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(54) Title: A PROCESS OF PREPARING AN AGROCHEMICAL OIL DISPERSION AND PRODUCT THEREOF

(57) Abstract: The present invention discloses a process for the preparation of stable agrochemical oil dispersion comprising: at least one active ingredient dispersed in an oil phase; and a colloid mixture of viscosity 3500-5000 cps, wherein said process comprising steps of: (1) preparing a dispersion by mixing at least one active ingredient in an oil phase; (2) separately, preparing a colloid mixture of viscosity 3500-5000 cps by mixing rheological additive in an oil phase; (3) adding colloid mixture of step (2) in the dispersion of step (1) to obtain agrochemical oil dispersion.

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A PROCESS OF PREPARING AN AGROCHEMICAL OIL DISPERSION AND PRODUCT THEREOF

TECHNICAL FIELD

The present invention relates to a process of preparing an agrochemical oil dispersion.

- 5 The present invention more particularly relates to a process of preparing an agrochemical oil dispersion and a product thereof. The process involves preparation of a colloid mixture of specific viscosity range that provides desired rheological support to the agrochemical oil dispersion. The presence of colloid mixture imparts long term stability to an agrochemical oil dispersion obtained according to the process of the present invention.

10 BACKGROUND OF THE INVENTION

Agrochemical oil dispersions (OD) are stable suspensions of agrochemical active ingredients, such as pesticides and crop protection chemicals, in non-aqueous media, which may contain other dissolved active ingredients and are usually intended for dilution with water before use.

- 15 OD formulations are fast becoming indispensable in the agrochemical industry as a way of formulating hydrolytically unstable actives. They are, however, notoriously difficult to structure effectively due to the high density of the active ingredients. This often leads to an unacceptably high viscosity formulation which still sediments to some degree. Typical non-aqueous media used in formulating OD include aromatic and non-aromatic
- 20 hydrocarbons, halogenated aromatic and non-aromatic hydrocarbons, aromatic and non-aromatic ethers, esters or amides and oils, including, but not limited to, vegetable oils and paraffin oils. Oil dispersions are particularly useful for formulating oil insoluble solid active ingredients.

- A common problem exist with most of the oil dispersion compositions is its stability upon
- 25 storage. The oil dispersion often shows phase separation after it is stored. Thus, storage even at ambient temperature frequently leads to aggregation effects, syneresis, lump formation or pronounced settling of the suspended phase. In the worst cases, the effects are irreversible, i.e. even shearing, for example by stirring, cannot re-homogenize the formulation.

A common method to solve this problem is to add to the dispersions, as anti-settling agent, a rheological additive that increases the viscosity of the system and acts as suspending agent by reducing the settling rate of the particles.

US 8,298,990 B2 discloses an agrochemical oil dispersion stabilized to particle
5 sedimentation by use of a combination of a clay or silica type rheology modifier and a polymer or oligomer capable of hydrogen bonding i.e. polyethylene glycol or polypropylene glycol.

US9012515 discloses oil formulations with rheological additives wherein rheological additive enables significant higher weight ratio of solid particles in the composition. The
10 rheological additives selected from cellulose-based rheological additives, for example hydroxyethyl cellulose, methylcellulose, hydroxypropyl cellulose and carboxymethyl cellulose; polymeric rheological additives formed from at least one of acrylates, alkylacrylates, and anhydrides; and organoclays. The process of preparing such oil formulations suggest mixing of active ingredient, oil medium and rheological additive and
15 further milling it to obtain the final composition.

WO2012080208A1 discloses a method for the preparation of an agrochemical oil dispersion by mixing solid agrochemical active ingredient and oil; and adding an amide rheological additive.

WO2009004281 discloses bentone clays suitable for use with low polarity organic systems
20 that can be activated by certain emulsifiers instead of polar species. It discloses a method of activating an organoclay stabiliser, which organoclay stabiliser is suitable for use with a low polarity liquid, comprising mixing the organoclay stabiliser with an emulsifier selected from the group consisting of alkyl ethoxylates, alkyl ethoxylate phosphate esters, alkyl sulphates, alkyl ammonium salts and castor oil ethoxylates, under high shear
25 conditions in the presence of a low polarity liquid.

EP0789999A2 discloses an oil suspension concentrate comprising at least one active ingredient, at least one surfactant selected from the group of alkyl ethoxylates and alkylaryl ethoxylates and one surfactant selected from the group of alkyl sulfonates and alkyl aryl sulfonates and a hydrophobized aluminium layer silicate from the bentonite
30 series as a rheological additive.

Therefore, attempts have already been made to incorporate rheological additives in various ways to the oil dispersion, such as activating rheological additives in the presence of a chemical activator in order to function as anti-settling agents with good gel strength; or activating it prior to its incorporation in the dispersion. There is still a need exist to
5 improve the physical stability of oil dispersions and oil suspension concentrates of a variety of agrochemical active ingredients using innovative as well as improved processes that solve the problem of phase separation, settling and unacceptable thickening of the oil dispersion formulation.

Objectives of the invention:

10 An object of the present invention is to provide a process of preparing stable agrochemical oil dispersion.

Another object of the present invention is to provide a process of preparing stable agrochemical oil dispersion that involves preparing colloid mixture of controlled viscosity.

Another object of the present invention is to provide stable agrochemical oil dispersion.

15 Another object of the present invention is to provide stable agrochemical oil dispersion with controlled viscosity.

Another object of the present invention is to provide stable agrochemical oil dispersion of triazolinone herbicide with controlled viscosity.

20 Yet another object of the present invention is to provide a method of controlling weeds using stable agrochemical oil dispersion of triazolinone herbicide.

Another object of the present invention is to provide use of the stable agrochemical oil dispersion of triazolinone herbicide.

Summary of the Invention:

25 According to an aspect of the present invention, a process for the preparation of stable agrochemical oil dispersion comprising: atleast one active ingredient dispersed in an oil phase; and a colloid mixture of viscosity 3500-5000 cps, wherein said process comprising steps of:

- (1) preparing a dispersion by mixing atleast one active ingredient in an oil phase;
- (2) separately, preparing a colloid mixture of viscosity 3500-5000 cps by mixing rheological additive in an oil phase;
- (3) adding colloid mixture of step (2) in the dispersion of step (1) to obtain agrochemical oil dispersion.

According to an aspect of the present invention, a process for the preparation of stable agrochemical oil dispersion comprising: atleast one active ingredient dispersed in an oil phase; and a colloid mixture of viscosity 3500-5000 cps, wherein said process comprising steps of:

- (1) preparing a dispersion by mixing atleast one active ingredient in an oil phase wherein said active ingredient is selected from the group comprising of triazinone, triazole, triazolecarboxamide, triazolinone, triazolinone, N-phenyltriazolinone and N-phenyltriazolinone herbicides.;
- (2) separately, preparing a colloid mixture of viscosity 3500-5000 cps by mixing rheological additive in an oil phase;
- (3) adding colloid mixture of step (2) in the dispersion of step (1) to obtain agrochemical oil dispersion.

According to an aspect of the present invention, a process for the preparation of stable agrochemical oil dispersion comprising: atleast one active ingredient dispersed in an oil phase; and a colloid mixture of viscosity 3500-5000 cps, wherein said process comprising steps of:

- (1) preparing a dispersion by mixing atleast one active ingredient in oil phase wherein active ingredient is selected from triazolinone herbicide;
- (2) milling the dispersion of step (1) to achieve desired particle size;

(3) separately, preparing colloid mixture of viscosity 3500-5000 cps by mixing rheological additive in an oil phase;

(4) adding colloid mixture of step (3) to the dispersion of step (1) to obtain agrochemical oil dispersion.

5 According to an aspect of the present invention, the stable agrochemical oil dispersion comprising:

atleast one active ingredient dispersed in an oil phase; and

a colloid mixture of viscosity 3500-5000 cps.

10 According to another aspect of the present invention, the stable agrochemical oil dispersion comprising:

atleast one active ingredient dispersed in an oil phase; and

5-15% colloid mixture of viscosity 3500-5000 cps

15 According to an aspect of the present invention, a method of controlling weeds wherein said method comprising applying onto weeds or to their locus, an agrochemical oil dispersion comprising:

atleast one active ingredient dispersed in oil phase; and

a colloid mixture of viscosity 3500-5000 cps.

According to another aspect of the present invention, an agrochemical oil dispersion is used as herbicide.

20 **Detailed Description of the invention:**

The particulars shown herein are by way of example and for purposes of illustrative discussion of the various embodiments of the present invention only and are presented in the cause of providing what is believed to be the most useful and readily understood

description of the principles and conceptual aspects of the invention. In this regard, no attempt is made to show details of the invention in more detail than is necessary for a fundamental understanding of the invention, the description making apparent to those skilled in the art how the several forms of the invention may be embodied in practice.

- 5 The present invention will now be described by reference to more detailed embodiments. This invention may, however, be embodied in different forms and should not be construed as limited to the embodiments set forth herein. Rather, these embodiments are provided so that this disclosure will be thorough and complete, and will fully convey the scope of the invention to those skilled in the art.
- 10 Unless otherwise defined, all technical and scientific terms used herein have the same meaning as commonly understood by one of ordinary skill in the art to which this invention belongs. The terminology used in the description of the invention herein is for describing particular embodiments only and is not intended to be limiting of the invention. As used in the description of the invention and the appended claims, the
- 15 singular forms "a," "an," and "the" are intended to include the plural forms as well, unless the context clearly indicates otherwise. All publications, patent applications, patents, and other references mentioned herein are expressly incorporated by reference in their entirety.

- Unless otherwise indicated, all numbers expressing quantities of ingredients, reaction
- 20 conditions, and so forth used in the specification and claims are to be understood as being modified in all instances by the term "about." Accordingly, unless indicated to the contrary, the numerical parameters set forth in the following specification and attached claims are approximations that may vary depending upon the desired properties sought to be obtained by the present invention. At the very least, and not as an attempt to limit
- 25 the application of the doctrine of equivalents to the scope of the claims, each numerical parameter should be construed in light of the number of significant digits and ordinary rounding approaches.

- Inventors of the present invention observed that, the incorporation of rheological additives in the oil dispersion shows marked increase in viscosity which subsequently
- 30 comes down upon standing. Such variability in viscosity creates problem in achieving

desired physico-chemical profile of an oil dispersion. Surprisingly, inventors found that a stable agrochemical oil dispersion composition can be obtained by a peculiar process that involves separately preparing a colloid mixture of viscosity 3500-5000 cps and incorporating it in the separately prepared mill base of the active ingredient(s) along with
5 other excipients. The rheological additive is first mixed in non-aqueous solvent separately and processed to obtain colloid mixture of viscosity ranging from 3500-5000. Such colloid mixture is added to the oil phase in which agrochemicals active ingredient(s) and other suitable dispersing agents are dissolved to obtain said agrochemical oil dispersion. Inventors found that the incorporation of a colloid mixture of viscosity 3500-5000 cps
10 prepared by separately mixing rheological additive in non-aqueous solvent result into stable oil dispersion with uniform viscosity as well as stable physico-chemical profile.

Inventors of the present invention observed that stage of addition of colloid mixture while developing an oil dispersion plays a critical role in terms of stability of the invention. When a colloid mixture is added along with active ingredient and other suitable dispersants in
15 the solvent and homogenized to obtain oil dispersion; it has been observed that over the period of time such oil dispersion thickens due to increase in viscosity and it becomes difficult to pour such oil dispersion from the storage vessel rendering it useless.

When active ingredient as well as suitable dispersants are added in the solvent and milled through suitable means to obtain homogenized mill base; and pre-formulated colloid
20 mixture is added to the mill base, results into a stable oil dispersion. Such oil dispersion exhibits long term storage stability with little or no alteration in viscosity.

As used herein, the term colloid mixture refers to a blend capable of providing structuring to the oil dispersion composition.

As used herein, the term rheological additive refers to compounds that provide requisite
25 thickening to the oil dispersion composition.

In the present disclosure, an oil phase or a non-aqueous solvent are used interchangeably and corresponds to the continuous phase of an oil dispersion composition.

According to an embodiment of the present invention, a process for the preparation of the stable agrochemical oil dispersion comprising steps of:

- (1) preparing a dispersion by mixing atleast one active ingredient in an oil phase;
- (2) separately, preparing a colloid mixture of viscosity 3500-5000 cps by mixing
5 rheological additive in an oil phase;
- (3) adding colloid mixture of step (2) in the dispersion of step (1) to obtain agrochemical oil dispersion.

According to an embodiment of the present invention, active ingredient used for preparing agrochemical oil dispersion is selected from herbicides, fungicides and
10 insecticides.

According to an embodiment of the present invention, active ingredient used for preparing agrochemical oil dispersion is selected from herbicides.

According to an embodiment of the present invention, active ingredient used for preparing agrochemical oil dispersion is selected from triazinone, triazole,
15 triazolecarboxamide, triazolone, triazolinone and N-phenyltriazolinone herbicides, their salts and derivatives thereof.

According to an embodiment of the present invention, active ingredient used for preparing agrochemical oil dispersion is a solid-state herbicide.

According to an embodiment of the present invention, active ingredient used for
20 preparing agrochemical oil dispersion is a triazolinone herbicide.

According to an embodiment of the present invention, active ingredient used for preparing agrochemical oil dispersion is a flucarbazone-sodium.

According to an aspect of the present invention, a process for the preparation of stable agrochemical oil dispersion comprising: atleast one active ingredient dispersed in an oil phase; and a colloid mixture of viscosity 3500-5000 cps, wherein said process comprising steps of:

- 5 (1) preparing a dispersion by mixing atleast one active ingredient in an oil phase wherein said active ingredient is selected from the group comprising of triazinone, triazole, triazolecarboxamide, triazolinone, triazolinone, N-phenyltriazolinone and N-phenyltriazolinone herbicides.;
- 10 (2) separately, preparing a colloid mixture of viscosity 3500-5000 cps by mixing rheological additive in an oil phase;
- (3) adding colloid mixture of step (2) in the dispersion of step (1) to obtain agrochemical oil dispersion.

According to an embodiment of the present invention, the process for the preparation of stable agrochemical oil dispersion comprising steps of:

- 15 (1) preparing a dispersion by mixing atleast one active ingredient in oil phase wherein active ingredient is selected from triazolinone herbicide;
- (2) milling the dispersion of step (1) to achieve desired particle size;
- (3) separately, preparing colloid mixture of viscosity 3500-5000 cps by mixing rheological additive in an oil phase; and
- 20 (4) adding colloid mixture of step (3) to the dispersion of step (1) to obtain agrochemical oil dispersion.

According to an embodiment, the process for the preparation of the stable agrochemical oil dispersion comprising steps of:

- (1). preparing a dispersion by mixing atleast one active ingredient in oil phase wherein active ingredient is selected from triazolinone herbicide and optionally safener, dispersing agents and other auxiliary agents;
 - (2). milling the dispersion of step (1) to achieve desired particle size of the suspended particles;
 - (3). separately, mixing rheological additive in a portion of oil phase to obtain colloid mixture;
 - (4). adding colloid mixture of step (3) to dispersion of step (2) under stirring to obtain stable agrochemical oil dispersion.
- 10 According to an embodiment of the present invention, dispersing agrochemical active ingredient in an oil phase in the presence of dispersants to obtain dispersed mixture is carried out in homogenizer or medium/high velocity disperser or similar equipment.
- According to an embodiment of the present invention, oil phase corresponds to the continuous phase of an oil dispersion composition.
- 15 According to an embodiment, safener is first melted in hot water bath prior to its mixing with an oil phase.
- According to an embodiment of the present invention, the process of preparing stable agrochemical oil dispersion involves milling the dispersed mixture to achieve desired particle size D_{50} from about 3 micron to about 12 micron of the suspended particles.
- 20 According to an embodiment of the present invention, the process of preparing stable agrochemical oil dispersion involves milling the dispersed mixture to achieve desired particle size D_{90} from about 6 micron to about 25 micron of the suspended particles.
- According to an embodiment of the present invention, milling process must be carried out in a mill suitable for solids and liquids, with glass or zirconium balls.
- 25 According to an embodiment of the present invention, colloid mixture is obtained by mixing rheological additive in a portion of oil phase under high shear for about 5 minutes to 30 minutes.

According to an embodiment of the present invention, colloid mixture is obtained by mixing rheological additive in a portion of oil phase under high shear for about 10 minutes.

According to an embodiment of the present invention, colloid mixture is obtained by mixing rheological additive in a portion of oil phase under high shear for about 20 minutes.

- 5 According to an embodiment of the present invention, colloid mixture is obtained by mixing rheological additive in a portion of oil phase under high shear for about 25 minutes.

According to an embodiment of the present invention, colloid mixture is kept aside for 5-7 hours before its incorporation to the dispersion of active ingredient.

- 10 According to an embodiment of the present invention, colloid mixture is kept aside for 5.5 hours before its incorporation to the dispersion of active ingredient.

According to an embodiment of the present invention, colloid mixture is kept aside for 6 hours before its incorporation to the dispersion of active ingredient.

According to an embodiment of the present invention, colloid mixture is kept aside for 6.5 hours before its incorporation to the dispersion of active ingredient.

- 15 According to an embodiment of the present invention, colloid mixture is obtained by mixing rheological additive selected from clay, fumed silica, precipitated silica, bentonite or a combination thereof; in a portion of oil phase under high shear.

According to an embodiment of the present invention, colloid mixture is obtained by mixing bentonite in a portion of oil phase under high shear.

- 20 According to an embodiment of the present invention, colloid mixture is obtained by mixing fumed silica in a portion of oil phase under high shear.

- 25 According to an embodiment of the present invention a process for the preparation of an agrochemical oil dispersion comprises dispersing at least one solid agrochemical active ingredient wherein solid agrochemical active ingredient is selected from triazolinone herbicide in oil phase in the presence of dispersants to obtain dispersed mixture.

According to an embodiment of the present invention, the solid agrochemical active ingredient is selected from triazolinone herbicide.

According to an embodiment of the present invention, the triazolinone herbicide is selected from the group comprising of flucarbazone, sulfentrazone, carfentrazone, thiencazabazone, bencarbazone and amicarbazone, their salts and derivatives thereof.

According to an embodiment of the present invention, triazolinone herbicide is
5 flucarbazone.

According to an embodiment of the present invention, the triazolinone herbicide is flucarbazone-sodium.

According to an embodiment, the process for the preparation of the stable agrochemical oil dispersion comprising: flucarbazone sodium and a safener, cloquintocet-mexyl in an
10 oil phase; and a colloid mixture of viscosity 3500-5000 cps, wherein said process comprising steps of:

(1). preparing a dispersion by mixing flucarbazone sodium, cloquintocet-mexyl, dispersing agents and other auxiliary agents;

(2). milling the dispersion of step (1) to achieve desired particle size of the
15 suspended particles;

(3). separately, mixing bentonite in a portion of oil phase to obtain colloid mixture;

(4). adding colloid mixture of step (3) to dispersion of step (2) under stirring to obtain stable agrochemical oil dispersion.

20 According to an embodiment, the process for the preparation of the stable agrochemical oil dispersion comprising: flucarbazone sodium and a safener, cloquintocet-mexyl in an oil phase; and a colloid mixture of viscosity 4000-4500 cps, wherein said process comprising steps of:

(1). preparing a dispersion by mixing flucarbazone sodium, cloquintocet-mexyl,
25 dispersing agents and other auxiliary agents;

(2). milling the dispersion of step (1) to achieve desired particle size of the suspended particles;

(3). separately, mixing bentonite in a portion of oil phase and keeping aside for 4-5 hours to obtain colloid mixture;

5 (4). adding colloid mixture of step (3) to the dispersion of step (2) under stirring to obtain stable agrochemical oil dispersion.

According to an embodiment of the present invention, the stable agrochemical oil dispersion comprising:

atleast one active ingredient dispersed in oil phase; and

10 a colloidal mixture of viscosity 3000-5000 cps.

The stable agrochemical oil dispersion developed according to the invention exhibits controlled viscosity ranging from 100 cps to 2000 cps.

The stable agrochemical oil dispersion developed according to the invention exhibits controlled viscosity ranging from 300 cps to 2000 cps.

15 The stable agrochemical oil dispersion developed according to the invention exhibits controlled viscosity which is lesser than 1500 cps.

The stable agrochemical oil dispersion developed according to the invention exhibits controlled viscosity which is lesser than 1000 cps.

20 The stable agrochemical oil dispersion developed according to the invention exhibits controlled viscosity which is lesser than 700 cps.

The stable agrochemical oil dispersion developed according to the invention exhibits controlled viscosity which is lesser than 500 cps.

The stable agrochemical oil dispersion developed according to the invention exhibits controlled viscosity ranging from 100-800 cps.

25 The stable agrochemical oil dispersion developed according to the invention remains pourable from the storage vessel.

According to an embodiment of the present invention, the stable agrochemical oil dispersion comprises at least one active ingredient selected from herbicide, fungicide or insecticide classes.

According to an embodiment of the present invention, the active ingredient is an herbicide.

According to an embodiment of the present invention, an herbicide is selected from the group comprising of phenoxy acid derivatives, bipyridyl derivatives, ureas and thioureas (phenyl or substituted ureas), organic phosphorus/ phosphonomethyl amino acids or inhibitors of aromatic acid biosynthesis, protoporphyrinogen oxidase inhibitors (PROTOX) DPE or non-DPE, triazines, triazolinones, triazolinones and triazoles, substituted anilines, amides and acetamides, dinitro compounds, triazolopyrimidines, imidazolinones, benzoic acids, carbamate and thiocarbamate compounds, methyl uracil compounds, polycyclic alkanic acids, dinitroaniline and nitriles.

According to an embodiment of the present invention, the active ingredient of triazolinone herbicide class is selected from the group comprising of flucarbazone, flucarbazone sodium and sulfentrazone.

According to an embodiment of the present invention, the active ingredient of triazolone herbicide is amicarbazone.

According to an embodiment of the present invention, the active ingredient comprises from about 1% w/w to about 70% w/w, preferably 5% w/w to about 50% w/w herbicide of the total weight of the stable agrochemical oil dispersion.

According to an embodiment, the stable agrochemical oil dispersion comprises of an oil phase.

According to an embodiment, oil phase forms the continuous phase of the oil dispersion.

According to an embodiment, oil comprising the oil phase is selected from the group comprising of alkyl esters, soybean oil, rapeseed oil, olive oil, castor oil, sunflower seed oil, coconut oil, corn oil, cottonseed oil, linseed oil, palm oil, peanut oil, safflower oil, sesame oil, tung oil kapok oil, papaya oil, camellia oil, rice bran oil, esters of any of the foregoing, alkoxylated oils of any of the foregoing, methyl and ethyl esters of fatty acids,

mineral oils, paraffinic and isoparaffinic oils, esters, aromatic solvents, and combinations of one or more of the foregoing; the stable agrochemical oil dispersion described above where the oil of an oil phase is selected from the group comprising of methylated rapeseed oil, ethoxylated soybean oil, methyl-5-(dimethylamino)-2-methyl-5-oxopentanoate, alkyl dimethylamide, 2-ethylhexyl lactate, methyl caprylate caproate, methyl stearate, canola oil, and combinations of one or more of the foregoing.

According to an embodiment, oil phase comprises of solvents conventionally used to form continuous phase of an oil dispersion.

According to an embodiment, oil comprising the oil phase is selected from the group comprising of solvent selected from the group consisting of octane, decane, dodecane, tetradecane, xylene, toluene, naphthalene, hexane, cyclohexane, benzene or its derivatives, petroleum solvents, paraffinic liquids, white mineral oil, silicone oils, and organic epoxides, and mixtures thereof.

According to an embodiment, oil phase comprises of an ester of aromatic carboxylic acid.

According to an embodiment, oil phase comprises of C₁-C₄ alkyl ester of aromatic carboxylic acid.

According to an embodiment, the stable agrochemical oil dispersion comprises of an oil phase selected from C₁₋₄ alkyl ester of benzoic acid, preferably butyl ester (butyl benzoate).

According to an embodiment, the stable agrochemical oil dispersion comprises from about 10% w/w to about 90% w/w, preferably 20% w/w to about 80% w/w oil phase of the total weight of the stable agrochemical oil dispersion.

According to an embodiment, the stable agrochemical oil dispersion comprises of a colloid mixture.

According to an embodiment, the colloid mixture is comprised of a rheological additive and an oil phase.

According to an embodiment, the rheological additive is selected from the group comprising of polyacrylics, polyacrylamides, hydrophobically modified cellulose

derivatives, modified starches, co-polymers of cellulose derivatives, carboxyvinyl or polyvinyl pyrrolidones, polyethylenes, polyvinyl alcohol and derivatives, clays, attapulgites, magnesium stearate, or aluminium/magnesium hydroxide stearate, silicas such as hydrophobic fumed silica, precipitated silica, and natural gums such as guar gum, gelatin, dextrin, collagen and derivatives or combination thereof.

According to an embodiment, the rheological additive is selected from the group comprising silica and silicone derivatives such as precipitated silica and fumed silica.

According to an embodiment, the rheological additive is selected from the group comprising natural clays and modified clays.

- 10 According to an embodiment, the natural clays and modified clays are selected from the group comprising of organically modified bentonite, hectorite and smectite clays, for example tetra alkyl ammonium bentonite (for example Bentone™ 34), tetra alkyl ammonium hectorite (for example Bentone™ 38), tetra(alkyl/aryl) ammonium bentonite (for example Bentone SD™-1, Bentone™ 52, Bentone™ 120, and Bentone™ 1000), alkylaryl ammonium hectorite (for example Bentone SD™-3).

According to an embodiment, the rheological additive is tetra(alkyl/aryl) ammonium bentonite.

- According to an embodiment, the stable agrochemical oil dispersion comprises from about 0.1 % w/w to about 20% w/w, preferably 0.5 % w/w to about 15% w/w rheological additive of the total weight of the stable agrochemical oil dispersion.

According to an embodiment, the stable agrochemical oil dispersion comprises of a colloid mixture of viscosity less than or equal to 5000 cps.

According to an embodiment, the stable agrochemical oil dispersion comprises of a colloid mixture of viscosity less than 4500 cps.

- 25 According to an embodiment, the stable agrochemical oil dispersion comprises of a colloid mixture of viscosity less than 4000 cps.

According to an embodiment, the stable agrochemical oil dispersion comprises of a colloid mixture of viscosity ranging from 3500-3800 cps.

According to another aspect of the present invention, the agrochemical oil dispersion comprising:

atleast one active ingredient dispersed in oil phase; and

5-15% colloid mixture of viscosity 3500-5000 cps

- 5 According to an embodiment of the present invention, the colloid mixture is obtained by mixing rheological additive selected from clay, fumed silica, precipitated silica, bentonite and combinations thereof; in an oil phase.

According to an embodiment, the colloid mixture is obtained by mixing rheological additive in oil phase in a ratio from 1:5 to 1:15.

- 10 According to an embodiment, the colloid mixture is obtained by mixing rheological additive in oil phase in 1:8 ratio.

According to an embodiment, the colloid mixture is obtained by mixing rheological additive in oil phase in 1:10 ratio.

- 15 According to an embodiment, the colloid mixture is obtained by mixing rheological additive in oil phase in 1:12 ratio.

According to an embodiment, the stable agrochemical oil dispersion comprises from about 5% w/w to about 15% w/w, preferably 6% w/w to about 12% w/w colloid mixture of the total weight of the stable agrochemical oil dispersion.

- 20 According to an embodiment, the stable agrochemical oil dispersion comprises of dispersing agents.

According to an embodiment, the stable agrochemical oil dispersion comprises of anionic and non-ionic dispersing agents.

- 25 According to an embodiment, the non-ionic dispersing agents are selected from the group comprising of fatty alcohol ethoxylates, tristerylphenol ethoxylates, alkylphenoethoxylates, castor oil ethoxylates, fatty acid ethoxylates, alkylpolyglucosides, sorbitan ethoxylates, and ethylene oxide-propylene oxide-ethylene oxide block copolymers.

According to an embodiment, the anionic dispersing agents are selected from the group comprising salts of dodecylbenzenesulfonate, sodium dioctylsulfosuccinate, salts of tristyrilphenol ethoxylate phosphates, salts of fatty acids, salts of alkyl sulfates, salts of alkylether sulfates, salts of alkylether phosphates, and sodium N-methyl-N-oleytaurate.

- 5 According to an embodiment, the stable agrochemical oil dispersion comprises from about 1% w/w to about 25% w/w, preferably 5% w/w to about 20% w/w dispersing agents of the total weight of the stable agrochemical oil dispersion.

According to an embodiment, the stable agrochemical oil dispersion comprises of safeners.

- 10 According to an embodiment, the safeners are selected from the group comprising of benoxacor, cloquintocet, cloquintocet mexyl, cumyluron, cyometrinil, cyprosulfamide, daimuron, dichlormid, dicyclonon, dimepiperate, fenclorazole-ethyl, fenclorim, flurazole, fluxofenim, furilazole, isoxadifen-ethyl, mefenpyr-diethyl, mephenate, methoxyphenone, naphthalic anhydride, oxabetrinil, N-(aminocarbonyl)-2-
15 methylbenzenesulfonamide and N-(aminocarbonyl)-2-fluorobenzenesulfonamide, I-bromo-4[(chloromethyl)sulfonyl]benzene, 2-(dichloromethyl)-2-methyl-1,3-dioxolane (MG 191), 4(dichloroacetyl)-I-oxa- 4-azospiro[4.5]decane (MON 4660).

According to an embodiment, the safener is cloquintocet-mexyl.

- 20 According to an embodiment, the stable agrochemical oil dispersion comprises from about 0.1% w/w to about 20% w/w, preferably 0.5% w/w to about 10% w/w safener of the total weight of the stable agrochemical oil dispersion.

- According to an embodiment of the present invention, the stable agrochemical oil dispersion may include one or more adjuvants selected from wetting agent, fertilizers, surfactants, compatibility agents, stabilizers, defoamers, antimicrobial agents, 25 antioxidants, correctives, absorbent, an antifreeze agent, a base, an acid, a buffer and spray colorants (dyes).

According to an embodiment of the present invention, the stable agrochemical oil dispersion comprises of wetting agent selected the group comprising of soaps; salts of aliphatic monoesters of sulphuric acid including but not limited to, sodium lauryl sulphate;

sulfoalkylamides and salts thereof including but not limited to N-methyl-N-oleoyltaurate Na salt; alkylarylsulfonates including but not limited to alkylbenzenesulfonates; alkyl naphthalenesulfonates and salts thereof and salts of ligninsulfonic acid.

According to an embodiment of the present invention, the stable agrochemical oil dispersion comprises of defoamer selected from the group comprising of silicone-based compounds, alcohols, glycol ethers, mineral spirits, acetylene diols, polysiloxanes, organosiloxanes, siloxane glycols, reaction products of silicon dioxide and organosiloxane polymer, polydimethylsiloxanes or polyalkylene glycols alone or in combination. Defoamers that are suitable includes Silcolapse-O and SAG-1572.

According to an embodiment, the stable agrochemical oil dispersion may further comprise of second active ingredient.

According to an embodiment, the second active ingredient is an herbicide.

According to an embodiment of the present invention, the second active ingredient is selected from the group comprising of triazinone, triazole, triazolecarboxamide, triazolone, triazolinone, N-phenyltriazolinone and N-phenyltriazolinone herbicides.

According to an embodiment of the present invention, the second active ingredient is selected from the group comprising of hexazinone, metamiltron, metribuzin, amicarbazone, flucarbazone, thienicarbazone, carfentrazone, sulfentrazone and their derivatives.

According to an embodiment, second active ingredient in the stable agrochemical oil dispersion is fenoxaprop-P-ethyl.

According to an embodiment, second active ingredient in the stable agrochemical oil dispersion is pyroxsulam.

According to an embodiment, the stable agrochemical oil dispersion comprises from about 1% w/w to about 70% w/w second active ingredient, from 5% w/w to about 15% w/w colloid mixture of the total weight of the stable agrochemical oil dispersion.

According to an embodiment, the stable agrochemical oil dispersion comprises from about 1% w/w to about 70% w/w second active ingredient, from about 10% w/w to about

90% w/w oil phase, from 5% w/w to about 15% w/w colloid mixture of the total weight of the stable agrochemical oil dispersion.

According to an embodiment, the stable agrochemical oil dispersion comprises from about 1% w/w to about 70% w/w flucarbazon sodium, from about 10% w/w to about 90% w/w butyl benzoate phase, from 5% w/w to about 15% w/w colloid mixture of the total weight of the stable agrochemical oil dispersion.

According to an embodiment, the stable agrochemical oil dispersion comprises from about 1% w/w to about 70% w/w active ingredient and from 5% w/w to about 15% w/w colloid mixture with 3500-5000 cps viscosity of the total weight of the stable agrochemical oil dispersion.

According to an embodiment, the stable agrochemical oil dispersion comprises from about 10% w/w to about 70% w/w active ingredient and from 5% w/w to about 15% w/w colloid mixture with 3800-4500 cps viscosity of the total weight of the stable agrochemical oil dispersion.

According to an embodiment, the stable agrochemical oil dispersion comprises from about 10% w/w to about 70% w/w active ingredient and from 5% w/w to about 15% w/w colloid mixture with 4200-5000 cps viscosity of the total weight of the stable agrochemical oil dispersion.

According to an embodiment, the stable agrochemical oil dispersion comprises from about 1% w/w to about 70% w/w active ingredient, from 5% w/w to about 15% w/w colloid mixture of the total weight of the stable agrochemical oil dispersion, wherein said colloid mixture comprises from about 0.1% w/w to about 10% w/w rheological aid and from about 1% w/w to about 50% w/w oil phase.

According to an embodiment, the stable agrochemical oil dispersion comprises from about 1% w/w to about 70% w/w flucarbazon sodium, from 5% w/w to about 15% w/w colloid mixture of the total weight of the stable agrochemical oil dispersion, wherein said colloid mixture comprises from about 0.1% w/w to about 10% w/w bentonite and from about 1% w/w to about 50% w/w butyl benzoate.

According to an embodiment, the stable agrochemical oil dispersion comprises from about 1% w/w to about 70% w/w flucarbazone sodium, from about 1% w/w to about 70% w/w fenoxaprop-P-ethyl, from 5% w/w to about 15% w/w colloid mixture of the total weight of the stable agrochemical oil dispersion, wherein said colloid mixture comprises
5 from about 0.1% w/w to about 10% w/w bentonite and from about 1% w/w to about 50% w/w butyl benzoate.

According to an embodiment, the stable agrochemical oil dispersion comprises from about 1% w/w to about 70% w/w flucarbazone sodium, from about 1% w/w to about 70% w/w pyroxsulam, from 5% w/w to about 15% w/w colloid mixture of the total weight of
10 the stable agrochemical oil dispersion, wherein said colloid mixture comprises from about 0.1% w/w to about 10% w/w bentonite and from about 1% w/w to about 50% w/w butyl benzoate.

According to an embodiment, the stable agrochemical oil dispersion comprises from about 10% w/w to about 70% w/w active ingredient, from 5% w/w to about 15% w/w
15 colloid mixture of the total weight of the stable agrochemical oil dispersion, wherein said colloid mixture comprises from about 0.1% w/w to about 10% w/w rheological aid and from about 1% w/w to about 30% w/w oil phase.

According to an embodiment of the present invention, colloid mixture is obtained by mixing rheological additive in a portion of oil phase under high shear for about 5 min to
20 30 min.

According to an embodiment of the present invention, colloid mixture is kept aside for 5-8 hours before its incorporation to the dispersion of active ingredient.

According to an embodiment of the present invention, colloid mixture is kept aside for 5-7 hours before its incorporation to the dispersion of active ingredient.

25 According to an embodiment of the present invention, colloid mixture is obtained by mixing rheological additive selected from clay, fumed silica, precipitated silica, bentonite or a combination thereof; in a portion of oil phase under high shear.

According to an embodiment of the present invention, colloid mixture is obtained by mixing bentonite in a portion of oil phase under high shear.

According to an embodiment of the present invention, colloid mixture is obtained by mixing fumed silica in a portion of oil phase under high shear.

According to an embodiment of the present invention, a method of controlling weeds wherein said method comprising applying onto weeds or to their locus, an agrochemical
5 oil dispersion comprising:

atleast one active ingredient dispersed in oil phase; and

a colloid mixture of viscosity 3500-5000 cps.

According to an embodiment of the present invention, a method of controlling weeds wherein said method comprising applying onto weeds or to their locus, an agrochemical
10 oil dispersion comprising:

atleast one active ingredient and a safener dispersed in an oil phase; and

a colloid mixture of viscosity 3500-5000 cps.

According to an embodiment of the present invention, a method of controlling weeds wherein said method comprising applying onto weeds or to their locus, an agrochemical
15 oil dispersion comprising: triazolinone herbicide; and a colloid mixture of viscosity 3500-5000 cps.

According to an embodiment of the present invention, a method of controlling weeds wherein said method comprising applying onto weeds or to their locus, an agrochemical
oil dispersion comprising: flucarbazone-sodium; and a colloid mixture of viscosity 3500-
20 5000 cps.

According to an embodiment of the present invention, a method of controlling weeds wherein said method comprising applying onto weeds or to their locus, an agrochemical
oil dispersion comprising: triazolinone herbicide and safener dispersed in an oil phase;
and a colloid mixture of viscosity 3500-5000 cps.

25 According to an embodiment of the present invention, a method of controlling weeds wherein said method comprising applying onto weeds or to their locus, an agrochemical

oil dispersion comprising: flucarbazone sodium, cloquintocet-mexyl dispersed in an oil phase; and a colloid mixture comprising of viscosity 3500-5000 cps.

According to an embodiment of the present invention, a method of controlling weeds wherein said method comprising applying onto weeds or to their locus, an agrochemical
5 oil dispersion comprising: flucarbazone sodium, cloquintocet-mexyl dispersed in an oil phase; a second herbicide pyroxsulam; and a colloid mixture comprising of viscosity 3500-5000 cps.

According to an embodiment of the present invention, a method of controlling weeds wherein said method comprising applying onto weeds or to their locus, an agrochemical
10 oil dispersion comprising: flucarbazone sodium, cloquintocet-mexyl dispersed in an oil phase; a second herbicide fenoxaprop-p-methyl; and a colloid mixture comprising of viscosity 3500-5000 cps.

According to an embodiment of the present invention, the agrochemical oil dispersion is diluted with water and then applied to plant foliage and/or soil by methods commonly
15 employed in the art, such as conventional high-volume hydraulic sprays, low-volume sprays, air-blast, and aerial sprays. The diluted composition may be applied to the plant foliage or to the soil or area adjacent to the plant. The dilution and rate of application will depend upon the type of equipment employed, the method and frequency of application desired, the recommended herbicide application rate, as well as the weeds to be
20 controlled.

According to an embodiment of the present invention, an agrochemical oil dispersion is used as herbicide.

According to an embodiment of the present invention, the agrochemical oil dispersion can be mixed with fertilizers or fertilizing materials (e.g. at the dilution step) before its
25 application to weeds/crop plants.

According to an embodiment of the present invention, the agrochemical oil dispersion can be utilized as the sole pesticidal agent or it can be employed in conjunction with other pesticidal agents such as, for example, microbicides, fungicides, other herbicides,

insecticides, and acaricides, particularly if these have not already been included in the agrochemical oil dispersion of the invention.

According to an embodiment of the present invention, the agrochemical oil dispersion can be used against various weeds including, but not limited to, the following genera and

5 species: *Abutilon theophrasti*, *Abutilum theophrasti*, *Adonis aestivalis*, *Adonis* spp., *Aethusa cynapium*, *Agrostemma githago*, *Alopecurus myosuroides*, *Amaranthus blitoides*, *Amaranthus retroflexus*, *Amaranthus* spp., *Ambrosia*, *Anagallis arvensis*, *Anthemis* spp., *Anthemis arvensis*, *Aphanes arvensis*, *Arenaria* spp., *Artemisia* spp., *Atriplex patula*, *Avena sterilis*, *Bilderdykia convolvulus*, *Boraginaceae* spp., *Brassica*, *Bromus*, *Bugloss*,
10 *Buglossoides arvensi*, *Calystegia sepium*, *Capsella bursa-pastoris*, *Centaurea cyanus*, *Cerastium arvense*, *Chamomilla recutita*, *Chenopodium album*, *Chrysanthemum*, *Chrysanthemum segetum*, *Cirsium arvense*, *Convolvulus* spp., *Convolvulus arvensis*, *Coronopus* spp., *Cytisus scoparius*, *Datura stramonium*, *Descurainia*, *Digitaria sanguinalis*, *Diploaxis eruroides*, *Diploaxis* spp., *Echinochloa*, *Echinochloa crus-galli*, *Epilobium angustifolium*, *Eriogonum*, *Eruca vesicaria*, *Fagopyrum*, *Fallopia*, *Fallopia convolvulus*,
15 *Fumaria officinalis*, *Galeopsis tetrahit*, *Gallium aparine*, *Gallium* spp., *Geranium molle*, *Helianthus annuus*, *Hypocoum procumbens*, *Kochia* spp., *Lactuca*, *Lamium amplexicaule*, *Lamium purpureum*, *Linum*, *Lithospermum arvense*, *Lolium rigidum*, *Malva sylvestris*, *Matricaria*, *Myosotis palustris*, *Myosotis arvensis*, *Panicum miliaceum*, *Papaver rhoeas*,
20 *Parietaria* spp., *Persicaria lapathifolia*, *Persicaria maculosa*, *Polygonum* spp., *Polygonum aviculare*, *Portulaca oleracea*, *Portulaca olerace*, *Raphanus raphanistrum*, *Reseda* spp., *Ridolfia saegetum*, *Rumex* spp., *Rumex acetosa*, *Rumex crispus*, *Rumex obtusifolius*, *Salsola kali*, *Senecio* spp., *Senecio vulgaris*, *Setaria verticillata*, *Setaria viridis*, *Silene* spp., *Sinapsis arvensis*, *Sisymbrium orientalis*, *Solanum americanum*, *Solanum nigrum*, *Sonchus*
25 *spp.*, *Sonchus oleraceus*, *Sorghum halepense*, *Spergula arvensis*, *Stellaria media*, *Thlaspi arvense*, *Ulex* spp., *Urdica dioica*, *Urtica urens*, *Vaccaria segetum*, *Veronica* spp., *Veronica hederifolia*, *Veronica persica*, *Viola* spp., *Viola arvensis*, *Viola bicolor*, *Volunteer rape*, *Volunteer sugar beet*, *Xanthium* spp., *Xanthium strumarium*, and *Xanthium spinosum*.

According to an embodiment of the present invention, the agrochemical oil dispersion
30 can be used to control weeds (such as any of those mentioned above) in crops plants such as cereals crops (e.g. corn, oats (winter and spring), wheat (winter wheat and summer

wheat), triticale, durum wheat, rye, barley (winter barley and spring barley), rice, etc.), maize, soya, potatoes, cotton, rapeseed, linseed, beet, sugar cane and also fruit plants.

According to an embodiment of the present invention, the agrochemical oil dispersion can also be used to control weeds in permanent grasslands and newly sown grass leys.

- 5 It will be understood that the specification and examples are illustrative but not limitative of the present invention and that other embodiments within the spirit and scope of the invention will suggest themselves to those skilled in the art. Other embodiments can be practiced that are also within the scope of the present invention. The following examples illustrate the invention, but by no means intend to limit the scope of the invention.

10 **Example 1: Preparation of colloid mixture**

11.7g butyl benzoate was charged in a beaker and 1.3g bentonite was added slowly to the beaker with high shear mixing in high-speed homogenizer (1500-1800 rpm) for around 10-20 minutes and kept aside for 5-8 hours to obtain colloid mixture of 10% strength having viscosity ranging from 3500 to 5000 cps (measured in spindle 64/60 rpm).

Ingredients	Quantity
Bentonite	1.3
Butyl benzoate	11.70
Total	13.00

15

Stabilizing time study

- The colloid mixture obtained in above step is kept aside for 5-7 hours to observe change in viscosity. The viscosity of colloid mixture was calculated several times after suitable intervals to observe behaviour of rheological additive in the presence of oil phase. It was
- 20 observed that with each passing time interval, viscosity gradually came down. Initial viscosity of the colloid mixture was 9500 which came down to 4370 cps in 7.3 hours and further came down to 3950 in 24 hours. Afterwards, the viscosity remained constant and does not change further. (Table 1)

Table 1

Time	Viscosity in Centipoise (cps)
20 min	9500
80 min	5200
140 min	4900
320 min	3890
440 min	4370
12 hours (min)	4190
Kept for 24 Hours (Undisturbed)	3950

Example 2: Preparation of 20% w/w Flucarbazone sodium oil dispersion (OD).**Step 1: Preparation of mill base**

- 5 4.11 g cloquintocet mexyl was melted separately on a hot water bath (water Batch Temp is 95 deg C) and kept aside. 46.85g butyl benzoate was charged in a mixing vessel and molten cloquintocet mexyl was added under stirring. 1g block copolymeric surfactant, 6g fatty alcohol ethoxylate, 2g calcium alkylbenzene sulfonate and 7g fatty acid ethoxylate were added to the same mixing vessel under continuous stirring. 20.04g flucarbazone sodium was then added and homogenized to obtain homogeneous mixture.
- 10

Ingredients	Quantity
Butyl benzoate	46.85
Cloquintocet mexyl (Molten)	4.11
Non-ionic surfactant	14
Anionic surfactants	2
Flucarbazone sodium	20.04
Total	87

Step 2: Preparation of colloid mixture

11.7g butyl benzoate was charged in a beaker and 1.3g bentonite was added slowly to the beaker with high shear mixing for around 10-20 minutes and kept aside for 8 hours to obtain colloid mixture of 3900 cps (10% gel).

Ingredients	Quantity
Bentonite	1.3
Butyl benzoate	11.70
Total	13.00

5

Step 3: Preparation of agrochemical oil dispersion

Under continuous stirring, colloid mixture was added to the mill base and homogenized for around 45-50 minutes to obtain stable agrochemical oil dispersion.

Ingredients	Quantity (w/w)
Mill base	87
Colloid mixture	13
Total	100

10

Example 3: Preparation of 20% w/w Flucarbazone sodium oil dispersion (OD) (comparative example)

An oil dispersion was prepared according to conventional process of preparation that involves mixing all the ingredients together, followed by milling the mixture to obtain desired particle size to obtain oil dispersion. Therefore, flucarbazone sodium herbicide, cloquintocet-mexyl, butyl benzoate, calcium alkylbenzene sulfonate, fatty acid ethoxylate, isotridecyl alcohol polyglycol ether, organoclay and polymeric dispersant were mixed together followed by milling the mixture upto desired particle size to obtain an oil dispersion.

20

Ingredients	Quantity (% w/w)
Butyl benzoate	59.94
Flucarbazone sodium	20.31
Cloquintocet-mexyl	4.02
Calcium alkylbenzene sulfonate	7
Fatty acid ethoxylate	2
Isotridecyl alcohol polyglycol ether	6
Organoclay	1.15
Polymer dispersant	1
Total	100

Comparative study of agrochemical oil dispersions

The formulation characteristics such as pourability and viscosity of the agrochemical oil dispersions developed according to Example-2 and Example-3 were compared. Pourability was measured by in house method: The 80ml OD composition was filled in 100ml HDPE bottle. First observation was made by tilting the graduated cylinder at 90 degree and pouring the entire content from graduated cylinder onto 100ml glass beaker. Entire amount of the composition came out from the cylinder. Again, The 80ml OD composition was filled in 100ml HDPE bottle and was given 10 inversions. Second observation was made by tilting the graduated cylinder at 90 degree and pouring the entire content from graduated cylinder onto 100ml glass beaker. Entire amount of the composition came out from the cylinder. It was observed that the composition of Example-2 retained good pourability of close to 100% in ambient as well as in 14 days AHS (Accelerated Heat Stability) study, both at 0 inversion and 10 inversions. Therefore, OD composition of Example-2 found to pass pourability test satisfactorily. Viscosity was measured as per CIPAC MT 192. Viscosity of the composition of Example-2 found to be 492 cps which increased within acceptable limit to 602 in 14 days AHS. Composition of Example-2 was kept under observation to check stability of the OD formulation in real time. The sample was kept for 15 months in ambient conditions. The viscosity of the composition of was found to be 296 cPs in real time. The slight change in viscosity observed was within acceptable range. Upon visual inspection, OD found clear

without any thickening or phase separation. OD formulation found to be acceptable after 15 months.

Similarly, composition of Example-3 found to be 100% pourable at ambient condition but it became too thick that viscosity could not be measured when sample was taken out after 14 days AHS. Composition of Example-3 found to have a viscosity of 578 cps in ambient and further in 14 days AHS, sample became too thick that viscosity could not be measured.

(Table 2)

Table 2

	14 days Aging Data of Pourability				
	Example 2			Example 3	
	0 days (Ambient)	14 days (AHS)	15 months (RT)	0 days (Ambient)	14 days (AHS)
0 inversion	100% pourable	91.32	95	100% pourable	Not measurable. (Thick non-pourable liquid.)
10 inversion	100% pourable	93.56	98	100% pourable	Not measurable (Thick non-pourable liquid.)
Viscosity (RVT, 60 RPM, #3 Spindle (cPs))	492	602	296	578	Not measurable (Thick non-pourable liquid.)
Remarks	Viscous free flowing liquid	Viscous free flowing liquid	Viscous free flowing liquid	Viscous free flowing liquid	Thick non-pourable liquid.

10

Example 4-8: Few more compositions of Flucarbazone 20% w/w OD were developed to ascertain effectiveness of colloid mixture in preparing agrochemical oil dispersions.

Compositions of Example-4-8 were prepared to confirm the stability of oil dispersion prepared as per the process disclosed in the present invention. Viscosity of colloid mixture prepared as per the process of the present invention by using various rheological additives such as bentonite, modified bentonite, fumed silica and oil phase made of butyl benzoate, mustard oil or soybean oil. The colloid mixture of the compositions of Example 4-8 found to be within desired range of 3500-5000 cPs. Also, viscosity of final OD compositions also found to be stable with viscosity in an acceptable range from 100-650 cPs. (Table 3)

20

Table 3

	Quantity				
Ingredients	Example-4	Example-5	Example-6	Example-7	Example-8
	% w/w				
Flucarbazon sodium	20.04	20.04	20.04	20.04	20.04
Cloquintocet mexyl	4.11	4.11	4.11	4.11	4.11
Butyl benzoate	46.85	44.85	19	46.85	46.85
Calcium alkylbenzene sulfonate	7	7	4.5	7	7
Fatty acid ethoxylate, POE 30	2	2	1.5	2	2
Fatty alcohol ethoxylate	6	6	4	6	6
Polymeric dispersant	1	1	1	1	1
Methyl ester derived from canola oil	0.00	0.00	32.05	0.00	0.00
	87.00	85.00	86.20	87.00	87.00
	Gel Part				
Bentonite clay	1.3	-	-	1.3	1.3
Modified bentonite clay	-	1.5	-	-	-
Hydrophilic fumed silica	-	-	0.80	-	-
Butyl benzoate	11.7	13.5	13.00	-	-
Mustard oil	-	-	-	11.7	-
Soybean oil	-	-	-	-	11.7
Viscosity@Gel (3500-5000 cps)	3850	4950	4880	3680	3712
Viscosity@Oil Dispersion (RVT, 60 RPM, #3 Spindle (cPs))	492	156	290	420	194

Pourability study:

The compositions of Example 4-8 prepared as per the process disclosed in the present invention were evaluated for pourability characteristic. It was observed that the compositions of Example 4-8 retained good pourability in ambient conditions, both in 0 inversion and 10 inversions. Viscosity of the composition of Examples 4-8 were also found to be within acceptable range. All the compositions found to be satisfactorily pourable in ambient conditions with more than 85% OD composition coming out from the storage container in 0 inversion and more than 95% OD composition coming out from the storage container in 10 inversions. Compositions of Example 4-6 were kept in AHS and rise in

viscosity of the final oil dispersion formulations was calculated. All the three formulations exhibited optimum viscosity with slight deviation within $\pm 6\%$. Compositions of Example-7 and 8 were evaluated in ambient conditions just after (zero day) and found to exhibit viscosity within acceptable limits. (Table 4)

5

Table 4

14 days Aging Data of Pourability

	Example 4		Example 5		Example 6		Example 7	Example 8
	Ambient	7D AHS	Ambient	7 D AHS	Ambient	7 D AHS	Ambient (0 days)	Ambient (0 days)
0 inversion	96.89	89.21	99.45	99.23	98.58	88.27	98.3	99.31
10 inversion	99.89	98.89	99.87	99.67	99	98.5	99.89	98.91
RVT, 60 RPM, #3 Spindle (cPs)	492	520	156	146	290	305	420	194
Remarks	Viscous free flowing liquid		Viscous free flowing liquid		Viscous free flowing liquid		Viscous free flowing liquid	Viscous free flowing liquid

Examples 9-11: The compositions of Example 9 and Example 10 were assessed for pourability and viscosity of the agrochemical oil dispersions developed according to the process described in the present invention. It was observed that the compositions of Example 9 & 10 retained good pourability in ambient conditions, both in 0 inversion and 10 inversions. Viscosity of the composition of Examples 9 & 10 were also found to be within acceptable range. Overall, it was observed that compositions of Example 9 & 10 remained quite pourable with more than 90% contents coming out of the vessel in 0 inversion as well as in 10 inversions. Viscosity of the final oil dispersion formulations also remained controlled without any significant rise and hence resulting into stable OD formulations. (Table 5 and Table 6)

Example 9: Amicarbazone 30% Oil Dispersion (OD)

Ingredients	Quantity
Amicarbazone	30.00
Butyl benzoate	40.07
Anionic Surfactant	8.00
Non-ionic surfactants	8.93
Gel Part	
Bentonite	1.3
Butyl benzoate	11.7

Amicarbazone, one part of butyl benzoate, anionic surfactant, non-ionic surfactant and gel part comprising bentonite and remaining part of butyl benzoate were mixed in above quantity and processed as per the Example-1 to obtain oil dispersion composition.

5 **Example 10: Sulfentrazone 20% w/w Oil Dispersion**

Ingredients	Quantity (% w/w)
Sulfentrazone	20.00
Butyl benzoate	50.5
Anionic surfactant	7.00
Non-ionic surfactants	9.5
Gel Part	
Bentonite	1.30
Butyl benzoate	11.70

Sulfentrazone, one part of butyl benzoate, anionic surfactant, non-ionic surfactant and gel part comprising bentonite and remaining part of butyl benzoate were mixed in above quantity and processed as per the Example-1 to obtain oil dispersion composition.

Table 5

14 days Aging Data of Pourability				
	Example 9		Example 10	
Pourability	AMBIENT	14D AHS	AMBIENT	14D AHS
Parameters				
0 inversion	100	100	100	98.8
10 inversion	99.95	99.9	100	99.85
RVT, 60 RPM, #3	183	153	406	184

Spindle (cPs)				
Remarks	Free flowing liquid		Viscous free flowing liquid	

Example 11: Flucarbazone 20% w/w + Pyroxsulam 6% w/w OD

Ingredients	Quantity (% w/w)
Flucarbazone-sodium	20.82
Cloquintocet-mexyl	4.87
Pyroxsulam	6.06
Butyl benzoate	39.05
Anionic surfactants	3
Non-ionic surfactants	12.5
Ethoxylated phosphate ester	1
Gel part	
Bentonite	1
Butyl benzoate	11.7
Total	100

Flucarbazone-sodium, cloquintocet-mexyl, pyroxsulam, one part of butyl benzoate, anionic surfactant, non-ionic surfactant, ethoxylated phosphate ester and gel part comprising bentonite and remaining part of butyl benzoate were mixed in above quantity and processed as per the Example-1 to obtain oil dispersion composition.

Table 6

1 Week viscosity data			
Parameters	Initial	1 Week	
Test	RT	0 °C	-10 °C
Appearance	Free Flowing Liquid	Not frozen, Free flowing	Not frozen, Free flowing
Appearance on thaw	N/A	Free Flowing Liquid	Free Flowing Liquid
Viscosity, RVT, 60 RPM, #3 Spindle (cPs)	220	222	222

The composition of Example-11 was evaluated for viscosity and visual observations. It appeared as free flowing liquid when tested at 0 day in ambient condition. The composition was kept in storage for cold temperature stability for 1 week at 0°C and -10°C. It remained free flowing at both 0°C and -10°C. Similarly, its viscosity remained

almost constant at 0 day (ambient) as well as after 1 week when kept for cold temperature stability.

Inventors of the present invention thus successfully prepared an agrochemical oil dispersion by a process described in the present invention that involves preparation of
5 colloid mixture of viscosity range 3500-5000 cps. The incorporation of colloid mixture comprising rheological additive and oil phase and its stage of addition during the process of preparing an agrochemical oil dispersion played critical role in obtaining stable composition. The compositions developed according to the process described in the present invention based by incorporating colloid mixture of 3500-5000 cps viscosity found
10 to remain stable with desired pourability and viscosity.

CLAIMS

1. A process for the preparation of stable agrochemical oil dispersion comprising: atleast one active ingredient dispersed in oil phase; and a colloid mixture of viscosity 3500-5000 cps, wherein said process comprising steps of:
- 5 (1) preparing a dispersion by mixing atleast one active ingredient in oil phase;
- (2) separately, preparing a colloid mixture of viscosity 3500-5000 cps by mixing rheological additive in an oil phase;
- (3) adding colloid mixture of step (2) in the dispersion of step (1) to obtain agrochemical oil dispersion.
- 10
2. The process as claimed in claim 1 wherein said active ingredient is selected from the group comprising of triazinone, triazole, triazolecarboxamide, triazolone, triazolinone and N-phenyltriazolinone herbicides.
3. The process as claimed in claim 1 wherein said active ingredient is selected from the group comprising of hexazinone, met amitron, metribuzin, amicarbazone, flucarbazone, thienicarbazone, carfentrazone, sulfentrazone and their derivatives.
- 15
4. The process as claimed in claim 1 wherein said rheological additive is selected from the group comprising of clay, fumed silica, precipitated silica, bentonite and combinations thereof.
- 20
5. The process as claimed in claim 1 wherein said oil dispersion comprises of 5% w/w to 15% w/w colloid mixture of the total weigh of the oil dispersion.
6. The process as claimed in claim 1 wherein said process comprising steps of:
- (1) preparing a dispersion by mixing atleast one active ingredient in oil phase wherein active ingredient is selected from triazolinone herbicide;
- 25 (2) milling the dispersion of step (1) to achieve desired particle size;

(3) separately, preparing colloid mixture of viscosity 3500-5000 cps by mixing rheological additive in an oil phase;

(4) adding colloid mixture of step (3) to the dispersion of step (1) to obtain agrochemical oil dispersion.

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7. The process as claimed in claim 4 wherein said triazolinone herbicide is flucarbazone-sodium.

8. The process as claimed in claim 1 wherein particle size of said active ingredient in dispersion is from about 3 micron to about 12 micron.

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9. The process as claimed in claim 1 wherein said colloid mixture is obtained by mixing said rheological additive and said oil phase and keeping it aside for 5-7 hours.

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10. A stable agrochemical oil dispersion comprising:

atleast one active ingredient dispersed in oil phase; and

a colloid mixture of viscosity 3500-5000 cps.

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11. The stable agrochemical oil dispersion as claimed in claim 10 wherein said active ingredient is flucarbazone-sodium.

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12. The stable agrochemical oil dispersion as claimed in claim 10 wherein said colloid mixture comprises of a rheological additive selected from the group comprising of clay, fumed silica, precipitated silica, bentonite or a combination thereof; and an oil phase.

13. The stable agrochemical oil dispersion as claimed in claim 10 wherein ratio of rheological additive and oil phase is from 1: 5 to 1: 15.

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14. The stable agrochemical oil dispersion as claimed in claim 10 wherein viscosity of said oil dispersion is from 100-800 cps.

15. The stable agrochemical oil dispersion as claimed in claim 10 wherein said oil dispersion comprises from about 1% w/w to about 70% w/w active ingredient and from 5% w/w to about 15% w/w colloid mixture with 3500-5000 cps viscosity of the total weight of the stable agrochemical oil dispersion.
- 5 16. The stable agrochemical oil dispersion as claimed in claim 10 wherein said oil dispersion comprises from about 1% w/w to about 70% w/w active ingredient, from 5% w/w to about 15% w/w colloid mixture of the total weight of the stable agrochemical oil dispersion, wherein said colloid mixture comprises from about 0.1% w/w to about 10% w/w rheological aid and from about 1% w/w to about 50% w/w oil phase.
- 10 17. The stable agrochemical oil dispersion as claimed in claim 10 wherein said oil dispersion comprises from about 1% w/w to about 70% w/w flucarbazone sodium, from 5% w/w to about 15% w/w colloid mixture of the total weight of the stable agrochemical oil dispersion, wherein said colloid mixture comprises from about 0.1% w/w to about 10% w/w bentonite and from about 1% w/w to about 50% w/w butyl benzoate.
- 15 18. The stable agrochemical oil dispersion as claimed in claim 10 wherein said oil dispersion further comprises of a second herbicide selected from aryloxyphenoxypropionic, anilide, acetamide, chloroacetamide, cyclohexanedione, diphenylether, triazolopyrimidine, triketone, triazinylsulfonylurea, phenylpyrazoline, pyridinecarboxylic acid group of herbicides.
- 20 19. The stable agrochemical oil dispersion as claimed in claim 10 wherein second active ingredient is selected from the group comprising of clodinafop, haloxyfop, cyhalofop, napropamide, propanil, acetochlor, butachlor, metolachlor, s-metolachlor, clethodim, sethoxydim, fomesafen, oxyfluorfen, florasulam, penoxsum, pyroxulam mesotrione, tembotrione, tribenuron, thifensulfuron, metulfuron, pinoxaden, halauxifen, their
25 derivatives and combinations thereof.
20. The stable agrochemical oil dispersion as claimed in claim 10 wherein said oil dispersion comprises from about 1% w/w to about 70% w/w flucarbazone sodium, from about 1% w/w to about 70% w/w fenoxaprop-P-ethyl, from 5% w/w to about 15% w/w colloid mixture of the total weight of the stable agrochemical oil dispersion, wherein said

colloid mixture comprises from about 0.1% w/w to about 10% w/w bentonite and from about 1% w/w to about 50% w/w butyl benzoate.

21. The stable agrochemical oil dispersion as claimed in claim 10 wherein said oil dispersion comprises from about 1% w/w to about 70% w/w flucarbazone sodium, from
5 about 1% w/w to about 70% w/w pyroxsulam, from 5% w/w to about 15% w/w colloid mixture of the total weight of the stable agrochemical oil dispersion, wherein said colloid mixture comprises from about 0.1% w/w to about 10% w/w bentonite and from about 1% w/w to about 50% w/w butyl benzoate.

22. A method of controlling weeds wherein said method comprises applying onto weeds
10 or to their locus, an agrochemical oil dispersion comprising: at least one active ingredient dispersed in oil phase; and a colloid mixture of viscosity 3500-5000 cps.

23. The method as claimed in claim 22 wherein said method comprises applying onto weeds or to their locus, an agrochemical oil dispersion comprising: flucarbazone-sodium;
15 and a colloid mixture of viscosity 3500-5000 cps.

INTERNATIONAL SEARCH REPORT

International application No.

PCT/IB2022/059211

A. CLASSIFICATION OF SUBJECT MATTER

A01N 25/02 (2006.01) A01N 43/42 (2006.01) A01N 43/653 (2006.01) A01N 47/38 (2006.01) A01N 43/90 (2006.01)

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EPOQUE 6.30.05 (PATENW): keywords (agrochemical, oil dispersion, triazinone, triazole, triazolecarboxamide, triazolinone, N-phenyl-triazolinone, hexazinone, metamilon, metribuzin, amicarbazone, flucarbazone, thienicarbazone, carfentrazone, sulfentrazone, clay, bentonite, silica, butyl benzoate, mustard oil, soybean oil, cloquintocet-mexyl, fenoxaprop-P-ethyl, pyroxsulam, weed and alternative names); IPC/CPC symbols (A01N25/02, A01N43/653, A01N43/42, A01N47/38) **STNext (AGRICOLA, Caplus, CASFORMULTNS, CROPB, CROPR, CROPU, FSTA, Registry):** Applicant/inventor names (UPL Limited, Rajan Ramakant Shirsat, Shiv Kumar Sharma, Pradeep Shamrao Sapkale); keywords (as above) **ONLINE DATABASES (Espacenet, Google Scholar/Patents/Search):** Applicant/inventor names (as above); keywords (as above); IPC/CPC symbols (as above) **INTERNAL DATABASES:** Applicant/inventor names (as above).

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
	Documents are listed in the continuation of Box C	

☒ Further documents are listed in the continuation of Box C☒ See patent family annex

* Special categories of cited documents:	
"A" document defining the general state of the art which is not considered to be of particular relevance	"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"D" document cited by the applicant in the international application	"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"E" earlier application or patent but published on or after the international filing date	"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	"&" document member of the same patent family
"O" document referring to an oral disclosure, use, exhibition or other means	
"P" document published prior to the international filing date but later than the priority date claimed	

Date of the actual completion of the international search
28 November 2022Date of mailing of the international search report
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INTERNATIONAL SEARCH REPORT		International application No.
C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		PCT/IB2022/059211
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 2017032644 A1 (BAYER CROPSOURCE AKTIENGELLESCHAFT) 02 March 2017 Abstract; Method 1; Example 1; pg. 10 ln 10-12; pg. 12 paras. 1-3; pg. 26 para. 1; claim 13	1-23
X A	WO 2018222726 A1 (ARYSTA LIFESOURCE INC.) 06 December 2018 Abstract; Example 2; Table 2; paras. [008], [0011], [0019] and [0024]; claims 3 and 43	10-23 1-9
X A	CN 105519559 A (TANG YUNHE ET AL.) 27 April 2016, Machine translation by Google Patents retrieved from Internet 17 November 2022 Abstract; Embodiments 1, 7 and 8	10-23 1-9
A	US 20200077645 A1 (ARYSTA LIFESOURCE INC.) 12 March 2020 Tables 1 and 2	1-9
E	WO 2022224275 A1 (RAJDHANI PETROCHEMICALS PRIVATE LIMITED) 27 October 2022 Abstract; Examples 1-9; Experiment 4	1-8, 10-12, 14-19, 22, 23

INTERNATIONAL SEARCH REPORT		International application No.	
Information on patent family members		PCT/IB2022/059211	
This Annex lists known patent family members relating to the patent documents cited in the above-mentioned international search report. The Australian Patent Office is in no way liable for these particulars which are merely given for the purpose of information.			
Patent Document/s Cited in Search Report		Patent Family Member/s	
Publication Number	Publication Date	Publication Number	Publication Date
WO 2017032644 A1	02 March 2017	None	
WO 2018222726 A1	06 December 2018	WO 2018222726 A1	06 Dec 2018
		AR 111838 A1	21 Aug 2019
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		CN 110944509 A	31 Mar 2020
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US 2022125054 A1	28 Apr 2022		
CN 105519559 A	27 April 2016	CN 105519559 A	27 Apr 2016
US 20200077645 A1	12 March 2020	None	
WO 2022224275 A1	27 October 2022	WO 2022224275 A1	27 Oct 2022
End of Annex			
Due to data integration issues this family listing may not include 10 digit Australian applications filed since May 2001.			