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(54) Title: EMULSION ADJUVANT FOR HERBICIDES

(57) Abstract: An oil-in-water emulsion liquid composition suitable for use as an agricultural adjuvant comprising an oil, a satu-
rated or substantially saturated aqueous solution of an ammonium salt and one or more of high molecular weight polymeric sur-
factants.



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Emulsion Adjuvant for Herbicides

Field of the invention

The invention relates to adjuvants for use in agriculture. More particularly, the invention relates to emulsion compositions having an oil and an ammonium salt solution as an
5 adjuvant for enhancing the effect of agrochemicals (also known as agrichemicals).

Background of the invention

In this specification where a document, act or item of knowledge is referred to or discussed, this reference or discussion is not an admission that the document, act or item of knowledge or any combination thereof was at the priority date publicly available, known to
10 the public, part of the common general knowledge or known to be relevant to an attempt to solve any problem with which this specification is concerned.

Adjuvants are used in agriculture to assist the performance of agrochemicals such as herbicides, insecticides, fungicides, growth promoters etc. Many different classes of chemicals are used as adjuvants to assist the agrochemical in different ways. For example:

- 15 • oils for foliar penetration or to reduce evaporation,
- surfactants (often also called emulsifiers) for surface wetting, coverage and/or penetration,
- buffering agents for protecting sensitive agrochemicals, and
- ammonium salts to provide hard water protection, promote compatibility and
20 stimulate plant uptake of agrochemicals.

When preparing a mixture for spraying, an applicator may need more than one class of adjuvant and thus create a complex tank mix containing multiple adjuvant products and crop protection product(s). This can lead to problems such as incompatibility between the various products added to the spray tank or other unforeseen problems such as crop
25 damage or toxicity to non-target organisms.

Recently, products which combined two or more classes of adjuvant have been made available. One such product combining lipophilic solvent (oil) and lipophobic plant nutrient (ammonium salt) is described by US patent no 7,314,848 which uses cationic surfactants to form a homogeneous liquid composition suitable as an agrochemical adjuvant, particularly for use with the herbicide glyphosate.

While cationic surfactants are powerful surfactants for a number of reasons, they are not the most desirable surfactants for use in agrochemical formulations. The use of cationic surfactants may increase the risk of tank mix incompatibility, foaming, driftable fines, crop phytotoxicity and toxicity to non-target organisms, such as beneficial insects, birds and aquatic life.

A liquid emulsion adjuvant product needs to be convenient to use (pourable and dispersible into water) and have a shelf-life (no substantial change) of at least a few months, and preferable greater than one year, to be useful. That is, such liquid emulsion products need to be stable, pourable and dispersible into water.

Generally, the amount of surfactant required to produce such a stable, pourable and dispersible liquid emulsion adjuvant product is related to the proportion of oil in the formulation. As a result, a higher proportion of surfactant is required to produce a stable emulsion containing a higher proportion of oil than emulsion containing a lower amount of oil. Further, an even higher proportion of surfactant is required to produce a stable emulsion if the aqueous phase contains electrolytes than an equivalent electrolyte-free formulation. For example, prior art formulations containing oil and saturated or nearly saturated solutions of ammonium salts using surfactants involve:

- a proportion of surfactant (both oil compatible and water soluble electrolyte tolerant surfactants) to oil which is generally greater than 1:1, and is always greater than 2:3 (US2008127661; see examples);
- a proportion of surfactant to oil of more than 1:1 (US20030125211 and US20070032382, see examples); and

- a proportion of surfactant (combination of nonionic and anionic surfactants) to oil of at least 1:2 (WO99/55645, see examples containing more concentrated ammonium salt solution (more than 5%)).

There is thus a need for improved adjuvant formulations which combine oils and ammonium salts in a form suitable for use as an agricultural adjuvant, but which also minimize the amount of powerful or harsh surfactants used, and thus reduce the detrimental effects of such surfactants.

Summary of the invention

It has surprisingly been found that certain polymeric surfactants can be used to prepare formulations suitable for use as agricultural adjuvants which contain oil and concentrated aqueous solutions of ammonium salts. Such adjuvant formulations contain a lower amount of surfactant than prior art formulations containing an equivalent amount of oil and ammonium salts. This has the benefit of reducing the detrimental effects due to the use of high levels of surfactants.

According to a first aspect of the invention, there is provided a liquid emulsion composition suitable for use as an agricultural adjuvant comprising:

- (a) an amount in the range of from 5 to 95% w/w (preferably 10 to 80% w/w) of an oil selected from the group consisting of mineral oils, vegetable oils, esters of fatty acids, aliphatic alcohols and mixtures thereof;
- (b) an amount in the range of from 5 to 95% w/w (preferably 20 to 80% w/w) of a saturated or substantially saturated aqueous solution of an ammonium salt selected from the group consisting of ammonium sulphate, ammonium phosphates, ammonium nitrate and mixtures thereof; and
- (c) an amount in the range of from 0.1 to 5% w/w of one or more high molecular weight polymeric surfactants having a molecular weight in the range of from 2000 to 12000;

wherein the proportion of total surfactants to oil in the composition is less than 1:3.

The liquid emulsion composition according to the invention is stable, pourable and dispersible into water.

The oil must be liquid and crop safe. Preferred mineral oils have a carbon number in the range of 12 to 30, are low in aromatic compounds and have a viscosity (at 40°C) in the range of approximately 5 to 50cSt. Suitable vegetable oils include all liquid vegetable oils. Preferably, the vegetable oils are refined so as to remove gums and excess acidity to ensure that the liquid emulsion composition is stable, liquid and sprayable. More preferably, the amount of oil used in the composition is in the range of from 10 to 60% w/w, even more preferably 15 to 50% w/w, and most preferably 20 to 50% w/w.

10 Suitable esters of fatty acids include alkyl esters of fatty acids wherein:

- the alkyl moiety may be methyl, ethyl, propyl or butyl; and
- the fatty acids are from any natural or synthetic source, are saturated and/or unsaturated and the majority of the fatty acids have a carbon number of from 12 to 22.

15 Typical embodiments are methyl esters of canola oil, where the major component is methyl oleate.

Where an aliphatic alcohol is the oil of choice, it may be selected from primary or secondary alcohols having a carbon number of from 8 to 22 and may be saturated or unsaturated. A typical embodiment is a C-12 Guerbet alcohol.

20 The concentration of the ammonium salt in the saturated or substantially saturated aqueous solution will vary depending on the ammonium salt selected. For example, where the ammonium salt is either sulphate, nitrate or phosphate, then a solution containing approximately 20 - 50% w/w of the ammonium salt in water may be used. Where ammonium nitrate is the preferred ammonium salt, then it is common to prepare an aqueous solution further containing urea. For example, UAN32 contains 45 parts ammonium nitrate, 35 parts urea and 20 parts water.

The examples of ammonium salt solutions above may not be the maximum concentrations technically possible, but reflect the commercial high concentration solutions typically used

in the fertilizer industry which must allow for cold temperatures and/or loss of water through evaporation without the risk of crystallization.

More preferably, the amount of saturated or substantially saturated aqueous solution of ammonium salts used in the composition is in the range of from 40 to 80% w/w, and most preferably 50 to 75% w/w.

The high molecular weight polymeric surfactants are present in the formulation to maintain the oil and the saturated or substantially saturated aqueous solution of ammonium salts together in a stable liquid formula. Preferably, the amount of high molecular weight polymeric surfactants used in the composition is 0.1 to 5% w/w, more preferably 0.1 to 3% w/w, and most preferably 0.2 to 2% w/w.

Suitable high molecular weight polymeric surfactants having a molecular weight in the range 2000 to 12000 are typically linear. Examples of suitable high molecular weight polymeric surfactants having a molecular weight in the range 2000 to 12000 include:

- polyalkylene oxide block copolymers of a simple primary alcohol (eg. ethylene oxide-propylene oxide block copolymers of butanol) such as Atlas™ G-5000, Termul™ 5429 or Tergitol™ XJ, XD or XH;
- polyisobutene succinic anhydride – polyethylene glycol such as Atlox™ 4914;
- polyhydroxystearate polyethers such as Atlox™ 4912, or
- polyalkylene oxide triblock copolymers of the type EO/PO/EO (where EO is ethylene oxide and PO is propylene oxide) such as Synperonic™ PE/F 127 and Poloxomer™ 338.

Preferably, the composition may include an additive such as acidifiers or dispersing agents which may be desirable to help the application of the composition. Acidifiers may be added where a lower pH is required in the tank, for example when using alkaline sensitive agrochemicals. Suitable acidifiers include sulphamic acid, propionic acid or citric acid but many others may be equally suitable.

A dispersing agent may be added where there is a need to more easily disperse the emulsion (e.g. in a mixer tank). The dispersing agent may be selected from the group consisting of nonionic surfactants (e.g. alcohol ethoxylates, fatty acid ethoxylates, glycerol monooleate, polysorbate, and alkyl polyglucosides), anionic surfactants (e.g. calcium dodecylbenzene sulphonate, sulphated or phosphated fatty alcohols/fatty alcohol polyethers, alkyl polyether carboxylates and salts) and mixtures thereof. Typically, the amount of dispersing agent used is up to 5% w/w of the total composition.

When determining the proportion of total surfactants to oil in the composition, the determination must include all surfactants in the composition, for example any dispersing agents which are surfactants and the polymeric surfactant (c). Preferably, the proportion of total surfactants to oil in the composition is less than 1:4, and more preferably less than 1:6.

Compositions according to the invention are typically comprised of at least 94% w/w of oil and the saturated or substantially saturated aqueous solution of ammonium salts with the total amount of surfactants (including the high molecular weight polymeric surfactants and any dispersing agents) typically not being more than 6% w/w. The invention thus enables the production of liquid emulsion compositions containing oil and concentrated ammonium salt solutions which contain very low levels of surfactants enabling their use in situations where the use of harsh surfactants is restricted.

According to a second aspect of the invention, there is provided a method for reducing one or more of the detrimental effects caused by high levels of surfactants used in adjuvants for agrochemical formulations, the method comprising combining one or more agrochemicals with a liquid emulsion composition suitable for use as an agricultural adjuvant comprising:

- (a) an amount in the range of from 5 to 95% w/w (preferably 20 to 80% w/w) of an oil selected from the group consisting of mineral oils, vegetable oils, esters of fatty acids, aliphatic alcohols and mixtures thereof;
- (b) an amount in the range of from 5 to 95% w/w (preferably 20 to 80% w/w) of a saturated or substantially saturated aqueous solution of an ammonium salt selected from the group consisting of ammonium sulphate, ammonium phosphates, ammonium nitrate and mixtures thereof; and

[illegible]

The ratio of surfactant to oil in each of these compositions is set out below.

Composition	Ratio of surfactant to oil
A	6:40
B	6:40
C	6:40
D	5.5:20 (highest)
E	2:40
F	2:40
G	2:40
H	0.5:91.8 (lowest)
I	2.4:10
J	6:40
K	3:30

Example 2

- 5 In this example, the adjuvant effect of Compositions C and D from Example 1 was compared with a range of commercially available adjuvants when applied with glyphosate in 2 water hardnesses to control oats in a glasshouse.

Treatment List

Product Name	Manufacturer	Active Constituents
ROUNDUP CT Broadacre herbicide	Monsanto	450g/l glyphosate as the isopropylamine salt
Deluge 1000 wetting agent	Victorian Chemicals	950g/l non-ionic fatty acid ethoxylates
Hot-Up Spray Adjuvant	Victorian Chemicals	190g/l mineral Oil 340g/l nonionic / cationic surfactants 140g/l ammonium sulphate
Assert Spray Adjuvant	Victorian Chemicals	420g/l ammonium sulphate
Infiltrator Spray Adjuvant	Victorian Chemicals	700g/l vegetable oil ester

Experimental conditions:

Weed Species:	Oats 1.5-2 leaf
Application technique:	Track sprayer
Water:	64l/ha
Nozzles:	11001
Speed:	6 kph
Glyphosate application rates:	50 & 100g a.i./ha
Water hardness:	All treatments were applied at 2 water hardness: Low (50ppm CaCO ₃) and High (300ppm CaCO ₃). High hardness is known to adversely affect the performance of the herbicide Glyphosate.
Replicates	6
Assessment:	Fresh weight 14 days after treatment. Results are reported as Fresh Weight % of untreated control

*Results*Table 2: Glasshouse trial results

Glyphosate Rate (ga.i./ha)	Water Hardness (as ppm CaCO ₃)	No Adjuvant	Deluge 1000	Assert	Hot-Up			Assert + Infiltrator	Composition C			Composition D		
					0.1	0.25	0.5		0.1	0.25	0.5	0.1	0.25	0.5
none			0.1	2.0	0.1	0.25	0.5	2.0 + 0.5	0.1	0.25	0.5	0.1	0.25	0.5
50	50	73	56	44	58	47	34	40	49	43	29	76	55	46
50	300	106	-	45	80	55	42	24	76	47	26	81	42	22
100	50	52	38	38	49	24	24	31	42	30	22	43	30	21
100	300	65	-	47	50	33	29	34	69	45	30	49	49	29

Discussion

- 5 The trial confirms the following well established facts regarding the use of glyphosate as a herbicide:
- Higher rate of Glyphosate results in lower fresh weight of weed.
 - Addition of wetting agent results in lower fresh weight of weed. (Deluge 1000 was not applied in hard water).
- 10
- Application in hard water increases fresh weight of weed.
 - Addition of ammonium sulphate results in lower fresh weight of weed and tends to limit the effect of hard water.
 - The combination of oil, cationic surfactants and ammonium sulphate in the product Hot-Up can be more effective than either wetting agent or ammonium sulphate alone. Higher application rate results in lower fresh weight of weed.
- 15

- Using an oil adjuvant (Infiltrator) and ammonium sulphate adjuvant (Assert) results in lower fresh weight than ammonium sulphate alone.

The trial also shows that the formulations according to the invention, Compositions C and D, when applied under the conditions of this experiment are effective adjuvants for the agrochemical glyphosate. Both of Compositions C and D provide herbicidal efficacy enhancement to glyphosate which is similar to the commercial product Hot-Up. Compositions C and D when applied at 0.5% result in equivalent or better adjuvant effects than those obtained with the combination of 0.5% Infiltrator (oil) plus 2.0% Assert (ammonium sulphate).

10 In summary, Compositions C and D provide results which are comparable to or better than the prior art adjuvant options, and yet contain very low levels of surfactants which are likely to enable their use in situations where the use of harsh surfactants is restricted.

The word 'comprising' and forms of the word 'comprising' as used in this description and in the claims does not limit the invention claimed to exclude any variants or additions.

15 Those skilled in the art will appreciate that the invention described herein is susceptible to variations and modifications other than those specifically described. It is to be understood that the invention includes all such variations and modifications. The invention also includes all of the steps, features, compositions and compounds referred to or indicated in this specification, individually or collectively, and any and all combinations of any two or
20 more of said steps or features.

Claims

1. An oil-in-water emulsion liquid composition suitable for use as an agricultural adjuvant comprising:
 - (a) an amount in the range of from 5 to 95% w/w of an oil selected from the group consisting of mineral oils, vegetable oils, esters of fatty acids, aliphatic alcohols and mixtures thereof;
 - (b) an amount in the range of from 5 to 95% w/w of a saturated or substantially saturated aqueous solution of an ammonium salt selected from the group consisting of ammonium sulphate, ammonium phosphates, ammonium nitrate and mixtures thereof; and
 - (c) an amount in the range of from 0.1 to 5% w/w of one or more high molecular weight polymeric surfactants having a molecular weight in the range 2000 to 12000,wherein the proportion of total surfactants to oil in the composition is less than 1:3.
2. The composition according to claim 1 wherein the proportion of total surfactants to oil in the composition is less than 1:4.
3. The composition according to either of claims 1 or 2 wherein proportion of total surfactants to oil in the composition is less than 1:6.
4. The composition according to any one of the preceding claims wherein the amount of oil used in the composition is in the range of from 20 to 50% w/w.
5. The composition according to any one of the preceding claims wherein the amount of saturated or substantially saturated aqueous solution of ammonium salts used in the composition is in the range of from 50 to 75% w/w.
6. The composition according to any one of the preceding claims wherein the amount of high molecular weight polymeric surfactant used in the composition is in the range from 0.1 to 3% w/w.

7. The composition according to any one of the preceding claims wherein the amount of high molecular weight polymeric surfactant used in the composition is in the range from 0.2 to 2% w/w.
8. The composition according to any one of claims 1 to 3 wherein the composition comprises:
 - (a) an amount in the range of from 10 to 60% w/w of an oil selected from the group consisting of mineral oils, vegetable oils, esters of fatty acids, aliphatic alcohols and mixtures thereof;
 - (b) an amount in the range of from 40 to 80% w/w of a saturated or substantially saturated aqueous solution of an ammonium salt selected from the group consisting of ammonium sulphate, ammonium phosphates, ammonium nitrate and mixtures thereof; and
 - (c) an amount in the range of from 0.1 to 3% w/w of one or more high molecular weight polymeric surfactants having a molecular weight in the range 2000 to 12000,

wherein the proportion of total surfactants to oil in the composition is less than 1:3.

9. The composition according to any one of claims 1 to 3 wherein the composition comprises:
 - (a) an amount in the range of from 20 to 50% w/w of an oil selected from the group consisting of mineral oils, vegetable oils, esters of fatty acids, aliphatic alcohols and mixtures thereof;
 - (b) an amount in the range of from 50 to 75% w/w of a saturated or substantially saturated aqueous solution of an ammonium salt selected from the group consisting of ammonium sulphate, ammonium phosphates, ammonium nitrate and mixtures thereof; and

- (c) an amount in the range of from 0.2 to 2% w/w of one or more high molecular weight polymeric surfactants having a molecular weight in the range 2000 to 12000,

wherein the proportion of total surfactants to oil in the composition is less than 1:3.

10. The composition according to any one of the preceding claims wherein the one or more high molecular weight polymeric surfactants are selected from the group consisting of polyalkylene oxide block copolymers of a simple primary alcohol, polyisobutene succinic anhydride – polyethylene glycol, polyhydroxystearate polyethers, polyalkylene oxide triblock copolymers of the type EO/PO/EO and mixtures thereof.
11. The composition according to any one of the preceding claims wherein the one or more high molecular weight polymeric surfactants are polyalkylene oxide block copolymers of a simple primary alcohol.
12. The composition according to any one of the preceding claims further comprising an additive to help the application of the composition selected from the group consisting of dispersing agents, acidifiers and mixtures thereof.
13. The composition according to claim 11 wherein the additive comprises one or more dispersing agents selected from the group consisting of nonionic surfactants, anionic surfactants and mixtures thereof.
14. The composition according to either of claims 11 or 13 wherein the dispersing agent is present in the composition in an amount up to 5% w/w of the total composition.
15. A method for reducing one or more of the detrimental effects caused by high levels of surfactants used in adjuvants for agrochemical formulations, the method comprising combining one or more agrochemicals with a liquid emulsion composition suitable for use as an agricultural adjuvant comprising:
 - (a) an amount in the range of from 5 to 95% w/w of an oil selected from the group consisting of mineral oils, vegetable oils, esters of fatty acids, aliphatic alcohols and mixtures thereof;

- (b) an amount in the range of from 5 to 95% w/w of a saturated or substantially saturated aqueous solution of an ammonium salt selected from the group consisting of ammonium sulphate, ammonium phosphates, ammonium nitrate and mixtures thereof; and
- (c) an amount in the range of from 0.1 to 5% w/w of one or more high molecular weight polymeric surfactants having a molecular weight in the range of from 2000 to 12000;

wherein the proportion of total surfactants to oil in the composition is less than 1:3.

16. The method according to claim 15 wherein the composition further comprises an additive to help the application of the composition selected from the group consisting of dispersing agents, acidifiers and mixtures thereof.

INTERNATIONAL SEARCH REPORT

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A. CLASSIFICATION OF SUBJECT MATTER

Int. Cl.

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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)

WPI, Epodoc, CA, Agricola and keywords: ammonium sulphate, ammonium phosphate, ammonium nitrate, oil, alcohol, surfactant and related terms

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 2008/127661 A1 (UNIQEMA AMERICAS, INC.) 23 October 2008 See abstract, paragraphs [00015]-[00022], [00025], [00026], [00027], [00029], [00030]-[00058], [00062], [00067], examples	1-16
X	WO 2007/030885 A1 (NUFARM AUSTRALIA LIMITED) 22 March 2007 See abstract, page 4 line 15 to page 7 line 17, page 9 line 17 to page 13 line 2, page 13 line 28 to page 14 line 5, page 15 line 23 to page 17 line 17	1-16
X	CA 2694028 A1 (BAYER CROPSCIENCE AG, DE) 18 December 2008 See abstract, page 4 line 8 to page 9 line 29, page 15 line 12 to page 17 line 25, examples	1-16



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
"E" earlier application or patent but published on or after the international filing date	"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
"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)	"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
"O" document referring to an oral disclosure, use, exhibition or other means	"&" document member of the same patent family
"P" document published prior to the international filing date but later than the priority date claimed	

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C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 2007/050090 A1 (VALENT U.S.A CORPORATION) 3 May 2007 See page 14 Formulation M, example 2	1-10, 12-16
X	US 2003/0125211 A1 (WOZNICA, Z. J. et al.) 3 July 2003 See abstract, paragraph [0010]-[0035], examples, Table 1	1-10, 12-16

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No.

PCT/AU2011/000189

This Annex lists the known "A" publication level 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 Cited in Search Report		Patent Family Member					
WO	2008127661	AR	065986	AU	2008239638	CA	2682150
		CN	101677527	CO	6220874	EP	2144499
		KR	20100016447	MX	2009011001	US	2010160168
WO	2007030885	AU	2006291952	AU	2006291953	AU	2006292036
		CA	2622568	EP	1933621	US	2008254983
		WO	2007030886	WO	2007030887		
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		CO	6241135	EP	2002719	EP	2157852
		KR	20100046110	MX	2009013536	US	2010179228
		WO	2008151725				
WO	2007050090	NONE					
US	2003125211	US	6642178	US	2003104947	US	6689720
Due to data integration issues this family listing may not include 10 digit Australian applications filed since May 2001.							
END OF ANNEX							