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ΚΥΠΡΙΑΚΟ ΓΡΑΦΕΙΟ ΔΙΠΛΩΜΑΤΩΝ ΕΥΡΕΣΙΤΕΧΝΙΑΣ
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(54) Aqueous herbicidal phenoxy-emulsion

(57) Storage-stable, herbicidally-active compositions in the form of a concentrated aqueous emulsion comprising 30 to 75% by weight of one or more herbicidally-active phenoxyalkanecarboxylic acid esters, 1.01 to 11.1 parts by weight per 100 parts of ester of one or more oil-soluble emulsifiers which are soluble in the esters and have an HLB value of 9 to 16, and one or more ionic or non-ionic water-soluble dispersants; the emulsion having a viscosity of 50 to 3,000 mPa.s, a droplet size of 1 to 5 μm and a pH value of 4 to 10; and a process for the preparation of such emulsion which comprises forming a solution of the active ester and emulsifier and mixing it with an aqueous solution of the dispersant to form a homogeneous mixture.

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SPECIFICATION
Aqueous phenoxy-emulsion

This invention relates to a storage-stable, herbicidally-active composition in the form of a concentrated aqueous emulsion of one or more herbicidally active phenoxyalkanecarboxylic acid esters, and to a process for the preparation thereof.

Phenoxyalkanecarboxylic acids are amongst the most commonly used herbicides. They are employed in the form of their amine salts and mineral salts, and also as esters, for example the isopropyl, butyl, butylglycol or 2-ethylhexyl esters. The advantage of the esters is that they penetrate more rapidly into the plant and accordingly their use is less dependent on the weather. Their solubility in water is low, but they are soluble in oils, such as diesel oil, kerosene, petroleum ether, xylenes, cyclohexanone, isophorone or highly refined hydrocarbons. Usually, the ester is dissolved in one of these solvents, and an emulsifier is added to the solution, giving a concentrated, clear, emulsifiable solution (referred to as an emulsion concentrate or EC), which, for field use, is diluted with water, thereby producing an oil-in-water emulsion.

According to Austrian Patent Specification 307,802 it is also possible to store pesticides by preparing a very viscous formulation of the oil-in-water type, consisting of active ingredient, mineral oil, water and formulation auxiliaries, which, for application, is inverted before use, by means of an additive consisting of solvent and dispersant, giving a mobile water-in-oil dispersion referred to as an "inverse emulsion".

Both types of formulation, namely the conventionally used emulsion concentrate and the very viscous dispersion, however, have serious disadvantages, the causes of which rest with the solvent used. All solvents conventionally used for this purpose are, like most organic solvents, at least detrimental to health, but in particular also flammable. Thus, highly purified, predominantly aliphatic hydrocarbons have a flash point of 40° to 80°C., isophorone has a flash point of 93°C., cyclohexanone has a flash point of 44°C. and the most commonly used xylenes have a flash point of only about 25°C.

Accordingly, the emulsion concentrates prepared with the aid of these solvents are also flammable, and in the past there have been a number of fires in stores of such concentrates, both on the manufacturer's premises and also on the user's premises.

In contrast to the prior art described above, it has now been found that by using certain auxiliaries it is possible to produce a storage-stable, highly concentrated emulsion of herbicide esters, which is water-based and is therefore non-flammable and free from solvents detrimental to health. The ability to use an aqueous medium to produce storage-stable emulsions of phenoxy-esters is surprising since the esters are somewhat water-soluble, albeit only slightly, and can therefore undergo hydrolysis.

Accordingly, the present invention provides a storage-stable herbicidally-active composition comprising a concentrated aqueous emulsion of one or more herbicidally active phenoxyalkanecarboxylic acid esters in which the emulsion contains 1.01 to 11.1 parts by weight, per 100 parts of ester, of one or more oil-soluble emulsifiers which are soluble in the esters, have an HLB value of 9 to 16, and are selected from fatty acid polyethylene glycol esters, polyethylene glycol ethers of fatty alcohols, polyethylene glycol ethers of glycerides, polyethylene glycol ethers of alkylphenols, polyoxyethylene/polyoxypropylene block copolymers and mixtures of the said polyethylene glycol esters, polyethylene glycol ethers or polyethylene glycol/polypropylene glycol block copolymers with alkylarylsulphonates; and one or more ionic or nonionic water-soluble dispersants selected from phosphated alkylaryl-polyethylene oxides and ethylene oxide condensates on fatty amines, the herbicidally-active ester being present in an amount of 30 to 75% by weight of the concentrated emulsion, the viscosity of the emulsion being 50 to 3,000 mPa.s, the droplet size being 1 to 5 μm and the pH value being 4 to 10.

The concentrated aqueous emulsion of the invention optionally may also contain one or more conventional anti-freeze agents, conventional anti-foam agents, thickeners or a mixture thereof.

The highly concentrated emulsion according to the invention is prepared by mixing the individual reactants or by mixing 2 solutions. Solution I contains the phenoxy-ester or phenoxy-esters and an emulsifier soluble in the esters, whilst solution II contains a dispersant, dissolved in water, and, optionally, anti-freeze agents, thickeners, anti-foam agents or colorants.

Thus, the invention also provides a process for the preparation of a storage-stable, herbicidally-active composition in the form of a concentrated emulsion of herbicidally active phenoxyalkanecarboxylic acid esters containing 30 to 75% of active compound having a viscosity of 50 to 3,000 mPa.s, which comprises preparing a solution I from one or more phenoxyalkanecarboxylic acid esters and at least one emulsifier which is soluble in the ester and has an HLB value of 9 to 16, in which the amount of emulsifier is 1 to 10 parts by weight per 100 parts by weight of the solution I, and mixing solution I with an aqueous solution II, which contains an ionic or nonionic water-soluble dispersant, in the amount of 0.5 to 5 parts by weight of dispersant per 100 parts of solution II, and subsequently homogenizing the mixture at a temperature within the range of 15° to 90°C. until the droplet size is 1 to 5 μm, while maintaining the pH of the mixture at a value of from 4 to 10, and then diluting the homogenizate to the desired final volume.

The emulsifiers which are soluble in the phenoxy-esters, i.e. fat-soluble emulsifiers, are responsible for the dispersion of the active substance in the continuous phase. Examples of such emulsifiers, having an HLB ("hydrophilicalipophilic balance") value of 9 to 16 are fatty acid polyethylene glycol esters, the polyethylene glycol ethers of fatty alcohols, of monoglycerides or diglycerides and of alkyl-phenols, and polyethylene glycol/polypropylene glycol block polymers. The emulsifiers may be used individually, as a mixture with one another or as a mixture with ammonium, calcium, magnesium, potassium, sodium or zinc salts of alkyl (C_8—C_{24})-benzenesulphonic acids. Fatty acid polyethylene glycol esters or polyoxyethylene/polyoxypropylene block polymers used together with alkylarylsulphonates are particularly suitable.

Solution I contains the fat-soluble emulsifiers in a concentration of 1 to 10 per cent by weight, i.e. 1.01 to 11.1 parts by weight of emulsifier per 100 parts by weight of ester, the range of 2 to 4 parts by weight per 100 parts by weight of solution I being particularly preferred. In the composition of the invention, the emulsifier is preferably present in an amount of 0.5 to 5% by weight, more preferably 1 to 2% by weight.

The water-soluble dispersant stabilizes the distribution of the disperse phase, for example through electrostatic charging of the particles or through other forces which cause repulsion, for example steric hindrance. This dispersant may be ionic or nonionic, but is preferably anionic and of high molecular weight. Examples of water-soluble ionic dispersants are phosphated alkylarylpolyethylene oxides, especially phosphorylated nonylphenylpolyethylene oxide containing from 2 to 20 mols of ethylene oxide per mol of nonylphenol, and salts thereof, as well as the ammonium, sodium and potassium salts of phosphorylated polystyrylphenyl-polyethylene oxide. Examples of nonionic dispersants are ethylene oxide condensates with fatty amines. The phosphated alkylaryl-polyethylene oxides are however particularly suitable dispersants.

Solution II contains the water-soluble dispersant in a concentration of 0.5 to 5 parts by weight, preferably 1 to 3 parts by weight, per 100 parts of solution II. The dispersant concentration in the composition of the invention is preferably 0.2 to 4% by weight, more preferably 0.4 to 2% by weight.

Usually, the storage-stable emulsion of the invention is prepared by preparing an aqueous solution II of a dispersant, which solution optionally contains anti-freeze agents, anti-foam agents or thickeners, and stirring the liquid phenoxy-ester or phenoxy-esters, containing the emulsifier dissolved therein, and constituting solution I, into solution II so as to form a homogeneous mixture. This stirring-in is effected by means of apparatus which generates a shearing rate of between 10^2 and 10^4 sec^{-1} in the emulsion. This corresponds, at high viscosity (3,000 mPa.s) to shearing stresses of 3.10^2 at 3.10^4 Pa, for which an apparatus such as a Homorex or Ultra-Turrax is suitable. At low viscosities (50 mPa.s), the stated shearing rates correspond to shearing stresses of 5 to 500 Pa, for which an apparatus such as a vibrator, a low-speed mixer or a centrifugal pump is suitable. Since the viscosity decreases with increasing temperature, it is possible also to use a low-speed apparatus if an elevated temperature, approximately from 50° to 90°C., is employed. The mixing is intended to produce homogeneous droplet dispersion, with droplet sizes of 1 to 5 μm, preferably of 2 to 3 μm, but in addition to the size of the individual droplets a very narrow size distribution and, at the same time, a low viscosity of the emulsion are of importance.

The viscosity of the concentrated emulsion is an important factor in its shelf life. The higher the viscosity, the better the shelf life. On the other hand, too high a viscosity adversely effects the ease of dilution with water and the spontaneous dispersibility on use. Using the concentrated emulsion prepared according to the invention, it is possible to prepare stable, and at the same time still easily dilutable, formulations having a viscosity within the range of 50 to 3,000 mPa.s. The best results in respect of dilutability and spontaneous dispersibility on the one hand, and stability, on the other hand, are achieved at viscosities of from 500 to 1,500 mPa.s.

Another essential factor in preparing a stable emulsion is the adjustment of the pH value. For each combination of active compound/emulsifier/dispersant there is an optimum pH value at which the emulsion is most stable. This physical stability is best in the alkaline range, but pH values of above 10 should be avoided, since in this range, on prolonged storage and at elevated temperature, partial hydrolysis of the ester may commerce. In an acid medium, pH values of less than 3 are to be avoided, since in this range, on prolonged storage, coalescence may occur, i.e. the emulsion increasingly becomes physically unstable. A pH range of 6.5 to 9, especially of 7.5 to 8.5, is preferred.

The composition according to the invention may be prepared with any herbicidally active phenoxyalkanecarboxylic acid esters, used individually or as mixtures with one another, especially the esters with alcohols of chain length C_4—C_8, for example the octyl esters of 2-(4-chloro-2-methylphenoxy)-propionic acid (CMPP-acid), (2-methyl-4-chlorophenoxy)acetic acid (MCPA-acid) or 2,4,5-trichlorophenoxyacetic acid (2,4,5-T-acid).

Since technical-grade phenoxyalkanecarboxylic acid esters, because of their method of preparation, usually have a pH value of about 3 (measured in a 10% strength aqueous dispersion), the desired pH is obtained by use of a conventional caustic alkali, for example sodium hydroxide or potassium hydroxide. The pH may be adjusted to the desired value during or after preparation of the emulsion, but it is also possible to use purified "neutral" esters, which makes subsequent adjustment of the pH value unnecessary.

To ensure adequate low temperature stability, conventional anti-freeze agents, such as ethylene glycol, glycerol, urea, glycol ethers or other alcohols may be added to the emulsions. Furthermore, it is possible to add known inorganic or organic thickeners, for examples xanthum gum, sodium polyacrylate, carboxymethylcellulose, colloidal silica or swelling clay minerals, such as bentonite, in order to adjust the viscosity to a particular value. To reduce foaming, anti-foam agents, such as long-chain alcohols, 2-ethylhexanol or cetyl alcohol, high-polymer glycols and especially silicones may be added.

For field use, the concentrated emulsions prepared according to the invention are diluted with water in exactly the same way as the hitherto customary flammable emulsion concentrates, and may be applied by means of the same spraying apparatus.

The Examples which follow describe the preparation, as well as the chemical and physical stability, of the compositions according to the invention. The technical-grade phenoxyalkanecarboxylic acid esters used conform to the guideline recommended by the World Health Organization. The stability was tested in a storage test, in which a 24-hour temperature cycle between −10°C. and +50°C. was followed. After storage for a period of 4 weeks, the following were measured:

1. Change in pH value
2. Consumption of 0.01 N sodium hydroxide solution, in order to restore the initial pH.
3. Viscosity change (measured on a Brookfield LVT, viscometer, spindle 2, 6 rpm or, in Examples 3, 13, 17, 18, spindle 1, 6 rpm)
4. Change in the turbidity of a 0.01% strength emulsion, correlated with the change in droplet size (measured by means of a Lange turbidimeter in 100 ml cells).
5. Supernatant liquid, in %
6. Coalescence (formation of an oily phase)
7. Re-emulsifiability.

The results of the measurements relating to the Examples have been summarized in a table. The results of the measurement show that in none of the Examples in question was the change in 1., 2., 3., 4. and 5. more than 10% after a storage time of 4 weeks with a 24-hour temperature cycle between −10°C. and +50°C. No coalescence occurred and any non-coalesced sediment which might be present was completely re-emulsifiable.

EXAMPLE 1

Solution I:	700 g.	of 2,4,5-T-ethylhexyl ester, technical grade (conforms to World Health Organization specification)
	20 g.	of polyoxyethylene triglyceride and alkylarylsulphonate, biologically degradable
Solution II:	22.7 g.	of phosphated nonylphenyl-polyethylene oxide (3 to 14 mols of ethylene oxide = EO), pH = 4.5 to 5.
	100 g.	of ethylene glycol
	877.0 g.	of distilled water
	3.0 g.	of nonionic silicone emulsion

200 ml of solution II were taken and solution I was stirred into it by means of a Homorex mixer. After completion of the addition, stirring was continued for 10 minutes at the highest speed, the pH was then adjusted to 7.25 with half-normal NaOH solution, and the mixture was made up to 1,000 g with solution II and briefly stirred again.

EXAMPLE 2

Solution I:	700 g.	of 2,4,5-T-ethylhexyl ester, technical grade (conforms to World Health Organization specification)
	20 g.	of polyoxyethylene triglyceride and alkylarylsulphonate, biologically degradable

Solution II:	22.7 g.	of phosphated nonylphenyl-polyethylene oxide (3 to 14 mols of ethylene oxide = EO), pH = 6.5
	100 g.	of ethylene glycol
	877.0 g.	of distilled water
	3.0 g.	of nonionic silicone emulsion

The method employed was as described in Example 1. The pH value was adjusted to 7.4.

EXAMPLE 3

Solution I:	600 g.	of MCPA-ethylhexyl ester, technical grade
	20 g.	of fatty alcohol polyglycol ether
Solution II:	886.1 g.	of distilled water
	98.5 g.	of ethylene glycol
	15.4 g.	of phosphated alkylaryl-polyethylene oxide

The method employed was as described in Example 1. The pH value was adjusted to 7.15.

EXAMPLE 4

Solution I:	636.4 g.	of 2,4,5-T-ethylhexyl ester, technical grade
	18.2 g.	of fatty acid polyglycol ester
Solution II:	21.0 g.	of phosphated nonylphenyl-polyethylene oxide (3 to 14 mols of EO), pH = 6.5
	91 g.	of ethylene glycol
	800 g.	of ethylene glycol
	1.8 g.	of nonionic silicone emulsion

The method employed was as described in Example 1. The pH value was adjusted to 6.85.

EXAMPLE 5

Solution I:	668.4 g.	of 2,4,5-T-ethylhexyl ester, technical grade
	19.0 g.	of a mixture of nonylphenyl-polyethylene ethylene oxide, polyoxyethylene-polyoxypropylene block polymer (molecular weight 1,800—9,000) and alkylarylsulphonate
Solution II:	22.0 g.	of phosphated nonylphenyl-polyethylene oxide (3 to 14 mols of EO), pH = 4.5—5
	95.0 g.	of ethylene glycol
	840 g.	of distilled water
	3 g.	of nonionic silicone emulsion

The method employed was as described in Example 1. The pH value was adjusted to 6.90.

EXAMPLE 6

Composition of the stock solution as in Example 1, but the NaOH required for neutralization was introduced with solution II; pH = 7.60

EXAMPLE 7

Solution I:	667 g.	of 2,4-DP-ethylhexyl ester, technical grade (conforms to World Health Organization specification)
	19 g.	of fatty acid polyglycol ester
Solution II:	383 g.	of distilled water
	95 g.	of ethylene glycol
	19.0 g.	of phosphated nonylphenyl-polyethylene oxide (3 to 14 mols of EO), pH = 4.5—5
	3.0 g.	of nonionic silicone emulsion

20 ml of solution II were taken and 72 g of solution I were stirred into it by means of a high-speed mixer (Ultra Turrax). In the course thereof, the temperature of the mixture rose to about 60°C. After stirring for 5 minutes, the pH was adjusted to 7.50 with half-normal NaOH solution and the mixture was made up to 100 g with solution II and briefly stirred again.

EXAMPLE 8

Solution I:	667 g.	of 2,4-DP-ethylhexyl ester, technical grade
	19 g.	of fatty acid polyglycol ester
Solution II:	383 g.	of distilled water
	95 g.	of ethylene glycol
	19.0 g.	of ethylene oxide condensate with a fatty amine (9 to 11 mols of EO)
	3.0 g.	of nonionic silicone emulsion

The method employed was as described in Example 7. The pH value was adjusted to 7.55.

EXAMPLE 9

Solution I:	655 g.	of MCPA-ethylhexyl ester, technical grade
	18.8 g.	of fatty acid polyglycol ester
Solution II:	823 g.	of distilled water
	94 g.	of ethylene glycol
	20.0 g.	of phosphated nonylphenyl-polyethylene oxide (3 to 14 mols of EO), pH = 6.5
	3.0 g.	of nonionic silicone emulsion

200 ml of solution II were introduced into a mixer (Starmix) and solution I was stirred in slowly, so that a homogeneous emulsion was formed. The mixture was heated to 70°C. and homogenized by passing it continuously through a centrifugal pump at about 50 ml/min. It was then neutralized with half-normal NaOH solution and made up to 1,000 g with solution II. The emulsion had a pH value of 7.40.

EXAMPLE 10

Solution I:	650.2 g.	of CMPP-butylglycol ester, purified
	18.6 g.	of fatty acid polyglycol ester
Solution II:	19 g	of phosphated nonylphenyl-polyethylene oxide (3 to 14 mols of EO), pH 4.5—5
	93 g.	of ethylene glycol
	817 g.	of distilled water
	3 g.	of nonionic silicone emulsion

The method employed was as described in Example 7. The CMPP-butylglycol ester was purified by thoroughly stirring an ether solution of the ester with $NaHCO_3$ solution, followed by distillation. The emulsion has a pH value of 6.85.

EXAMPLE 11

Solution I:	650.2 g.	of CMPP-butylglycol ester, purified
	18.6 g.	of fatty acid polyglycol ester
Solution II:	19 g.	of an ethylene oxide condensate with a fatty amine (9 to 11 mols of EO)
	93 g.	of ethylene glycol
	817 g.	of distilled water
	3 g.	of nonionic silicone emulsion

The method employed was as described in Example 10. The pH value was adjusted to 8.30.

EXAMPLE 12

Solution I:	700 g.	of CMPP-ethylhexyl ester, purified as in Example 10
	10 g.	of polyoxyethylene triglyceride and alkylarylsulphonate
Solution II:	881.7 g.	of distilled water
	100 g.	of ethylene glycol
	20 g.	of an ethylene oxide condensate with a fatty amine (9 to 11 mols of EO)
	3.0 g.	of nonionic silicone emulsion

The emulsion was prepared analogously to Example 10; the pH assumed a value of 9.50.

EXAMPLE 13

Solution I:	250 g.	of 2,4-D-i-propyl ester, technical grade
	250 g.	of MCPA-ethylhexyl ester, technical grade
	20 g.	of fatty acid polyglycol ester
Solution II:	20 g.	of phosphated nonylphenyl-polyethylene oxide (3 to 14 mols of EO), pH = 6.5
	100 g.	of ethylene glycol
	880 g.	of distilled water
	3 g.	of nonionic silicone emulsion

The method employed was as described in Example 1. The pH value was adjusted to 7.5.

EXAMPLE 14

Solution I:	700 g.	of 2,4,5-T-ethylhexyl ester, technical grade
	20 g.	of polyoxyethylene triglyceride and alkylarylsulphonate, biologically degradable
Solution II:	22.7 g.	of phosphated nonylphenyl-polyethylene oxide (3 to 14 mols of EO), pH = 6.5
	100 g.	of urea
	877.0 g.	of distilled water
	3.0 g.	of nonionic silicone emulsion

The method employed was as described in Example 1. The pH value was adjusted to 7.15.

EXAMPLE 15

Solution I:	700 g	of 2,4-DP-ethylhexyl ester, technical grade
	20 g.	of a mixture of nonylphenyl-polyethylene oxide, polyoxyethylene-polyoxy-propylene block polymer (molecular weight 1,800 to 9,000) and alkylphenyl-sulphonate
Solution II:	881.7 g.	of distilled water
	100 g.	of glycerol
	20.0 g.	of phosphated nonylphenyl-polyethylene oxide (3 to 14 mols of EO), pH = 2
	3.0 g.	of nonionic silicone emulsion.

200 ml of solution II were taken and solution I was stirred into it by means of a Homorex mixer. After completion of the addition, stirring was continued for 10 minutes at the highest speed, the pH was then adjusted to 7.50 with half-normal NaOH solution, and the mixture was made up to 1,000 g with solution II and briefly stirred again.

EXAMPLE 16

Solution I:	700 g.	of 2,4-DP-ethylhexyl ester, technical grade
	20 g.	of a mixture of nonylphenyl-polyethylene oxide, polyoxyethylene-polyoxy-propylene block polymer (molecular weight 1,800 to 9,000) and alkylphenyl-sulphonate
Solution II:	881.7 g.	of distilled water
	100 g.	of glycerol
	20 g.	of phosphated nonylphenyl-polyethylene oxide (3 to 14 mols of EO), pH = 2
	3.0 g.	of nonionic silicone emulsion

200 ml of solution II were taken and solution I was stirred into it by means of a Homorex mixer. After completion of the addition, stirring was continued for 10 minutes at the highest speed, and the mixture was made up to 1,000 g with solution II and briefly stirred again. The pH value was 4.45.

EXAMPLE 17

Solution I:	500 g.	of MCPA-ethylhexyl ester, technical grade
	20 g.	of fatty acid polyglycol ester
Solution II:	889 g.	of distilled water
	100 g.	of ethylene glycol
	25.0 g.	of an ethylene oxide condensate with a fatty amine (9 to 11 mols of EO)
	3.0 g.	of nonionic silicone emulsion

Mixing and homogenization were carried out as described in Example I. The pH value was adjusted to 8.05.

EXAMPLE 18

Solution I:	400 g.	of MCPA-ethylhexyl ester, technical grade
	20 g.	of fatty acid polyglycol ester
Solution II:	889 g.	of distilled water
	100 g.	of ethylene glycol
	25.0 g.	of an ethylene oxide condensate with a fatty amine (9 to 11 mols of EO)
	3.0 g.	of nonionic silicone emulsion

Mixing and homogenization were carried out as described in Example 1. The pH value was adjusted to 8.1.

EXAMPLE 19

Solution I:	500	g.	of MCPA-ethylhexyl ester, technical grade
	20	g.	of fatty acid polyglycol ester
Solution II:	880	g.	of distilled water
	100	g.	of ethylene glycol
	10	g.	of an ethylene oxide condensate with a fatty amine (9 to 11 mols of EO)
	1.5	g.	of nonionic silicone emulsion
	80.0	g.	of a 2% strength solution of xanthan gum

400 ml of solution II were taken and solution I was stirred in as in Example 1, the pH was adjusted to 7.80 with half-normal NaOH and the mixture was made up to the required volume with a 9:1 water:ethylene glycol mixture.

EXAMPLE 20

Solution I:	400	g.	of MCPA-ethylhexyl ester, technical grade
	20	g.	of fatty acid polyglycol ester
Solution II:	880	g.	of distilled water
	100	g.	of ethylene glycol
	10	g.	of an ethylene oxide condensate with a fatty amine (9 to 11 mols of EO)
	1.5	g.	of nonionic silicone emulsion
	130	g.	of a 2% strength solution of xanthan gum

The method employed was as described in Example 18. The pH value was adjusted to 7.9.

EXAMPLE 21

Solution I:	600	g.	of MCPA-ethylhexyl ester, technical grade
	20	g.	of alkylaryl-polyglycol ether
Solution II:	886	g.	of distilled water
	98.4	g.	of ethylene glycol
	15.5	g.	of phosphated alkylaryl-polyethylene oxide

The method employed was as described in Example 1. The pH value was adjusted to 7.45.

EXAMPLE 22

Solution I:	650	g.	of 2,4-D-butyl ester
	20	g.	of fatty acid polyglycol ester
Solution	897.7	g.	of distilled water
	99.7	g.	of ethylene glycol
	13.9	g.	of phosphated alkylaryl-polyethylene oxide

The method employed was as described in Example 1. The pH value was adjusted to 7.13.

TABLE

	Initial values			after 4 weeks' storage at −10/+50°C.				
Example	pH value	Viscosity in mPa.s	Turbidity	pH value	mg of NaOH/g	Viscosity in mPs.s	Turbidity	% of supernatant liquid
1	7.25	2,900	91.5	6.75	0.07	2,880	91.0	0
2	7.40	2,800	90.0	6.95	0.04	2,690	97.5	1
3	7.15	80*	82	6.90	0.1	75*	82.0	0
4	6.85	490	92.0	6.60	0.05	475	95.0	5
5	6.90	1,775	82.5	6.55	0.05	1,850	75.0	2
6	7.60	2,225	85.5	7.20	0.03	2,010	90.0	0
7	7.50	2,825	85.0	7.00	0.07	2,820	84.0	1
8	7.55	240	88.0	7.50	0.01	260	87.0	3
9	7.40	700	84.5	6.70	0.10	680	86.5	1
10	6.85	725	64.0	6.60	0.02	810	59.0	0
11	8.30	116	75.0	7.50	0.06	120	75.0	6
12	9.50	960	73.0	8.55	0.10	1,050	71.0	1
13	7.50	50*	72.0	7.45	0.01	50*	74.0	7
14	7.15	600	79.5	8.00	–	580	76.0	1
15	7.50	3,000	85.5	7.30	0.02	2,980	90.0	0
16	4.45	1,230	83.0	3.50	0.10	1,170	74.7	9
17	8.05	125*	75.0	7.35	0.05	220*	76.0	3
18	8.10	75*	56.0	7.50	0.07	70*	80.5	2
19	7.80	450	82.5	7.80	–	455	81.5	0
20	7.90	550	73.0	7.70	0.01	525	73.0	0
21	7.45	690	86.0	7.00	0.08	675	85.0	7
22	7.13	250	87.0	6.90	0.1	250	86.0	9

* measured with spindle I.

CLAIMS

1. A storage-stable herbicidally-active composition comprising a concentrated aqueous emulsion of one or more herbicidally active phenoxyalkanecarboxylic acid esters in which the emulsion contains 1.01 to 11.1 parts by weight, per 100 parts of ester, of one or more oil-soluble emulsifiers which are soluble in the esters, have an HLB value of 9 to 16, and are selected from fatty acid polyethylene glycol esters, polyethylene glycol ethers of fatty alcohols, polyethylene glycol ethers of glycerides, polyethylene glycol ethers of alkylphenols, polyoxyethylene/polyoxypropylene block copolymers and mixtures of the said polyethylene glycol esters, polyethylene glycol ethers or polyethylene glycol/polypropylene glycol block copolymers with alkylarylsulphonates; and one or more ionic or non-ionic water-soluble dispersants selected from phosphated alkylarylpolyethylene oxides and ethylene oxide condensates on fatty amines, the herbicidally-active ester being present in an amount of 30 to 75% by weight of the concentrated emulsion, the viscosity of the emulsion being 50 to 3,000 mPa.s, the droplet size being 1 to 5 μm and the pH vlaue being 4 to 10.

2. A composition according to claim 1, which additionally contains one or more conventional anti-freeze agents, conventional anti-freeze agents, thickeners or a mixture thereof.

3. A composition according to claim 1 or 2, in which the pH of the emulsion is 6.5 to 9.

4. A composition according to any one of claims 1 to 3, in which the emulsifier is one or more fatty acid polyethylene glycol esters and the dispersant is one or more phosphated alkylarylpholyethylene oxides.

5. A composition according to any one of claims 1 to 3, in which the emulsifier is a mixture of one or more polyethylene glycol/polypropylene glycol block polymers with alkylarylsulphonates and the dispersant is one or more phosphated alkylaryl-polyethylene oxides.

6. A composition according to any one of the preceding claims, in which the herbicidally active phenoxyalkanecarboxylic acid ester is one or more octyl esters of phenoxyalkanecabroxylic acids.

7. A composition according to any one of the preceding claims, in which the emulsifier is present in an amount of 0.5 to 5% by weight.

8. A composition according to claim 7, in which the amount of emulsifier is 1 to 2% by weight.

9. A composition according to any one of the preceding claims, in which the amount of dispersant is 0.2 to 4% by weight.

10. A composition according to claim 9, in which the amount ot dispersant is 0.4 to 2% by weight.

11. A composition according to any one of the preceding claims, in which the viscosity of the emulsion is 500 to 1,500 mPa.s.

12. A composition according to claim 1, in which the droplet size is 2 to 3 μm.

13. A process for the preparation of a storage-stable, herbicidally-active composition in the form of concentrated emulsion of herbicidally active phenoxyalkanecarboxylic acid esters containing 30 to 75% of active compound having a viscosity of 50 to 3,000 mPa.s., which comprises preparing a solution I from one or more phenoxyalkanecarboxylic acid esters and at least one emulsifier which is soluble in the ester and has an HLB value of 9 to 16, in which the amount of emulsifier is 1 to 10 parts by weight per 100 parts by weight of the solution I, and mixing solution I with an aqueous solution II, which contains an ionic or nonionic water-soluble dispersant, in the amount of 0.5 to 5 parts by weight of dispersant per 100 parts of aqueous solution II, and subsequently homogenizing the mixture at a temperature within the range of 15° to 90°C, until the droplet size is 1 to 5 μm, while maintaining the pH of the mixture at a value of from 4 to 10, and then diluting the homogenizate to the desired final volume.

14. A process according to claim 12, in which solution II additionally contains one or more conventional anti-freeze agents, conventional anti-foam agents, thickeners or a mixture thereof.

15. A storage-stable herbicidally-active composition according to claim 1, and substantially as hereinbefore described with reference to the Examples.

16. A process for the preparation of a storage-stable herbicidally-active composition according to claim 13, and substantially as hereinbefore described with reference to the Examples.

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