

SYNERGISTIC HERBICIDE EMULSIONBACKGROUND OF THE INVENTION

Butachlor is the common name for 2-chloro-2',-6'-diethyl-N-(butoxymethyl)acetanilide herbicide which is an effective herbicide for the pre-emergence control of most annual grasses and certain broadleaf weeds in both seeded and transplanted rice grown in certain Asian, South American, European and African countries.

Butachlor herbicide is commercially available as an emulsifiable concentrate formulation sold under the tradename Machete EC by the Monsanto Company. (Machete is a Registered Trade Mark). An emulsifiable concentrate is a formulation containing the active ingredient, i.e., butachlor, dissolved in an organic solvent with added emulsifiers.

As will be appreciated by one skilled in the art, emulsifiable concentrate formulations have the disadvantage that they contain an organic solvent, i.e., a petroleum based solvent. As the price of crude petroleum escalates, so does the cost of organic solvents. Shortages of crude petroleum may occur resulting in a shortage of petroleum by-products making organic solvents difficult to obtain. Further, organic solvents are often irritants to the skin or eyes when handled, they may be hazardous from the aspect that the organic solvent may be quite flammable and they are often detrimental to the environment.

Accordingly, a herbicidal formulation which eliminates the necessity of an organic solvent and uses only water as the carrier for the active ingredient is highly desirable in that the use of expensive and/or scarce organic solvents is eliminated and the possibility of skin and/or eye irritation and hazards due to flammability are reduced or eliminated.

It is accordingly the object of this invention to provide an emulsion formulation of butachlor,

2-chloro-2',6'-diethyl-N-(butoxymethyl) acetanilide which consists of the active ingredient butachlor, dispersed in a water carrier with added emulsifiers. Unexpectedly, the emulsion formulations described herein provide superior weed control when compared to the known prior art Machete EC formulation and an increase in crop yield on a kilogram per hectare basis.

While the exact forces or interactions which lead to the superior synergistic emulsion formulations of this invention are not known, one possible explanation is that the emulsion formulations of this invention are more compatible with water when mixed in the field and, accordingly, the emulsion formulation mixes with water more quickly and has superior dispersion properties than the traditional emulsifiable concentrate formulation.

DETAILED DESCRIPTION OF THE INVENTION

The present invention relates to an aqueous emulsion formulation of butachlor herbicide, i.e., the active herbicidal ingredient, butachlor is dispersed in a water carrier containing a certain emulsifier to form a two-phase system consisting of an organic phase containing the butachlor herbicide and an aqueous or water phase.

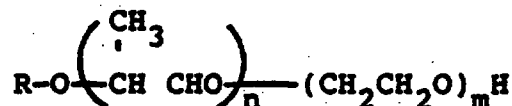
The synergistic herbicidal emulsion formulation of the present invention consists essentially of the following components:

<u>Ingredient</u>	<u>% by Weight</u>
A. Butachlor	30.0 - 70.0
B. Emulsifier	0.75 - 5.0
C. Formulation	
Adjuvants	0 - 15.0
D. H ₂ O	<u>Balance</u>
Total	100.0

As used herein, butachlor is understood as being the common name for 2-chloro-2',6'-diethyl-N-(butoxymethyl)acetanilide herbicide and is the active

ingredient in the emulsion formulations described herein. The emulsion formulations described herein will contain from 30.0 to 70.0 percent by weight of butachlor, for instance from 45.0 to 60.0 percent by weight, preferably from 40.0 to 55.0 percent by weight and most preferably 47.5 to 50.0 percent by weight, for example 48.0 percent by weight.

The term "emulsifier" as used herein, refers to the class of nonionic emulsifiers generically known as polyalkylene glycol ethers that are block co-polymers of propylene oxide and ethylene oxide. While the precise nature of such emulsifiers is generally proprietary to the manufacturers of said nonionic emulsifiers, in general, they will have the formula



where R is equal to alkyl, aryl or alkylaryl; n is approximately equal to 1250 and m is approximately equal to 2200 for an approximate molecular weight of 3500. As used herein the term "alkyl" refers to a straight or branched chain alkyl containing 1-5 carbon atoms. The term "aryl" refers to an aromatic group containing 6-12 carbon atoms, e.g., phenyl or naphthyl. The term "alkylaryl" refers to an alkyl substituted phenyl group wherein the alkyl group may be straight or branched chain and which may contain 1-20 carbon atoms.

Similar block co-polymers of propylene oxide and ethylene oxide are known in the art which have a molecular weight of approximately 3500, which are formed by polymerizing the required quantity of propylene oxide and then adding ethylene oxide.

The above-described emulsifiers are readily available and are exemplified by, for example, "Tergitol" XH and XD, the tradenames of polyalkylene glycol ether nonionic emulsifiers available from Union Carbide Corporation, Chemicals and Plastics, 270 Park Avenue, New York, N. Y. 10017 U.S.A.; "Flo Mo XHA", the tradename for the block co-polymer propylene oxide and

ethylene oxide manufactured by Sellers Chemical Corp.,
1320 Sams Ave., P.O. Box 23523, Harahan, Louisiana
70183, U.S.A.; and "Pluronic P-65", the tradename of a
propylene oxide/ethylene oxide block co-polymer,
5 manufactured by BASF Wyandotte Corp., Textile Colors &
Chem. Dept., 100 Cherry Hill Road, Parsippany, N. J.
07054, U.S.A. (Pluronic is a Registered Trade Mark).

There are many emulsifiers of the type
described above known in the art and such emulsifiers
10 may be found in McCUTCHEON's Detergents and Emulsifiers,
North American Ed., 1980 Annual, McCutcheon Division, MC
Publishing Co., 175 Rock Road, Glenrock, New Jersey
07542, U.S.A.

The concentration of the emulsifier present in
15 the emulsion formulations described herein will range
from 0.75 to 5.0 percent by weight, for example from 1.5
to 5.0 percent by weight, preferably from 1.5 to 3.5 percent
by weight and most preferably at 2.0 percent by weight.

The term "formulation adjuvants" as used
20 herein, refers to minor quantities of one or more
components such as dyes, e.g., methyl violet, density
balancing agents, e.g., sodium chloride, calcium
chloride, ammonium sulfate, ammonium chloride and the
like, antifreeze agents, for example, ethylene glycol
25 and propylene glycol, anti-foam agents, for example,
silicone anti-foam agents, anti-bacterial agents and the
like. It may be desirable to incorporate such
components into the emulsion formulation described
herein if it is to be stored for any extended period of
30 time prior to use, particularly under adverse storage
conditions.

The concentration of such formulation
adjuvants present in the synergistic emulsion formulations of the
present invention will range from 0 to 15.0 percent by
35 weight preferably from 0 to 5.0 percent by weight.

The superior, synergistic emulsion formulation
of the present invention is prepared by a novel process

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which consists essentially of the following steps:

1) mixing with agitation from 1.5 to 5.0 percent by weight based on the weight of the final formulation of a polyalkylene glycol ether emulsifier as hereinbefore defined which is in molten form with from 20.0 to 53.5 percent by weight based on the weight of the final formulation of a water carrier, said water carrier being at from 15°C to room temperature and agitating said mixture until said emulsifier is dissolved;

2) adding with agitation to the mixture of Step 1 from 45.0 to 60.0% by weight based on the weight of the final formulation of butachlor herbicide and continuing said agitation until said butachlor herbicide is dispersed throughout the mixture of Step 1 to form an emulsion; and

3) homogenizing said emulsion using a homogenizing means.

As used herein, the term "homogenizing means" refers to any mechanical means which is sufficient to produce the degree of shear sufficient to form an homogenized emulsion. Examples of such homogenizing means are: Colloid Mill, Ross Mill, Waring Blender, "Tekmar" manufactured by the Tekmar Co., P.O. Box 37202, Cincinnati, Ohio 45222, U.S.A., "Gaulin" homogenizer, available from the Gaulin Corporation, 33 Gordon Street, Everett, Mass. 02419, U.S.A.

In order that those skilled in the art may more completely understand the present invention and the preferred methods by which the same may be carried into effect, the following specific examples are offered.

EXAMPLE 1

<u>Component</u>	<u>% by Weight</u>	<u>Grams/Liter</u>
Butachlor (93.1%) ¹	61.78	644.49
Tergitol XH ² (Tergitol 2.00 is a Registered Trade Mark)		20.86
Water	36.22	377.85
Total	100.00	1043.20

¹Technical grade butachlor was used in this example.

²Tergitol XH is a solid, polyalkylene glycol ether, nonionic surfactant manufactured by Union Carbide Corp.

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71.41 Kg of water at room temperature was charged to a batch tank. 3.95 Kg. of the "Tergitol XH" surfactant was melted and thereafter added to the water with agitation. Agitation was continued until all the surfactant was dissolved. 121.81 Kg. of technical grade butachlor was added to the water/emulsifier mixture over a period of 5 minutes. Agitation was continued until all the butachlor was dispersed in the water/emulsifier mixture. Thereafter the mixture was homogenized using a high speed Tekmar homogenizer at about 1800 psig.

Example 2

<u>Component</u>	<u>% by Weight</u>	<u>Grams/Liter</u>
Butachlor (91.2%)	52.89	548.26
Flo Mo XHA ¹	2.00	20.73
15 Water	<u>45.11</u>	<u>467.61</u>
Total	100.0	1036.60

¹ Technical grade butachlor was used in this example.

² Flo Mo XHA is a block co-polymer of propylene oxide/ethylene oxide manufactured by Sellers Chemical Corporation.

24.81 Kg. of distilled water at room temperature was charged to a batch tank. 1.100 Kg. of Flo Mo XHA was melted and added to the water with agitation. Agitation was continued until all the emulsifier was dissolved.

29.09 Kg. of butachlor (91.2%) was added to the water/emulsifier mixture over a period of 5 minutes with agitation. Agitation was continued until the butachlor was dispersed evenly throughout the water/emulsifier mixture and an oil/water emulsion was formed. The emulsion was homogenized using a Tekmar homogenizer.

Utilizing the procedure described in Example 1, the following emulsions were prepared.

Example 3

	<u>Component</u>	<u>% by Weight</u>	<u>Grams/Liter</u>
	Butachlor (89%)	50.37	539.3
	Tergitol XH	3.50	37.5
5	Calcium Chloride	46.13	493.9
	(8.2% aqueous solution)		
		<u>100.00</u>	<u>1070.7</u>

Example 4

	<u>Component</u>	<u>% by Weight</u>	<u>Grams/Liter</u>
10	Butachlor (89%)	50.42	539.3
	Tergitol XD	2.00	21.4
	Calcium Chloride	47.58	509.0
	(8.2% aqueous solution)		
		<u>100.00</u>	<u>1069.7</u>

Example 5

	<u>Component</u>	<u>% by Weight</u>	<u>Grams/Liter</u>
	Butachlor (89%)	63.03	671.27
	Tergitol XH	1.00	10.65
	Calcium Chloride	35.97	383.08
20	(8.2% aqueous solution)		
		<u>100.00</u>	<u>1064.90</u>

The synergistic action of the emulsion formulation of the present invention is illustrated by the following examples. It will be seen that, unexpectedly, the synergistic emulsion formulation of this invention exhibits superior weed control when compared to a commercially available Machete EC formulation, i.e., a formulation containing as the active ingredient 527 grams of technical grade butachlor (92.0%) per kilogram of formulation, which is equivalent to 500 grams of

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active ingredient per liter of formulation. Additionally, rice fields treated with the emulsion formulation of this invention yielded more rice per hectare than did fields treated with Machete EC.

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Example 6

Rice plant seedlings were planted in plots 18 square meters in size. The herbicide treatment was thereafter applied to the plots as shown below. There were three replications for each treatment. The active ingredient, i.e., the herbicide, was applied to the soil as a sandmix. Machete EC and the emulsion formulation of Example 2 were applied to the soil at 0.75 and 1.25 Kg of active ingredient (butachlor) per hectare. Three plots were planted which received no herbicide treatment and these plots served as the control. Visual assessment of weed control was carried out at 40 days after treatment and the results recorded as % inhibition. The results are summarized in Table I.

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Table 1

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<u>FORMULATION</u>	<u>% INHIBITION</u>		
	<u>GRASSES</u>	<u>SEDGES</u>	<u>BROADLEAVES</u>
Emulsion Formulation of Example 2 (500 g active/liter)	86	83	87
25 Machete EC (500 g active/liter)	83	83	85

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The above results demonstrate that the emulsion formulation of the present invention provides 2% better control of broadleaf weed species and 3% better control of grass weed species than does the comparable prior art Machete EC formulation.

Example 7

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Rice fields treated with the butachlor emulsion formulation of Example 2 were compared to rice fields treated with Machete EC and also to rice fields which had no chemical treatment but were hand-weeded. The fields were harvested and the average yields in Kg/Ha

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were compared. The test was conducted according to the the following procedure.

Rice plant seedlings were planted in plots, 30-40 square meters in size. The plots were flooded with 5-10 cm of standing water.

The experimental treatments were as follows:

	<u>Treatment</u>	<u>Rate Kg/Ha</u>
10	1. Emulsion Formulation of Example 2	1.25 1.75 2.25
	2. Machete EC	1.25 1.75 2.25
15	3. Control - Handweeded plots	-- -- --

The herbicide was applied as a sandmix with the active ingredient being applied at rates shown above. The rice plants were allowed to mature and were thereafter harvested using conventional techniques. The yield for each experimental treatment at each rate of application was averaged to give an overall yield per treatment. The results are summarized in Table II.

25	<u>Table II</u>		
	<u>Treatment</u>	<u>Yield of Rice Kg/Ha</u>	<u>% Increase over Control</u>
30	Emulsion Formulation of Example 2 (500 g active/liter)	5210	8.0
	Machete EC (500 g active/liter)	5033	4.3
	Control (hand weeded)	4826	

As shown in Table II, rice fields treated with the butachlor emulsion formulation of the present invention yielded an average increase of 8.0% per hectare as compared to hand-weeded rice fields and an average increase of 3.7% over rice fields tested with

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Machete EC formulation.

The results summarized in Tables I and II indicate that the butachlor emulsion formulations of the present invention are unexpectedly superior to the known Machete EC formulation in that the emulsion formulations of the invention provide superior grass and broadleaf weed control and also crop yield is increased.

The emulsion formulations described herein may be applied in the usual manner using conventional techniques and equipment. For example, the emulsion formulations of the present invention may be sprayed or sprinkled using a "Shaker Bottle"; alternatively, the emulsion formulations described herein may be mixed on a sand carrier and the sand granules may thereafter be applied to the soil. In general, in order to obtain effective weed control 0.75-1.875 Kg per hectare of active ingredient, i.e., butachlor, should be applied per hectare of soil; preferably, 0.75-1.25 Kg/Ha.



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CLAIMS

1. A herbicidal emulsion formulation consisting essentially of the following components:
 - (A) from 30.0 to 70.0% by weight of 2-chloro-2',6'-diethyl-N-(butoxymethyl)acetanilide herbicide;
 - (B) from 0.75 to 5.0% by weight of a polyalkylene glycol ether emulsifier as hereinbefore defined;
 - (C) from 0 to 15.0% by weight of formulation adjuvant; and
 - (D) balance being made up of water.
2. A herbicidal emulsion according to Claim 1 having the following components:
 - (A) 47.5 to 50.0% by weight of 2-chloro-2',6'-diethyl-N-(butoxymethyl)acetanilide herbicide;
 - (B) 1.5 - 3.5% by weight of the polyalkylene glycol ether emulsifier;
 - (C) 0 - 5.0% by weight of a formulation adjuvant selected from the group consisting of sodium chloride, calcium chloride, propylene glycol, ethylene glycol, and methyl violet;
 - (D) balance being made up of water.
3. A herbicidal emulsion according to Claim 1 having the following components:
 - (A) 48.0% by weight 2-chloro-2',6'-diethyl-N-(butoxymethyl)acetanilide herbicide;
 - (B) 2.0% by weight of the polyalkylene glycol ether emulsifier; and
 - (C) balance being made up of water.
4. A herbicidal emulsion according to Claim 2 wherein said formulation adjuvant is calcium chloride.
5. A herbicidal emulsion formulation substantially as described in any one of Examples 1 to 5.
6. A process for preparing a synergistic herbicidal emulsion formulation which process comprises the steps of:



(1) mixing with agitation from 1.5 to 5.0 percent by weight based on the weight of the final formulation of a polyalkylene glycol ether emulsifier as hereinbefore defined which is in molten form with from 20.0 to 53.5 percent by weight based on the weight of the final formulation of a water carrier, said water carrier being maintained at from 15°C. to room temperature wherein said agitation is maintained until said emulsifier is dissolved;

(2) adding, with agitation, to the mixture of Step 1 from 45.0 to 60.0% by weight based on the weight of the final formulation of 2-chloro-2',6'-diethyl-N-(butoxy-methyl)acetanilide herbicide wherein said agitation is continued until said herbicide is dispersed throughout the mixture of Step 1 and an emulsion is formed; and

(3) homogenizing said emulsion.

7. A process according to Claim 6 wherein said water carrier contains 0-5.0% by weight based on the weight of the final formulation of a formulation adjuvant selected from the group consisting of sodium chloride, calcium chloride, propylene glycol, ethylene glycol and methyl violet.

8. A process according to Claim 7 wherein said formulation adjuvant is calcium chloride.

9. A process according to Claim 6 wherein the concentration of said emulsifier is 2.0% by weight based on the weight of the final formulation; and wherein the concentration of said herbicide is 48.0% by weight based on the weight of the final formulation.

10. A process according to Claim 6 wherein the temperature of said water carrier is maintained at room temperature.

11. A process for preparing a synergistic herbicidal emulsion formulation substantially as described in any one of Examples 1 to 5.

12. A synergistic herbicidal emulsion formulation when prepared by the process of any one of Claims 6 to 11.