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(57) Abstract

Sustained release of an agricultural active ingredient selected from trace elements, chelates and pesticides is achieved by contacting irrigation water with a solid matrix of water-soluble wax through which the active ingredient is dispersed and which also includes an erosion-inhibiting amount of particles of water-soluble gel blocking synthetic polymer.

PRODUCTS AND PROCESSES FOR THE SUSTAINED RELEASE
OF AGRICULTURAL ACTIVE INGREDIENTS

5 This invention relates to the sustained release of agricultural (including horticultural) active ingredients to plants as a result of contacting water that is to be used for irrigation of the plants with sustained release compositions which release the horticultural or other agricultural active ingredients over a prolonged period.

10 In regions where there is inadequate rainfall, or in artificially covered growth areas such as greenhouses, it is conventional to promote the growth of plants by conducting an irrigation process in which water is flowed to the plants or to the substrate in which the plants are growing or are to be grown. Thus the water may be sprayed
15 over the plants or it may be applied to the soil or other substrate (for instance a soilless compost).

Conventional nutrient fertilisers such as NPK fertilisers are made available to the plants either by
20 incorporation of the powder fertiliser in the substrate or, more usually, by dissolving the required amount of fertiliser in the irrigation water merely by mixing the fertiliser with the water. However it is not satisfactory to apply specialised and trace active ingredients such as
25 trace elements, chelates of pesticides in this manner since they are usually required in very low concentrations over prolonged periods, and it is not practicable to achieve this by direct distribution of the active ingredient into the irrigation water.

30 There have therefore been various proposals for providing sustained release of such materials. For instance in U.S. 4,845,888 the nutrient is incorporated in a water-soluble polymeric film which is covered by another polymeric film which includes water resistant polymer to
35 retard the degradation rate of the laminate. In WO91/01086 the active ingredient is absorbed into a thermoplastic microporous carrier. In GB 2,163,419 the plant nutrient is

impregnated into a polyurethane foam or other porous substrate. In WO91/03940 the active ingredient is incorporated into a matrix formed from an amylaceous melt. Thermoplastic or thermosetting polymer (eg polyethylene) may be included in the melt. Such processes have the disadvantage that they leave an insoluble residue, which has to be removed or else it contaminates the growing area.

This can also be a problem with other polymeric matrices, such as in the matrices made by polymerisation of the monomer as described in U.S. 3,933,458 (which have the additional disadvantage that it can be undesirable to polymerise a monomer in the presence of the nutrient) or the matrices described in Fertiliser Research 1994, 38, 1, pages 53 to 59 in which the polymeric matrix is hydrated in a solution of nutrient. This has the additional disadvantage that it is difficult to achieve absorption of sufficient nutrient in the matrix coupled with slow release from the matrix.

In GB 1,598,458 the nutrient is included in microcapsules which are bonded into tablets using a wax matrix. The matrix will release the nutrient but the release of the nutrient from the microcapsules will depend upon the structure of the microcapsule shell, and this requires careful control of the manufacture of the microcapsules.

In US 5,248,700 the active ingredient is adsorbed into a microporous polymeric powder and this is distributed in a melt of a degradable polymer. This requirement to adsorb the active ingredient into a microporous powder places severe constraints on the type and amount of active ingredient that can be used. Also, there will again be residues unless the microporous powder itself is made of a degradable material.

Numerous sustained release systems for a variety of other active ingredients are, of course, well known and include various mechanisms for controlling the rate of degradation or release of various cast, compression molded

or other products. Examples include US 4,927,636, 4,911,858 and 5,188,755. However none of these are concerned with the particular problems of sustained release of specialised agricultural ingredients and none afford the opportunity of achieving this sustained release using a product which can be wholly soluble or dispersible in water, easy to manufacture with a high active content and capable of giving controlled release over prolonged periods.

10 The use of erodible blocks to supply water treatment chemicals into a flowing stream is well known. In US 3,435,618 it is proposed to make molded articles of particulate polyelectrolyte flocculant in a matrix of water-soluble wax and to use these molded articles to give sustained release of the polymeric flocculant into a flowing aqueous stream. In order to increase the rate of dissolution, it is proposed in US 3,435,618 to include a simple inorganic electrolyte in the block, for instance ammonium phosphate or ammonium nitrate. In WO95/2179 we describe blocks containing anionic polymeric flocculant of a type such that the blocks function as agricultural soil conditioners. We also describe in WO95/21796 processes of flocculating suspended solids in a flowing stream by eroding a block comprising a water-soluble wax matrix and containing particles of a flocculant and optionally also containing a multivalent metal coagulant.

25 In WO96/02126 (not published at the priority date of this application) we describe certain agricultural irrigation processes wherein the irrigation water erodes a block of water-soluble wax matrix containing polymeric flocculant.

35 The invention may provide a system for the sustained release of specialised trace agricultural active ingredients from a composition which can have a relatively high loading of the active ingredient and which is relatively easy and cheap to manufacture and which can be eroded completely away so that no undesirable residues

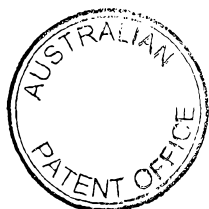


are left when the release of the active ingredient terminates. In particular, it is desirable to provide compositions that will provide the desired release over prolonged periods when included in whatever irrigation process is convenient for the particular plants involved, for instance drip irrigation or flooding irrigation. None of the proposals discussed above meet these objectives.

An irrigation process according to the invention for promoting the growth of plants comprises contacting water with a composition which releases agricultural active ingredient into the water and then applying this water to the plants or to a substrate in which the plants are grown or to be grown, wherein the composition is a sustained release composition comprising a solid matrix of a water-soluble wax through which is dispersed a water-soluble or water-dispersible agricultural active ingredient selected from trace elements, chelates and pesticides, and an erosion-inhibiting amount of particles of a water-soluble, gel-blocking, synthetic polymer.

An agricultural sustained release composition according to the invention comprises a solid matrix of a water-soluble wax through which is dispersed a water-soluble or water-dispersible active ingredient selected from trace elements, chelates and pesticides, and an erosion-inhibiting amount of particles of water-soluble, gel-blocking, synthetic polymer, said particles being present in an amount of 15 to 40% by weight based on the weight of the composition.

Thus, in the invention, we control the release of the trace element, chelate or pesticide from the water-soluble wax matrix by incorporating into the matrix particles of a polymer which creates a gel-blocking effect, as described below, and which is present in an amount to give the desired amount of gel-blocking thus the desired control of the rate of erosion of the matrix and, therefore, the rate of release of the active ingredient. The amount and type of the gel-blocking polymer which is required for any particular process will depend in part on the nature of the contact between the composition and the water. The contact



is generally achieved by flowing the water over the composition either continuously or intermittently and the rate of erosion will be selected according to the turbulence and duration of the flow.

5 One preferred irrigation process of the invention is drip irrigation in which the composition is positioned in or on the substrate and the irrigation water is dripped on to it. This method is particularly preferred for the irrigation of trees, shrubs and vines for example in Israel
10 and the USA. For instance a block (such as a tablet) of the composition may be placed adjacent each plant and the irrigation water may be dripped on to the block.

In other process, water is occasionally flooded through a growth area and the composition may be
15 distributed as blocks throughout the flood area (eg adjacent to each tree) so as to dissolve gradually during flooding. This is a preferred method for example with citrus trees in South Africa and field crops in Spain.

In a further irrigation process water is run down
20 furrows which are adjacent to lines of growing plants. In this method the composition may be placed at the top of each furrow. This method is used for example in the USA, South Africa and Australia.

In other irrigation processes irrigation water may be
25 sprayed onto plants or a substrate. Examples of this type of process are Centre Pivot and Wheel line irrigation (used in Saudi Arabia and the USA). In spray irrigation the composition is sited as a block at a convenient location in the water supply.

30 In all of the above irrigation systems water may be stored in a tank or holding area having an inlet and an outlet and from which the irrigation water flows (eg by draining or pumping) intermittently or continuously and the composition can be located in the holding area or its inlet
35 or outlet.

The invention is of particular value when the irrigation water is applied in excess, thereby saturating

the substrate with water with the consequential risk of losing active ingredient in the excess water which drains from the substrate.

5 The agricultural active ingredient may be any of the conventional specialised agricultural (including horticultural) active ingredients which need to be applied in small quantities over prolonged periods and which can thus be summarised as comprising trace elements, chelates and pesticides.

10 The water-soluble or water-dispersible agricultural active ingredient is usually present in the composition as a dispersion of particulate active ingredient in the wax matrix. In particular, the active ingredient should therefore preferably be capable of being present in
15 particulate, water-soluble or water-dispersible form. Preferably it is water-soluble.

Suitable water-soluble or water-dispersible powders which can be used in the invention are pesticides, by which we include materials such as insecticides, fungicides,
20 acaracides, nematocides and herbicides.

The preferred active ingredients for use in the invention are trace elements and metal chelates. Trace element nutrients include molybdenum (for instance as sodium or ammonium molybdate) and boron (for instance as
25 borax, boric acid or sodium tetraborate).

Metal chelates may be chelates of, for instance, zinc, iron, magnesium, manganese, copper, calcium or cobalt or various mixtures of these. Any of the conventional metal chelates used in agriculture (including horticulture) can
30 be used in the invention. Examples of common chelating agents are ethylenediamine tetraacetic acid, hydroxyethylethylene diamine triacetic acid, diethylenetriamine penta acetic acid, ethylenediamine dihydroxy phenylacetic acid,
35 ethylenediaminedihydroxymethylphenylacetic acid and ethylenediaminedihydroxysulphonylphenylacetic acid.

Products containing a trace element and/or a chelate

and/or a pesticide with other agriculturally useful active ingredient may be used as the active ingredient in the compositions of the invention. Blends of the trace element or pesticide with other materials may be used, for instance
5 blends of metal chelate with phosphate or phosphonate may be used when, as is conventional, it is desired to administer the chelate and phosphate simultaneously to the plant or substrate.

If the active ingredient is merely dispersed in the
10 wax matrix, without the water-soluble, gel-blocking, polymer adequate control of release from the wax matrix cannot easily be achieved. If the matrix has high solubility then very rapid release will occur and if it has low solubility, for instance as a result of including
15 hydrophobic wax material, then release may be uncontrollably slow and residual material may remain in the irrigation stream. This can be undesirable and may result in problems such as blockage of spray nozzles in spray irrigation processes.

20 In the invention, the rate of release of the agricultural active ingredient to the plant is controlled very easily merely by including an appropriate amount of particles of water-soluble gel-blocking synthetic polymer within the matrix. It is easily possible to select water-
25 soluble waxes and gel-blocking synthetic polymers that will dissolve completely and which are environmentally acceptable and thus by the invention a composition is provided which does not leave any undesirable residue, which is extremely easy to formulate from readily available
30 materials, and which gives slow release of the active ingredient into the irrigation water. Further, it is possible to position this composition at exactly the position where the trace element nutrient or pesticide is required, for instance in a drip irrigation process.

35 The wax can be any water-soluble substance or mixture which provides a water-soluble solid matrix at ambient temperatures but which can be melted at higher

temperatures. Usually the melting point of the wax is above 30°C and often above 40°C. It is generally undesirable to have to heat the wax to too high a temperature during manufacture of the composition and so preferably the melting point of the wax is not above 200°C and preferably it is not above 120°C. The wax may consist of a single material or a blend of materials, for instance a blend of waxes. Minor amounts of solvent or surfactant or other non-wax material may be incorporated in the wax matrix. Generally at least 50%, preferably at least 80% and most preferably substantially all the matrix is formed of a material such as a polyethylene glycol (for example PEG 1450, PEG 4000 or PEG 8000), a polyethylene glycol ester (e.g., PEG 8000 distearate), a fatty acid (e.g., stearic acid or sodium, ammonium or other stearic acid or fatty acid salt), a fatty acid amide (e.g., stearic acid diethanolamide) or a fatty alcohol.

Gel blocking is the mechanism which is known to be liable to occur whenever a sufficiently high molecular weight water-soluble synthetic polymer powder is added to water, unless precautions are taken to avoid it. It arises because the surface of the synthetic polymer particles starts dissolving and then forms a gel layer which tends to protect the incompletely dissolved polymer from further dissolution and tends to cause the formation of aggregates of partially dissolved polymer particles.

Usually the gel blocking effect which arises with high molecular weight synthetic water-soluble polymers is considered undesirable but in the invention we make use of it to control the rate of erosion of the wax matrix. Thus the water-soluble, gel-blocking, polymer appears to achieve the desired control of dissolution rate by providing a gel-blocking effect around the surface of the matrix and accordingly the polymers which are suitable are those which have the capacity to cause gel-blocking. Whether or not a polymer has this capacity can easily be determined merely by adding finely divided polymer (eg below 200µm) rapidly

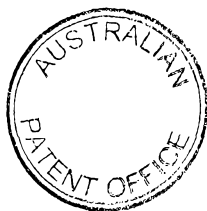
onto a small surface area of water. If this test shows that the polymer tends to hydrate to give swollen, mainly discrete gel particles then the polymer is not suitable as a gel-blocking polymer. However if the test shows that
5 the polymer does have a significant tendency to swell rapidly to form gel lumps (often with a dry core), then it can be tested for use in the invention by incorporating it in the wax matrix. A suitable gel blocking test is shown in Example 3 below and the preferred synthetic polymers for
10 use in the invention are those which give a gel blocking value of at least 8, preferably at least 9, and further preferably at least 10 and most preferably 12 to 20 or higher in that test.

It is necessary, in practice, to use a synthetic
15 polymer in order that predictable and controllable gel-blocking performance can be guaranteed. Although some batches of some natural polymers may have a tendency to gel-block, their properties tend to be unpredictable and to vary from batch to batch and so it is unreliable to rely
20 upon a natural polymer as an essential contributor to the gel-blocking effect. Preferably the gel-blocking effect is due to a synthetic polymer since this can be manufactured to a reproducible and consistent standard, thus permitting the formulation of sustained release compositions having
25 predictable sustained release properties.

The synthetic polymer must be water-soluble in the sense that it is eventually completely eroded away by the irrigation water.

The synthetic polymer is generally a polymer which has
30 been formed by addition polymerisation of ethylenically unsaturated monomer wherein the monomer is either a water-soluble monomer or monomer blend or is a water insoluble monomer and the polymer is subsequently solubilised or otherwise modified. For instance hydrolysed polyvinyl
35 acetates (i.e., polyvinyl alcohol) can be used especially when they are chemically modified by carboxylation.

The preferred polymers are polymers of water-soluble ethylenically unsaturated monomer or monomer blends. These



can be non-ionic but ionic monomers or monomer blends (for instance 5-95% ionic and 95-5% non-ionic) tend to increase gel-blocking capacity and so are usually preferred.

Any ionic monomer can be cationic, for instance
5 diallyldimethyl ammonium chloride or dialkylaminoalkyl (meth) -acrylate or methacrylate as acid addition or quaternary ammonium salt, for instance quaternised dimethylaminoethyl acrylate.

Preferably any ionic monomer is anionic

10 Anionic monomer that can be used can be any of the conventional anionic ethylenically unsaturated carboxylic or sulphonic monomers, such as sodium AMPS (US trade mark).

The carboxylic monomers are generally preferred and the use of sodium acrylate (or other acrylic acid salt) is
15 convenient.

Acrylamide is the preferred non-ionic monomer, but water-soluble non-ionic monomers can be used.

The intrinsic viscosity of the polymer is a contributing factor which influences its gel-blocking
20 characteristics. Preferred anionic polymers often have intrinsic viscosity in the range 10 to 30 dl/g, and suitable cationic polymers have intrinsic viscosities in the range 6 to 15 dl/g. However polymers with IV values of 4 or less can be used. All intrinsic viscosities are
25 measured by suspended level viscometer in 1N sodium chloride solution buffered to pH 7 at 25°C.

The polymer is usually formed in the absence of any added cross-linking agent but if desired a small amount of cross linking agent can be included in the monomers from
30 which the polymer is formed so as to render the polymer highly branched and to impart greater gel-blocking tendency than the corresponding polymer made in the absence of cross-linking agent. The intrinsic viscosity of the resultant polymer may not then be capable of accurate
35 measurement but if an apparent IV can be measured it should be within the ranges given above. The polymerisation conditions are preferably such that the IV would be within

the ranges given above if the cross-linker had been omitted.

The polymer is preferably present in the wax matrix in the form of particles having a size at least 90% by weight above 10 μ m and generally above 50 μ m. The particles can be coarse, for instance up to 1mm but generally at least 90% by weight are below 800 μ m and preferably below 300 μ m or 200 μ m. It is particularly preferred that at least 90% by weight of the particles should be below 100 or 150 μ m, and in particular the particles can be fines recovered from a reverse phase or gel polymerisation process.

The polymer particles can be made by conventional techniques such as gel polymerisation followed by comminution and drying (and often fractionation) but it is particularly preferred that the particles should be made by reverse phase polymerisation. Thus the preferred particles are fine beads made by reverse phase bead polymerisation of aqueous monomer droplets in a non-aqueous liquid followed by drying and recovering the fine beads (often as a fraction separated from coarser beads) from the non-aqueous liquid.

Instead of using beads or other particles above 10 μ m, it is also possible to use polymer particles below 10 μ m made by reverse phase emulsion polymerisation generally followed by dehydration to form a substantially dry dispersion of very small particle in non-aqueous liquid, typically having a size 90% between 0.01 or 0.05 μ m and 3 or 5 μ m.

The preferred way of making the composition of the invention is by forming a melt dispersion of the particulate polymer and the active ingredient in a melt of the wax and converting the melt dispersion into the desired shape of the agricultural composition and cooling. However when the particles are made by reverse phase polymerisation it is also possible to make the melt dispersion by forming a substantially anhydrous reverse phase dispersion of the polymer particles in a volatile

organic liquid by conventional techniques, mixing the wax into the organic liquid and evaporating the organic liquid by distillation at a temperature above the melting point of the wax.

5 The melt dispersion is cooled and shaped into the desired agricultural composition. For instance it can be cast as a large cooled block and then cut to size or it can be cast or extruded or otherwise shaped into the desired final size and cooled before, during or after the shaping.

10 The composition is usually in the form of blocks which can be small or large. Small blocks, which can be referred to as tablets or granules, typically have mean diameters of 100 μ m to 4mm and are suitable for use when they are scattered, for instance by hand or by a fertiliser
15 spreader. Larger blocks typically have volume of 10ml to 2l and are suitable for localised release (eg adjacent a tree) or when prolonged release is required.

20 The proportions of wax, polymer particles and active ingredient will influence the ease of manufacturing self-sustaining blocks or tablets and the rate of release. It is generally necessary that the amount of wax is in the range 30 or 35%, preferably at least 40%, more preferably from 40 to 70% by weight, and often around 45 to 60%, by weight of the total block. If the amount is reduced it can be difficult to make a self-sustaining block, and if the
25 amount is increased it can be difficult to achieve both controlled release and a sufficient amount of active ingredient.

30 The amount of polymer particles will be selected having regard to the particular polymer and the other components so as to achieve the desired amount of gel blocking. The amount is generally in the range 15 to 40% by weight of the total composition. If the amount is below 15% it is difficult to achieve adequate gel blocking and control of release. If the amount is too much then the
35 rate of release, and the amount of active ingredient which is to be released, can be undesirably low. Often the amount is not more than 30%.



The amount of active ingredient is generally in the range 1 to 50% by weight of the composition. For instance amounts of 1-10% are preferred for trace elements and 20 to 40% for chelates. If the amount is too high it can be difficult to form a self-sustaining composition and to achieve controlled release of the active ingredient.

It is sometimes desirable to provide a plant or plant substrate with a high dose of nutrient or other active ingredient for a short while and then a lower dose, for instance if a nutrient deficiency is observed. A preferred composition according to the invention comprises a multi-part block wherein one part is a casing or an upper layer which is contacted initially by the water and, after erosion of this part, exposes the core or a lower part.

The core or lower part is formed of a composition according to the invention and the outer or upper part is formed of a composition which provides more rapid release of the nutrient or other active ingredient. This part can consist solely of the nutrient, bonded to the block by wax or other suitable water-soluble binder, or can consist of a dispersion of the nutrient in a wax matrix which optionally contains particles of water-soluble gel blocking polymer in proportions and using materials such as to give faster release than from the core part.

The following are examples of the invention.

Example 1

A block was formed by melting together 0, 20 or 40 parts polymer, 20 parts of a proprietary iron chelate under the trade name Libfer SP and the balance, to 100 parts by weight, PEG 4000. The blending was conducted while the PEG 4000 was molten and the product was then cast into a block having a size of 10g and cooled. 80g soil was placed in a 10cm diameter pot, one block was placed on top of this and a further 50g soil was placed above it. The soil was then irrigated and the amount of iron chelate in the water which runs from the pot in the irrigation was measured. The



irrigation was repeated 15 times, and the cumulative amount of iron leached for the irrigations was determined.

This process was also repeated in a process in which the iron chelate, without any PEG wax, was put in the pot.

5 The polymer A used in this experiment was a reverse-phase bead copolymer of 70% acrylamide and 30% sodium acrylate having intrinsic viscosity 13 dl/g having size 100% below 125 μ m.

10 Polymer B is a copolymer of 70% acrylamide, 30% sodium acrylate with IV 24 dl/g. Polymer C is a copolymer of 60% acrylamide, 40% 2-Acrylamido-2 methylpropane sulphonic acid as sodium salt with IV 18 dl/g. Both products had a similar particle size of 100% < 200 μ m. Polymers B and C were preparing using a gel polymerisation process.

15 The results are shown in Table 1 in which the first horizontal line is the number of irrigations and the others are the cumulative % of iron leached from the block.

Table 1

Additive	1	2	3	4	5	6	7	8	9	10	15
0% Polymer A	9.3	38.7	58.2	68.1	72.6	75.7	77.2	78.5	79.4	81.2	83.6
20% Polymer A	0.4	15.0	23.5	30.2	34.5	37.7	40.5	43.10	45.4	49.8	56.2
40% Polymer A	0.2	10.5	16.5	22.4	26.3	29.7	32.3	34.7	36.8	40.3	46.3
Libfer SP	13.0	35.6	47.2	58.7	62.7	69.6	72.1	74.4	76.6	78.5	81.8
40% Polymer B	0.3	13.2	21.1	27.0	31.5	34.7	37.2	39.6	41.7	43.4	51.1
40% Polymer C	0.2	7.4	14.0	20.3	24.1	26.9	28.9	30.9	32.9	34.8	41.5

5

These results clearly demonstrate that the incorporation of the iron chelate in the wax matrix in the absence of polymer does not significant alter the rate of erosion (compare the 0% polymer and the Libfer lines) but that including the polymer particles does greatly reduce the rate of erosion.

Example 2

In order to compare the dissolution rate achieved with different polymers, 20g blocks were prepared by blending 50 parts molten PEG 4000, 30 parts iron chelate (Libfer SP) and 20 parts polymer fines having a size mainly in the range 10 to 200 μ m to form a 20g block. In all the experiments a homogeneous block was obtained but it was found in some experiments that when the amount of polymer was increased to 25% and the amount of PEG 4000 was reduced to 45% it was more difficult to obtain a homogeneous block.

Loose material was brushed off each block which was then put into a part-filled bottle containing 250g water which was then sealed and tumbled for 15 minutes.

The contents of the bottle were then drained through a sieve (to retain the block) and the block was then weighed so as to indicate the loss of the block on tumbling. The block was then tumbled again in fresh de-ionised water. After a further 15 minutes the block was removed and reweighed. This process was repeated until a total of 75 minutes tumbling had been performed. The results are summarised in Table 2 where the first horizontal line is the time in minutes and the remainder are the % of block dissolved.

Table 2

Additive	15	30	45	60	75
A	29.7	45.4	65.3	78.2	88.1
D	22.2	41.5	61.3	75.2	83.1
E	14.5	41.9	62.0	76.0	85.7
F	10.3	21.4	40.9	53.6	61.3
G	20.6	38.4	55.8	70.8	81.2

5 Polymers D-G are described in Table 3:

Table 3

Polymer	% Acrylamide	% Na acrylate	IV dl/g	Type
D	80.7	16.3	-	bead
E	100	0	12	bead
F	69.5	30.5	21	gel
G	89.2	8.84	11	bead

10 Polymer D is very lightly cross-linked.

It is apparent that the polymer having the highest sodium acrylate content and the highest IV, namely Polymer F, gives the greatest control of erosion and the longest sustained release of the active ingredient.

Example 3

15 In order to compare the dissolution rate achieved with other additives further blocks (15g) were prepared and tested as follows. All blocks contain 50 parts PEG 4000, 25 30 parts Libfer SP and 20% additive under test. Blocks were tumbled for 30 minutes in a similar manner to Example 2. After tumbling blocks were retrieved and the dry weight of the remaining material was measured. This weight was used to calculate the % of block dissolved. The results 30 can be summarised as followed:

Table 4

Additive	% Dissolved
Polymer H	36.6
Polymer I	39.6
5 Polymer J	42.0
Polymer K	47.2
Carboxylated PVA	51.4
Polymer A	59.0
PVA	71.3
10 Citric Acid	86.0
Sucrose	95.4
Manganese Carbonate	69.0
Further Libfer SP	67.6
Clay	100.0

15 Polymer H is a gel polymer containing 30% NaAc and 70%
Acm with IV 24.

Polymer I is a gel polymer containing 100% NaAc with
IV 13.

20 Polymer J is a gel polymer containing 95% NaAc and 5%
Acm with IV 16.

Polymer K is a bead polymer containing 3% DMAEAqMeCl
and 88% Acm with IV 7.

25 It is apparent that the various synthetic polymers
which are tested all had a significantly improved
dissolution-inhibiting effect compared to the other
additives. In order to correlate the dissolution
inhibiting effect with the gel blocking potential of the
polymer, Polymers A and H to K were subjected to the
following gel blocking test:

30

Gel Blocking Test

A glass funnel having a stem of 100 mm length and 7.5 mm internal diameter is positioned with the bottom of the stem 60 mm above the surface of 600 ml tap water in a 1 litre beaker. 3.0 g of the powdered polymer under test is put into the funnel while the bottom of the stem is closed with a finger. The finger is removed (whereby the powder drops onto a small area of the water) and timing is started. After 1 minute the beaker is removed and the contents are rapidly poured through a 4 mm mesh sieve above a 5 litre plastic beaker. The sieve is bounced on the top of the beaker for 30 seconds whereby polymer solution and slurry of individual particles will pass through the sieve but the sieve will retain any gel blocked polymer aggregates. The weight of wet gel (in grams) remaining on the sieve is then measured and is the gel-block value of that polymer. The process is repeated sufficient times to give 95% confidence limits.

The results were as follows:

20 Table 5

Polymer	% Solubility in Previous Test	Gel Block Value	IV
H	36.6	15.95	24
I	39.6	11.85	13
J	42.02	12.97	16
K	47.20	10.14	7
A	58.9	8.35	13

The direct correlation between increasing the gel blocking value and reducing the rate of erosion of the block is apparent from this table.

It can be seen from these results that polymers which give the highest gel blocking value (and which can be cationic or anionic) give slowest dissolution of the block but that polymers which give a lower gel blocking result give faster dissolution. Preferably the weight retained on

the sieve is at least 3 times the dry weight of test sample in this test (ie a value of 9). Non-polymeric materials such as citric acid and inorganic salts all result in unacceptable fast dissolution of the block.

5 Example 4

Potting compost was treated with lime (20 g/l) in order to induce iron deficiency in subsequently cultivated plants. Two maize seeds were planted in each 180 mm diameter pot (containing 715 g of limed compost). The
10 maize was grown under standard conditions until nine true leaves were present, at which time chlorosis was clearly visible. Pots were treated with either no iron chelate, standard iron chelate or an equivalent amount of iron chelate formulated within a slow release block. The block
15 tested contained 50 parts PEG 4000, 30 parts Libfer SP and 20 parts Polymer A from Example 1.

After treatment, half of the pots were watered such that the compost retained all of the water (pots watered to a set weight (1320 g) with no run off) and half were
20 watered such that the compost was saturated and run off occurred (pots water to set weight with an additional 600 ml of water).

The treatments are summarised as follows:

Treatment	Rate	Iron Content	Watering Regime
25 1. Control	NA	0	under saturation
2. Control	NA	0	over saturation
3. Standard chelate	0.23 g	0.0136 g	under saturation
4. Standard chelate	0.23 g	0.0136 g	over saturation
5. Chelate block	0.77 g	0.0136 g	under saturation
30 6. Chelate block	0.77 g	0.0136 g	over saturation

Three replicates were included for each treatment.

It would be expected that the under saturation regime using the standard chelate would result in good vigour

(good reduction of chlorosis) because the under saturation would mean that the dissolved chelate would not be washed through the pot but would instead remain in the pot.

The maize was assessed visually eight days after treatment. Vigour (resistance to chlorosis) was assessed against maize grown under standard conditions in un-limed compost the results were expressed as a percentage of the vigour in the un-limed control.

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Treatment	Vigour (% un-limed control)			
	Rep. 1	Rep. 2	Rep. 3	Average
1. Control	65	70	75	70.0
2. Control	70	70	65	68.3
3. Standard chelate	85	75	80	80.0
4. Standard chelate	65	70	65	66.7
5. Chelate block	75	75	80	76.7
6. Chelate block	80	80	75	78.3

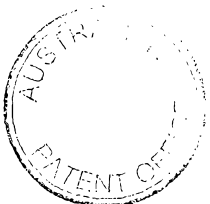
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This shows that underwatering with the standard chelate does indeed give good results and that there is no advantage by the use of a chelate block. However it also shows that overwatering with a chelate block gives substantially equivalent results whereas overwatering with the standard chelate gives very poor results. Accordingly, this data demonstrates that the use of the block does give sustained release of the chelate and that this is biologically beneficial when the irrigation system is such that the chelate would otherwise be flushed through the pots.

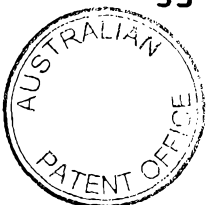
Throughout this specification and the claims which follow, unless the context requires otherwise, the word "comprise", and variations such as "comprises" and "comprising", will be understood to imply the inclusion of a stated integer or step or group of integers or steps but not the exclusion of any other integer or step or group of integers or steps.



THE CLAIMS DEFINING THE INVENTION ARE AS FOLLOWS:

1. An agricultural sustained release composition comprising a solid matrix of a water-soluble wax through which is dispersed a water-soluble or water-dispersible agricultural active ingredient selected from trace elements, chelates and pesticides, and an erosion-inhibiting amount of particles of water-soluble, gel-blocking synthetic polymer, said particles being present in an amount of 15 to 40% by weight based on the weight of the composition.
2. A composition according to claim 1 formed from 40 to 70% by weight of the wax matrix, 1 to 50% by weight of the active ingredient and 15 to 40% by weight of the polymer particles.
3. A composition according to claim 1 or claim 2 in the form of a block, tablets or granules.
4. A composition according to any preceding claim in which the wax matrix is formed of polyethylene glycol.
5. A composition according to any preceding claim in which the polymer particles have a size at least 90% below 300 μ m.
6. A composition according to any preceding claim in which the synthetic polymer is an addition polymer of water-soluble ethylenically unsaturated monomer or monomer blend.
7. A composition according to claim 6 in which the polymer is selected from anionic polymers having intrinsic viscosity 10 to 30 dl/g and cationic polymers having intrinsic viscosity 6 to 15 dl/g.
8. A composition according to any preceding claim in which the synthetic polymer has a gel blocking value of at least 9.
9. An agricultural sustained release composition substantially as herein described with reference to the Examples.

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10. An irrigation process for promoting the growth of plants in which water is contacted with a composition which releases agricultural active ingredient into the water and the water is then applied to the plants or to a substrate in which the plants are grown or to be grown and in which the composition is a composition according to any preceding claim whereby a solution of the active ingredient is slowly released from the composition and carried to the substrate or plants by the irrigation water.

11. A process according to claim 10 in which the composition is in the form of a block which is positioned in or on the substrate and the irrigation water is dripped onto it or flooded over it.

12. Use of a composition according to any one of claims 1 to 9 for providing sustained release of agricultural active ingredient to plants or a substrate in which plants are grown or are to be grown.

DATED this TWENTY-SEVENTH day of APRIL 1999

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