plot yields were taken at 14% moisture. The trial was situated at Cold Overton, Leicestershire with four 1.9 x 2.5m plots per treatment.

Fungicide efficacy was assessed by counting the number of seedlings with primary infections of <u>Ascochyta pisi</u> produced by an 8% infected seed stock cv Birte. The trial was in north Lincolnshire with three 1.7 x 12m replicates per treatment.

## RESULTS

## Physical effects of coating chemicals onto seeds

Film-coating had several advantages over conventional applications. First the firm retention of chemical on the seed improved safety for the operator through the reduction of the dust at application, handling and drilling, which occurred with powder but also with slurry applications. Second more even seed-to-seed distribution of chemical was achieved (Table 1). Although both methods of application gave good average target doses, the distribution of chemical using the film coat was much more uniform. Third, faster trouble free drill flows were possible. Flow rates obtained with coated Bay+an flowable on barley and wheat seeds and Ferrax on barley seeds were 26, 19 and 14% higher respectively than with comparable conventional treatments due a) to less friction between treated seeds and b) no dust to impede or block the drill mechanism.

#### TABLE 1

Seed-to-seed distribution of a broad spectrum cereal fungicide

5	Percentage slurry application	of target dose film coat application
	104	0.2
Average	106	92
Range	33 - 397	46 - 124
95% conf. limits	59 - 152	82 - 102
sample SD	103	23
population SD	106	22

20 seeds analysed from each sample

## Film coats and the control of barley mildew

The composition of the coat used to apply broad-spectrum seed treatments may influence the systemic activity of those chemicals against foliar mildew attacks. In small  $(lm^2)$  disease observation experiments on the winter barley cv Igri in 1985, the effectiveness of Baytan DS was significantly (P = 0.05) reduced over the dose range tested when it was applied in a coat with a high polymer content (SFCA) rather than conventionally (Table 2). SFCA also significantly reduced the effectiveness of mildew control with Baytan DS on the spring barleys Golden Promise and Koru (1985), and of Baytan IM on the winter barleys Igri (1984) and Monix (1985) in other small plot disease observation trials.

TABLE 2

Effect of SFCA and conventional applications of Baytan DS on mildew control

triadimenol pp	m on seed	percent control*		
Conventional SFCA		Conventional SF		
290	272	92	69	
198	164	89	49	
140	123	72	54	

\* where 12.5% of the surface area of the 3rd leaf on untreated plants was infected by mildew. (triadimenol is normally applied at 375 ppm to seeds).

In 1986 the lm plot tests were repeated on the spring barleys Golden Promise and Triumph. In this trial Baytan DS treatments were also applied in a coat containing less polymer (SFCB) and in the fully soluble SFCC coat. However, no consistent trends in mildew control were seen between treatments. Mean values for the control of mildew on Golden Promise for SFCA, SFCB, SFCC and conventional applications of Baytan DS were 66, 60, 69 and 70% respectively and on Triumph 58, 66, 60 and 52% respectively. Meaned over both varieties the range of control achieved by the different applications thus fell between 62 and 65%.

Although both polymer coats gave good mildew control in 1986, because of the previous poor results with SFCA, only the low strength polymer coat, SFCB, has been proceeded with. SFCB has in fact proved itself an effective carrier of mildewicides on both spring (cvs Golden Promise and Golf) and winter (cvs Igri and Otter) barley crops grown in replicated field trials in Lincolnshire and Norfolk over a range of soil types and climatic conditions. Average mildew control with SFCB and conventional applications of Baytan flowable on winter barley (1987) were 72% and 66% respectively (means of 18 observations) and on 1986 and 1987 crops of spring barley 61 and 57% respectively (means of 18 observations). With another seed-applied broad-spectrum mildewicide, Ferrax, the average control with SFCB and conventional applications were a) on winter barley (1987) 78 and 74% respectively (mean of 56 observations) and on the 1986 and 1987 crops of spring barley 81 and 76% respectively (mean of 64 observations). None of the comparisons were significantly different.

Film coats and the control of cereal seed borne pathogens

## Leaf stripe

As with mildew control the SFCA applications of Baytan IM failed to control a 40% leaf stripe infection on the winter barley Monix in 1985. However, in 1986, SFCB applications of Baytan DS completely controlled a 40% infection on the spring barley Triumph which gave 34 infected tillers per  $m^2$  in untreated plots while the other application methods, conventional, SFCA and SFCC all allowed a small number of infected tillers (0.33, 0.67 and 0.33 per  $m^2$  respectively) to develop. Similarly in 1987 Baytan flowable and Ferrax applied both in SFCB and conventionally completely controlled a 40% infection on Triumph which gave 32.5 infected tillers per  $m^2$  in untreated plots.

### Loose smut

Infections were completely controlled by both coated and conventional applications of Baytan DS and flowable. In 1985 Baytan DS applied conventionally and in SFCA, SFCB and SFCC controlled a 2% infection on the spring barley Natasha which gave 27.3 smutted heads per m<sup>2</sup> in untreated plots . Similarly SFCB and conventional applications of Baytan flowable completely controlled infections on the spring barleys Natasha and Triumph (1987) and the winter barley Panda (1985 and 1987), which gave 15.3, 32.5, 10.5 and 67 smutted heads per m<sup>2</sup> respectively in untreated plots.

#### Net blotch

SFCA, SFCB and SFCC and conventional applications of Baytan DS gave good control, 85.7, 92.1, 95.7 and 87.3% respectively of a 30% infection in the spring barley Golden Promise. The level of control given by the coated applications was thus at least equivalent to that obtained with the conventional.

Septoria seedling blight

Coated, (SFCA and SFCB), and conventional application of Baytan DS dramatically increased (163 - 172%) the emergence of a winter wheat cv Vuka carrying a 40% infection of Septoria (Table 3).

#### TABLE 3

Septoria control with coated and conventional applications of Baytan DS

Application	Total seedlings per m <sup>2</sup> plot	Emerged healthy	Emerged diseased	Non emerged decaying	
SFCA	625	118	0	0	
SFCB	626	127	2	0	
Conventional	660	115	15	5.3	
Untreated	333	31	57.3	53	

Mean number of seedlings per lm length of row

Coating Baytan on to the seed gave excellent control of disease on the emerged seedlings and no diseased non-emerged seedlings were found. Conventional applications of Baytan gave good control but were not as effective as the coated in reducing disease on the emerged seedlings (significant at P = 0.05). The considerable improvements in emergence were probably due in part to control of <u>Fusarium</u>.

#### Peas

Conventional applications of Aliette Extra at 2.9g/kg of seed are approaching the limits of slurry applications to pea seeds. Adding a powder formulation of an insecticide, carbosulfan 40% DB, to this by conventional means is possible but causes chemical retention problems during handling and drilling. Coating alleviated these problems. Both coated and conventional applications of Aliette Extra plus carbolsulfan 40% DB were safe giving good emergence and no phytotoxicity on the cultivar Progreta (Table 4). Although the film-coat application at 3.75g a.i./kg gave excellent control of Sitona larvae it was not significantly better than the conventional applications. Both coated and conventional applications of Aliette Extra and carbosulfan 40% DB at all the rates tested gave significant (P=0.01) yield increases (124 - 137%) over the untreated. However, coatings are the only practical commercial method of applying such high powder loadings. In separate trials on the cultivar Birte coated and conventional applications of Aliette Extra plus carbosulfan 40% DB (3.75g a.i./kg) both controlled an 8% seed borne infection of A.pisi.

#### TABLE 4

Effect of conventional and coated applications of carbosulfan 40% DB on peas (cv Progreta)

carbos g a.i.	sulfan 40% DB ./kg application	Emer (pla ls	gence nts/m <sup>2</sup> ) t 2nd	% Sitona control
1 07				
1.8/	conventional	73	71	72
3.75	conventional	69	75	89
7.50	conventional	77	74	75
3.75	coated	74	72	97
untrea	ited	76	73	0

All carbosulfan 40% DB applications were combined with Aliette Extra at 2.9g/kg.

## DISCUSSION

Coated seeds offer definite physical advantages to agrochemical companies and growers. First increased safety, accuracy of application and improved drill flows may enhance the attractiveness of the product to growers and may thus increase its market penetration. Second is the ability with coatings to apply high doses of novel product mixes without expensive reformulation to permit conventional application. Reformulation may not in any case be cost effective or indeed practical. For the pea grower such mixtures offer the convenient placement of insecticide and fungicide into the soil via the seed giving substantial yield benefits. Many types of coat give the physical advantages listed. However, some coats eg SFCC, though they performed well biologically would fail since in a moist atmosphere they would become sticky and flow badly. Others eg SFCA and SFCB, although they gave similar physical characteristics to the coated cereal seed affected differently the biological performance of the products they carried. With SFCA the high level of polymer gave reduced mildew control but excellent <u>Septoria</u> and <u>Fusarium</u> control with Baytan DS. The polymer coat of less strength SFCB gave a highly effective compromise and conferred all the physical advantages combined with good control of both foliar and soil-borne pathogens. Therefore coatings have to be optimised for any particular task and if this is done physical and biological benefits will result.

#### ACKNOWLEDGEMENTS

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## 1987 BCPC MONO. No. 39 APPLICATION TO SEEDS AND SOIL

EVALUATION OF A SMALL SCALE FLUIDISED BED SEED TREATMENT APPARATUS

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## ABSTRACT

A small scale fluidised bed seed treatment apparatus designed to treat 5-10 g batches of seed has been constructed. Some advantages of using this method of application of chemicals to seeds over the more conventional "rolling jar" method used for small scale work were demonstrated. With the fluidised bed technique, large quantities of N,N-dimethylformamide could be applied to susceptible varieties without the significant damage to the seed which occurred using the rolling jar method. Within the range of conditions employed, more even distribution of chemical seed-to-seed and more complete overall deposition of chemical on the seed were obtained using the fluidised bed technique. If penetration of test compounds into the seed is required, the rolling jar method would be preferred. It is anticipated that the small scale, single vessel, fluidised bed seed treater prototype can be elaborated to give a useful multi-channel version suitable for screening purposes.

## INTRODUCTION

There is a growing interest in using seed as a primary delivery system for applying crop protection and enhancement products. At the screening stage for such materials, many thousands of unformulated chemicals, often available in milligram quantities only, may have to be applied to small quantities of seed.

A favoured technique for this application has been the "rolling jar" method, in which seed is rolled with solutions of the test compounds in a small glass jar. While this is a rapid and convenient technique, it does have disadvantages. For example, for certain classes of compound it may be necessary to use relatively large amounts of powerful solvents. Prolonged contact of seed with solvent which occurs in the rolling jar method may damage the seed.

An alternative technique is that of the "fluidised bed", in which test solutions are sprayed into a bed of seed fluidised by a current of air. In this method, the residence time of solvent on the seed is extremely short so that high volumes of solvent can be used if necessary without damage to the seed. Commercial fluidised bed machines exist (Aeromatic, Glatt, etc) but the smallest machines available typically treat around 200 g of seed as a minimum batch size.

We have, therefore, constructed a miniature fluidised bed machine which can treat 5-10 g quantities of seed and have conducted preliminary experiments to compare its performance with that of the rolling jar.

## MATERIALS AND METHODS

#### General outline

In the biological work, N,N-dimethylformamide (DMF) was applied to seeds under various conditions by the fluidised bed and rolling jar techniques. The seeds were sown and the effect of the solvent was assessed by comparing seedling emergence from the treated seeds.

In the physical work, a fixed mass of the indicator 4-nitrophenol (NP) was applied to a fixed number of seeds in every experiment. This amount was applied under various conditions by each method. After treatment, samples of seeds from each experiment were analysed for NP to determine the retention and seed-to-seed distribution of the indicator.

#### Fluidised bed

#### Treatment vessel

This was an all glass yessel with a treatment volume of 35 cm<sup>3</sup> and expansion volume of 160 cm<sup>3</sup>. The overall dimensions were 54 mm diameter x 140 mm height. The glass vessel fitted into a base incorporating the fluidising air plate.

#### Base

The base was made of 'Arborite'. It was 8 mm thick x 114 mm diameter containing a central well 3 mm deep x 54 mm diameter to take the base flange of the glass treatment vessel. A radial pattern of 96, 1.0 mm holes located centrally in the well within a 23 mm diameter circle formed the fluidising air inlet. A central 2 mm diameter hole was positioned to locate over the liquid spray nozzle.

#### Base support

This was a 280 mm high x 360 mm wide x 280 mm deep box with a circular nylon mounting in top face to take the treatment vessel base. The liquid atomising nozzle was fixed centrally in this mounting to locate precisely in, and protrude 1.0 mm through, the central hole in the fluidising air plate. The liquid feed was constructed of 1.0 mm diameter stainless steel tube located centrally in a 2.0 mm diameter nylon atomising air tube. Controls for adjusting fluidising air pressure and temperature, and atomising air pressure were set in the front face.

#### Pump

Liquid was pumped by a Watson-Marlow 501 U/M pump assembly fitted with 1.0 mm diameter silicone rubber tubing. It was controlled by a split speed/time unit to give liquid feed rates in the range 0.01-2.0 ml/min (in two phases if required).

#### Air supply

Both fluidising and atomising air were taken from the house high pressure supply. Fluidising air could be pre-heated to a given temperature  $+ 2^{\circ}$ C if required.

## Rolling jar

## Treatment vessels

These were wide-necked glass jars of 60 ml nominal capacity. The internal diameter was 37 mm, the height 72 mm. The jars were fitted with plastic caps lined with cellophane inserts.

## Roller

The glass jars were rotated on their longitudal axis by a Luckham "Multimix Major" bottle roller.

## Biological evaluation

Seeds of wheat (cv. Broom) and sugar beet (cv. Primo) were used in batches of 200. The seeds were treated with 0, 0.5, 1.0 and 2.5 ml of DMF (including a trace of 0il Red Tax dye as a guide to solvent coverage) using either the rolling jar or the fluidised bed. On the rolling jar, the seeds were rolled in 60 ml glass powder jars at 60 rev/min for 10 minutes at room temperature. On the fluidised bed, the seeds were fluidised for 10 minutes using a fluidising air pressure of 0.1 bar, and an atomising air pressure of 0.05 bar. The fluidising air was heated to  $30 \pm 2^{\circ}C$ . The different volumes were applied continuously over the 10 minute period by adjusting the flow rate of the peristaltic pump accordingly. There were also untreated controls which had no rolling or fluidising.

After treatment, the seeds were kept in closed jars overnight, before being sown in 70 mm pots of a soil-based compost which were placed in a glasshouse (minimum temperature  $20^{\circ}$ C). For each treatment there were ten replicates of four seeds per pot. Twelve days after sowing, the numbers of emerged seedlings were recorded.

#### Physical evaluation

A constant mass (5.0 mg) of the indicator 4-nitrophenol (NP) was applied to a constant number (200) of wheat seeds (cv. Galahad) in all experiments. The indicator was applied as 0.5 ml of 10 g/l, 1.0 ml of 5 g/l or 2.5 ml of 2 g/l NP aqueous solutions.

With the fluidised bed, the atomising air pressure was kept constant at 0.05 bar. The fluidising air pressure was maintained at 0.15-0.20 bar and the temperature at  $40 \pm 2^{\circ}$ C. The solutions were applied during 2 or 10 minutes. Dry, treated seeds were stored in sealed glass bottles to await analysis.

The same three NP solutions were applied to the seed by the rolling jar technique, the bottles being rolled at 60 rev/min for 2 or 10 minutes at room temperature. Damp, treated seeds were stored in sealed glass bottles pending analysis.

All the above experiments were replicated five times. In the case of the fluidised bed work, the 30 individual runs were conducted in a random order. Using the rolling jar method, the 15 two minute applications were run as one batch followed by the 15 ten minute applications run as one batch.

Thirty seeds from each 200 seed batch were selected at random and each seed was soaked in water (2 ml) for 30 minutes. In another experiment, six seeds were selected at random from each of the five replicates of the six rolling jar treatments. On this occasion, each seed was soaked in water (2 ml) for 48 hours. Samples (200 ul) from these solutions were analysed for NP using a Dynotech MR610 Autoreader with a 405 nm filter. Solutions from untreated seeds soaked in water (2 ml) and standard NP solutions were also analysed at the same time.

## RESULTS

## Biological evaluation

All wheat seeds were killed by treatment in DMF using the rolling jar technique, whereas emergence was not reduced by the fluidised bed application, even when using the highest volume (Table 1). Subsequent excavation of the pots confirmed that where no seedlings had emerged the seed had failed to germinate.

Sugar beet emergence was depressed to some extent by the highest volumes applied using the fluidised bed, but again the effect was much more severe when the seeds were treated in the rolling jar. The absence of any clear volume effect in the rolling jar treatment of sugar beet may have been because an excess of solvent remained after rolling, even with the lowest volume. Thus, the scatter in the rolling jar data for sugar beet may be the result of some seeds steeping for varying periods in the excess DMF.

#### TABLE 1

Apparatus	DMF volume	€ Em	ergence
	(ml)	Wheat	Sugar beet
Fluidised bed	2.5	93	70
	1.0	93	80
	0.5	93	83
	0	98	88
Rolling jar	2.5	0	68
	1.0	0	38
	0.5	0	45
	0	93	93
Control		93	88
LSD (P<0.05)		10	18

Seedling emergence percentage of wheat and sugar beet following treatment with N,N-dimethylformamide in either the fluidised bed or the rolling jar

#### Physical evalution

A detailed statistical analysis was performed on the raw data from the 30 min soak experiment. The means and variances for each replicate (30 values) were obtained. This analysis showed that the fluidised bed data were fairly normal, the rolling jar data were highly skewed with a long right hand tail and that the variances were very dissimilar.

It was found that the variability of the data was strongly dependent on the value of the mean for a given experiment. In order to compare the variability between experiments, a log transformation was used. This stabilised the variance and produced more normal data.

In summary, the analysis revealed:

- (i) The fluidised bed method produces less variable values than the rolling jar method.
- (ii) The mean values from the fluidised bed method are significantly higher than those from the rolling jar method.
- (iii) The magnitude of the difference between values from each method depends on the volume of solution used and the time of treatment.

The data from these experiments are summarised in Table 2.

TABLE 2

Mass of 4-nitrophenol (NP) recovered on wheat seeds after 30 minutes extraction with water, as function of treatment method and application conditions.

Method	Time (min)	Vol. <sup>(1)</sup> (ml)	Conc. (mg/ml)	Mean NP per seed <sup>(2)</sup> (ug)	SD	%CV
Fluidised Bed	2	2.5 1.0 0.5	2 5 10	19.9 22.8 19.9	2.7 3.6 3.7	14 16 19
	10	2.5 1.0 0.5	2 5 10	18.3 16.9 16.3	2.8 2.5 2.9	15 15 18
Rolling Jar	2	2.5 1.0 0.5	2 5 10	2.2 6.4 10.2	0.4 1.8 5.3	18 28 52
	10	2.5 1.0 0.5	2 5 10	1.3 3.9 8.2	0.3 0.9 2.1	23 23 26

(1) Applied to 200 seeds.

(2) Mean of 5 replicates, 30 seeds per replicate.

Theoretical mean value for 100% recovery = 25 ug NP per seed.

Seeds in this experiment were soaked for only 30 min, so it is probable that only chemical (NP) on, or very close to, the seed surface was dissolved into solution for analysis. In the rolling jar experiments, seeds remained wet with NP solution after treatment and were stored in this condition for at least 24 hours before analysis. During this time they could imbibe the excess liquid and presumably absorb NP. It was shown that longer soaking times gave no significant increases in NP recoveries from fluidised bed treatments, but significant increases were obtained from seeds treated in the rolling jar.

Analysis of solutions obtained from a 48 hour soak of seeds from all the rolling jar treatments gave the data in Table 3. It is immediately obvious that NP recoveries were much higher than were obtained from the 30 min soak, and that there was less sensitivity to treatment method. All NP recoveries were lower than the lowest obtained from the fluidised bed treatments. The pattern of variance was still the same, with the 2 min/0.5 ml treatment again giving a high coefficient of variation. The distribution of NP seed-to-seed was more normal than that obtained after the 30 min soak.

#### TABLE 3

Mass of 4-nitrophenol (NP) recovered on wheat seeds after 48 hours extraction with water, as function of application conditions in the rolling jar method

Time (min)	Vol. <sup>(1)</sup> (ml)	Conc. (mg/ml)	Mean NP per seed (ug)	SD	&CV
2	2.5	2	12.0	3.7	28
	1.0	5	12.1	3.5	26
	0.5	10	14.4	9.6	61
10	2.5	2	10.8	4.0	24
	1.0	5	10.9	2.7	22
	0.5	10	15.6	3.1	26

(1) Applied to 200 seeds.

(2) Mean of 5 replicates, 6 seeds per replicate. Theoretical mean value for 100% recovery = 25 ug NP per seed.

## DISCUSSION

The preliminary experiments to compare the fluidised bed and rolling jar methods for applying small amounts of chemical to small batches of seed, demonstrated some clear advantages for the former method. Generally, it is less sensitive to the application conditions used, it gives better recoveries of applied chemical and the chemical is more evenly distributed seed-to-seed.

A distinct advantage of the fluidised bed technique is the very short contact time of solvent on the seed. This allows the use of potentially damaging solvents which may be dictated by the solubility characteristics of test compounds. It is also known (from work not reported here) that this method can be used for co-application of thin coatings of inert film-forming materials, if required. These may be useful if it is needed to retain volatile, particulate or highly water soluble materials close to the seed during subsequent biological evaluation. As evidenced by the different soaking times required to extract NP from seeds treated by each method, the rolling jar (or similar) technique should be used if the materials under test need to penetrate the seed for their effect. This technique obviously allows long contact time between seeds and the applied solutions. In contrast, residues from the solutions applied by the fluidised bed tend to stay on the seed surface, at least for short periods after application (although this effect will also depend on the physical and chemical nature of the residual material and the type of seed).

Two advantages of the rolling jar method are its simplicity and rapidity. However, given a modest investment in equipment, the miniature fluidised bed machine described above can easily be modified to give a multi-channel version to allow efficient screening of many compounds.