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(54) Title: HERBICIDAL COMPOSITIONS WITH REDUCED PHYTOTOXICITY

(57) Abstract: A herbicidal composition with reduced phytotoxicity, containing, as a combination, (a) 2-{2-chloro-4-mesy1-3-[(tetrahydrofuran-2-ylmethoxy)methyl]benzoyl}- cyclohexan-1,3-dion; and (b) at least one phytotoxicity-reducing substance selected from the group consisting of benzoisothiazolinone derivatives, benzamide derivatives, benzothiazole derivatives, benzoxazolinone derivatives, benzoxazine derivatives, thiazinyl, isoprothiolane, hexamethylenetetramine, potassium oxalate and vanillin derivatives.



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## Description

Herbicidal composition with reduced phytotoxicity

## Technical Field

The present invention relates to a herbicidal composition with reduced phytotoxicity. More particularly, the present invention relates to a herbicidal composition with reduced phytotoxicity, containing a compound having a herbicidal activity and a specific compound having a phytotoxicity-reducing action.

## Background Art

Herbicidal benzoylcyclohexadiones are known in several literatures (for example, WO 98/29406, WO 00/21924, WO 01/07422 and WO 2004/063187).

Further, 2-{2-chloro-4-mesy-3-[(tetrahydrofuran-2-ylmethoxy)-methyl]benzoyl}-cyclohexan-1,3-dion (general name: described as tefuryltrion in abbreviation) being one of benzoylcyclohexadiones has superior herbicidal activity, but has a problem that it comparatively cause phytotoxicity on crop plants depending on use conditions, applied stage, applied amount, object crop plants and the like. Therefore, compounds having a phytotoxicity-reducing action for suppressing such phytotoxicity are desired to be searched and found.

## Disclosure of Invention

The present inventors have found that a combination shown below reduces a phytotoxic action on a crop generated by a herbicidal active compound and manifests a desired herbicidal effect, leading to completion of the present invention.

There is therefore provided in accordance with the present invention a herbicidal composition with reduced phytotoxicity, containing, as a combination,

- (a) 2-{2-chloro-4-mesy-3-[(tetrahydrofuran-2-ylmethoxy)methyl]benzoyl}-cyclohexan-1,3-dion; and

- (b) at least one compound selected from the group consisting of benzoisothiazolinone derivatives, benzamide derivatives, benzothiazole derivatives, benzoxazolinone derivatives, benzoxazine derivatives, thiazinyl, isoprothiolane, hexamethylenetetramine, potassium oxalate and vanillin derivatives.

Surprisingly, according to the present invention, when the above-mentioned herbicidal compound (a) and the compound (b) are used in combination, the compound (b) can reduce the phytotoxic action of the herbicidal compound (a), and accordingly, the herbicidal compound can be used in a crop cultivation district in which the use of the herbicidal compound was difficult, which is an extremely advantageous effect. Further, it has been found that such a phytotoxicity-reducing action of the compound (b) is a novel and extremely useful action not known until now.

Of the compounds (b), examples of benzoisothiazolinone derivatives include 1,2-benzoisothiazolin-3-one and a sodium salt thereof, probenazole and saccharin.

Examples of benzamide derivatives include phthalimide and benzamide.

Examples of benzothiazole derivatives include 2-hydroxybenzothiazole.

Examples of benzoxazolinone derivatives include 2-benzoxazolinone.

Examples of benzoxazine derivatives include 2H-1,4-benzoxazin-3(4H)-one and isatic anhydride.

Examples of vanillin derivatives include vanillin, vanillic acid, vanillyl alcohol and ethylvanillin.

The compounds (b) are those well known in the field of organic chemistry.

The compound (b) has itself a phytotoxicity-reducing action, and in the herbicidal composition with reduced phytotoxicity of the present invention, the mixing ratio of the herbicidal compound (a) and the compound (b) can be varied in a relatively wide range depending on the kind of the herbicidal compound, and the application stage, application district and application method of the composition, and the like. Generally, the compound (b) can be used in a ratio of 0.001 to 5 parts by weight, preferably 0.004 to 3 parts by weight per part by weight of the herbicidal compound (a).

The herbicidal composition with reduced phytotoxicity of the invention is most preferably characterized in that it can manifest a selective herbicidal action between crop plants and weeds. Such a selective herbicidal action renders the composition useful in the following plants.

Dicotyledon weeds of the genera such as mustard (*Sinapis*), cress (*Lepidium*), bed straw (*Galium*), chickweed (*Stellaria*), goosefoot (*Chenopodium*), annual nettle (*Urtica*), groundsel (*Senecio*), pigweed (*Amaranthus*), purslane (*Portulaca*), cocklebur (*Xanthium*), morning glory (*Ipomoea*), knotweed (*Polygonum*), ragweed (*Ambrosia*), spear thistle (*Cirsium*), sow thistle (*Sonchus*), field cress (*Rorippa*), deadnettle (*Lamium*), speedwell (*Veronica*), thornapple (*Datura*), violet (*Viola*), hemp-nettle (*Galeopsis*), poppy (*Papaver*), knapweed (*Centaurea*), gallant soldier (*Galinsoga*), *Rotala indica*, Falsepimpernel (*Lindernia*), and the like;

dicotyledon cultures of the genera such as cotton (*Gossypium*), soya bean (*Glycine*), chard/beet (*Beta*), carrot (*Daucus*), bean (*Phaseolus*), pea (*Pisum*), potato (*Solanum*), flax (*Linum*), sweet potato/morning glory (*Ipomoea*), broad bean (*Vicia*), tobacco (*Nicotiana*), tomato (*Lycopersicon*), groundnut (*Arachis*), field musard/napa/turnip/cabbage (*Brassica*), lettuce (*Lactuca*), cucumber/melon (*Cucumis*) and marrow (*Cucurbita*);

monocotyledon weeds of the genera such as barnyard grass (*Echinochloa*), foxtail/millet (*Setaria*), wild millet (*Panicum*), crabgrass (*Digitaria*), herd's-grass/timothy (*Phleum*), bluegrass/meadow grass (*Poa*), fescue (*Festuca*),

goosegrass (*Eleusine*), yardgrass (*Lolium*), cheat (*Bromus*), oats (*Avena*), galingale/papyrus/nutgrass (*Cyperus*), sorghum (*Sorghum*), quackgrass (*Agropyron*), *Monochoria*, fimbriistylis (*Fimbristylis*), arrowhead (*Sagittaria*), spikerush (*Eleocharis*) bulrush (*Scirpus*), paspalum (*Paspalum*), *Ischaemum*, redtop (*Agrostis*), meadow foxtail (*Alopecurus*) and Bernuda grass (*Cynodon*); and

monocotyledon cultures such as rice (*Oryza*), maize/pop corn (*Zea*), wheat (*Triticum*), barley (*Hordeum*), oats (*Avena*), rye (*Secale*), sorghum (*Sorghum*), millet (*Panicum*), sugarcane (*Saccharum*), pineapple (*Ananas*), asparagus (*Asparagus*) and onion (*Allium*).

Further, the composition according to the invention is in no way restricted to these plants but also embraces other plants, in the same way.

The composition according to the invention may be used for controlling weeds in perennial plant cultivation, such as forestation, ornamental planting, fruit farm, grape farm, citrus fruit farm, nut fruit farm, banana cultivation field, coffee cultivation field, tea cultivation field, rubber cultivation field, Guinean oil palm cultivation field, cocoa cultivation field, small fruit farm and hop cultivation field. Also, the present composition may be used for selectively controlling weeds in annual plant cultivation.

The composition may be formulated into a conventional preparation for use in controlling weeds. Examples of the preparation form include dusts, granules, wettable powders, water dispersible granules, emulsifiable concentrates, flowable formulation or aqueous suspensions, emulsions, jumbo formulation-packages, floatable granules and the like.

These preparations may be prepared in a known manner per se, for example, by mixing a herbicidal compound (a) and a compound (b) with a liquid or solid diluent or carrier, optionally, together with surfactants or formulation adjuvants thereby to prepare the formulation.

Examples of the solid carrier and/or solid diluent which can be used in formulating the composition of the present invention include:

inorganic substances made of minerals such as clay, kaolin, talc, calcium carbonate, diatomaceous earth, zeolite, bentonite, acid clay, activated clay, attapulgite clay, vermiculite, pearlite, pumice, silica sand, silica and the like;

synthetic products such as hydrophilic silica, hydrophobic silica, white carbon such as calcium silicate and the like, synthetic zeolite, titanium dioxide and the like;

vegetable organic substances such as soy bean powder, tobacco powder, corn cob powder, walnut powder, wheat powder, wood powder, starch, crystalline cellulose and the like;

synthetic or natural polymer compounds such as coumarone resins, petroleum resins, alkyd resins, polyalkylene glycol, ketone resins, ester gum and the like;

waxes such as carnauba wax, beeswax and the like; and

water-soluble substances such as urea, lactose, sucrose, ammonium sulfate, potassium chloride and the like.

Examples of the appropriate liquid carrier and/or liquid diluent include:

fats and oils such as palm oil, rape seed oil, corn oil, soy bean oil, rice bran oil and the like;

paraffin type or naphthene type hydrocarbon-based solvents such as kerosene, mineral oil, spindle oil, white oil, n-paraffin, isoparaffin, naphthene and the like;

aromatic hydrocarbon-based solvents such as xylene, alkylbenzene, alkylnaphthalene and the like;

alcohols such as ethanol, benzyl alcohol, isopropanol, cyclohexanol and the like;

poly-hydric alcohols such as ethylene glycol, diethylene glycol, propylene glycol, polyethylene glycol and the like;

ether alcohols such as ethylene glycol ethyl ether, ethylene glycol phenyl ether, diethylene glycol ethyl ether, diethylene glycol butyl ether and the like;

ketones such as methyl ethyl ketone, diisobutyl ketone, cyclohexanone, acetophenone, isophorone,  $\gamma$ -butyrolactone and the like;

esters including fatty acid methyl esters such as palm oil fatty acid methyl ester and the like, dibasic acid methyl esters such as succinic dimethyl ester, glutamic dimethyl ester, adipic dimethyl ester and the like, and ethyl acetate, amyl acetate, ethylene glycol acetate, diethylene glycol acetate and the like;

ethers such as dioxane, tetrahydrofuran and the like;

polar solvents such as dimethylformamide, dimethylacetamide, dimethyl sulfoxide, N-alkylpyrrolidone and the like, or water and the like.

Additionally, for the purpose of emulsification, dispersion, wetting, spreading, decomposition prevention, effect reinforcement and the like of an active ingredient and/or for the purpose of improvement of physical properties of a preparation (disintegrating property control, flowability improvement, freezing prevention, rain resistance impartment and the like), surfactants, binders and other auxiliary agents can be used. As the surfactant, any types of compounds such as nonionic, anionic, cationic and ampholytic compounds can be used, and usually, nonionic and/or anionic compounds can be used. As suitable nonionic surfactants, mentioned are, for example, sorbitan fatty acid esters, polyoxyalkylene sorbitan fatty acid esters, sucrose fatty acid esters; polyoxyalkylene fatty acid esters, polyoxyalkylene resin acid esters, polyoxyalkylene fatty diesters; polyoxyalkylene castor oil, polyoxyalkylene hardened castor oil; polyoxyalkylene alkyl ethers; polyoxyalkylene alkylphenyl ethers,

polyoxyalkylene dialkylphenyl ethers, polyoxyalkylene alkylphenyl ether formalin condensates; polyoxyethylene/polyoxypropylene block polymers, alkylpolyoxyethylene/polyoxypropylene block polymer ethers, alkylphenylpolyoxyethylene/polyoxypropylene block polymer ethers; polyoxyalkylenealkylamines, polyoxyalkylene fatty amides; polyoxyalkylene benzylphenyl (or phenylphenyl) ethers, polyoxyalkylene styrylphenyl (or phenylphenyl) ethers; polyoxyalkylene ether and ester types silicon- and fluorine-based surfactants, and the like.

Examples of suitable anionic surfactants include alkylsulfate salts, polyoxyalkylene alkyl ether sulfate salts, polyoxyalkylene alkylphenyl ether sulfate salts, polyoxyalkylene benzyl (or styryl) phenyl (or phenylphenyl) ether sulfate salts, polyoxyethylene/polyoxypropylene block polymer sulfate salts; paraffin (alkane) sulfonate salts,  $\alpha$ -olefin sulfonate salts, dialkyl sulfosuccinate salts, alkylbenzene sulfonate salts, mono or dialkylnaphthalene sulfonate salts, naphthalene sulfonate-formalin condensate salts, alkyldiphenyl ether disulfonate salts, ligninsulfonate salts, polyoxyalkylene alkylphenyl ether sulfonate salts, polyoxyalkylene ether sulfosuccinic acid half ester salt; fatty acid salts, N-methylfatty sarcosinate, resin acid salts; polyoxyalkylene alkyl ether phosphate salts, polyoxyalkylene mono or dialkylphenyl ether phosphate salts, polyoxyalkylene benzylated (or styrylated) phenyl (or phenylphenyl) ether phosphate salts, polyoxyethylene/polyoxypropylene block polymer phosphate salts; polyacrylic acid salts, polycarboxylic acid salts and the like.

The cationic surfactant includes ammonium types such as alkyltrimethyl ammonium chloride, methyl-polyoxyethylene-alkyl ammonium chloride, alkyl-N-methylpyridinium bromide, mono or dialkylmethylated ammonium chloride, alkylpentamethylpropylenediamine dichloride and the like; benzalkonium types such as alkyldimethylbenzalkonium chloride, benzethonium chloride and the like.

The ampholytic surfactant includes betaine types such as dialkyldiaminoethyl betaine, alkyldimethylbenzyl betaine and the like.



As the binder, exemplified are bentonite, casein, gelatin, starch, dextrin, ligninsulfonate, alginate, gum Arabic, xanthan gum, carboxymethylcellulose, methylcellulose, polyvinyl alcohol, polyvinylpyrrolidone and the like.

As other auxiliary agents, disintegration controlling agents, anti-freezing agents, antiseptics, flowability improving agents, defoaming agents, humectants, spreading agents, rain resistance imparting agents and the like are mentioned.

The above-mentioned carriers and surfactants, binding agents and various auxiliary agents can be appropriately used singly or in combination depending on the object in view of the form of a preparation, application stage and the like.

The preparation can contain, in general, the herbicidal compound (a) and the compound (b) in a total concentration of 0.1 to 95 wt%, preferably 0.5 to 90 wt%.

The composition of the present invention can be used as it is or in the form of its preparation for preventing and controlling weeds, alternatively, can also be mixed in a tank in use. Further, the composition can be mixed with other known active compounds, particularly, active compounds usually used, for example, fungicide, insecticides, plant growth regulating agents, plant nutrition supplements, soil improving agents, fertilizers and the like.

The composition of the present invention can be used as it is or in the form of its preparation, or in the form of application forms prepared by further diluting the preparation, for example, in the form of ready-to-use solutions, emulsions, suspensions, dusts, wettable powders or granules. Those forms can be applied to paddy field in a conventional manner, for example, watering, spraying, atomizing, dusting, spraying granules and the like.

The composition of the present invention can be used both before and after germination of a plant. According to the present invention, phytotoxicity can be reduced also by applying the compound (b) before or after application of the herbicidal compound (a).

According to the present invention, the application amount of the composition can be varied in a substantial range. The application amount can be, for example, 0.01 to 10 kg/ha, preferably 0.5 to 5 kg/ha in terms of the total amount of the herbicidal compound (a) and the compound (b).

The excellent advantageous effect of the composition of the present invention will be further illustrated specifically by the following examples. However, the present invention should not be limited to only these examples.

## EXAMPLES

Biological test example and preparation example  
(Test compound)

- (a): tefuryltrion
- (b-1): sodium salt of 1,2-benzisothiazolin-3-one
- (b-2): probenazole
- (b-3): phthalimide
- (b-4): benzamide
- (b-5): 2-hydroxybenzothiazole
- (b-6): 2-benzoxazolinone
- (b-7): 2H-1,4-benzoxazin-3(4H)-one
- (b-8): isatoic anhydride
- (b-9): hexamethylenetetramine
- (b-10): potassium oxalate
- (b-11): vanillin
- (b-12): vanillic acid
- (b-13): ethylvanillin

## Test Example 1

### Phytotoxicity-reducing effect test (I)

#### Test method:

30 ml each of drug solutions containing 1 ppm of the compound (a) and 20 ppm of a test compound belonging to the compound (b) was charged in a standard bottle with a volume of 50 ml.

After cutting the root portion of rice plant at 2.0 to 2.2-leaf stage to 1 cm, it was immersed in drug solution and left at rest in a growth cabinet (at a temperature of 25°C, a humidity of 80%, an irradiation light quantity of 6 KLx and 12 hour-light-dark cycle) for 3 days. Water which evaporates during being left at rest was supplied so that water volume was maintained at 30 ml. After the rice plant was taken out after 3 days and the drug solution adhering on the rice plant was rinsed with water, it was transferred to water culture medium (30 ml) containing a fertilizer and grown in the growth cabinet (at a temperature of 25°C, a humidity of 80%, an irradiation light quantity of 6 KLx and 12 hour-light-dark cycle).

For the evaluation, the foliage portion and root portion of rice plant treated in each treatment compartment marked by the kind of the compound (b) contained in the drug solution were observed by the naked eye, after 12 days of immersion in the drug solution.

Evaluation was carried out according to the following evaluation standard.

+++; Growth equal to no addition. The rhizogenesis of root and the growth of foliage portion and root portion were accelerated.

++; The rhizogenesis of root and the growth of foliage portion and root portion were accelerated in comparison with the single agent of the compound (a).

+; They were similar level as the single agent of the compound (a).

-; They were suppressed more than the single agent of the compound (a).

The test result is shown in Table 1.

[Table 1]

Compound (b)	Result of visual observation	
	Foliage portion	Root portion
(b-1)	++	+++
(b-2)	-	+++
(b-3)	+++	++
(b-4)	++	++
(b-5)	++	++
(b-6)	+++	+++
(b-7)	+++	-
(b-8)	++	-
(b-9)	++	+++
(b-10)	++	+++
(b-11)	+++	+++
(b-12)	+++	-
(b-13)	+++	++

## Test Example 2

### Phytotoxicity-reducing effect test (II)

#### Test method:

1/5000a Wagner's pots filled with paddy soil were provided in a KOITOTRON (artificial weather machine) and a greenhouse. The temperature setting of KOITOTRON was carried out at the daily ranges of 13°C (12 hrs) to 15°C (12 hrs) from transplantation to after 14 days, and 20°C (12 hrs) to 25°C (12 hrs) after 14 days. Water in the bench where pots were provided was cooled so that the temperature in the greenhouse was set at 12.5°C in average from transplantation to after 14 days. Cooling was stopped after 14 days and the temperature was at 21.8°C in average.

Three paddy rice plants (brand; NIHONBARE, 2.2 to 2.6-leaf stage) per one pot were transplanted at a transplantation depth of 2 cm. After the transplantation, the pots were under irrigation state of 3 cm and the water level was kept during the test period.

After 5 days of the transplantation, the fixed amounts of each of the granule with 3% of the compound (a) and the granules with the compound (a) plus the compound (b) with fixed concentrations were applied to respective pots in irrigation water surface.

Water leakage treatment at 0.5 cm/day was carried out for 7 days from one day after the treatment.

For the evaluation, the measurements of the number of stems, the weight of root and the weight of foliage were carried out after 5 weeks of the treatment with the compounds.

Evaluation was carried out according to the following evaluation standard.

It is described as comparison %, referring to the single agent treated with the compound (a) as 100%.

Exceeding 100%; Phytotoxicity was reduced in comparison with the treatment with the single agent of the compound (a).

100 to 90%; Although influence was observed in comparison with the treatment with the single agent of the compound (a), recovery is expected and influence is little for yield and the like.

90 to 80%; Stronger influence was observed in comparison with the treatment with the single agent of the compound (a).

Less than 80%; Clearly stronger influence was observed in comparison with the treatment with the single agent of the compound (a).

The test result is shown in Table 2.

Table 2

	Treatment amount (kg/ha)	KOITORON			Greenhouse		
		Number of stem	Weight of root	Weight of foliage	Number of stem	Weight of root	Weight of foliage
Granule with 3% of compound (a)	15	13.0	0.63g	3.30g	3.5	0.29g	3.90g
Added with 0.1% of compound (b-9)	15	-	-	-	135.7%	104.4%	113.3%
Added with 0.5% of compound (b-9)	15	148.0%	160.0%	134.6%	128.6%	106.1%	118.6%
Added with 1.0% of compound (b-9)	15	132.0%	146.4%	132.9%	-	-	-
Added with 0.1% of compound (b-1)	15	164.0%	168.8%	184.5%	157.1%	112.3%	111.0%
Added with 0.5% of compound (b-1)	15	156.0%	186.4%	177.7%	142.9%	98.2%	102.7%
Added with 0.2% of compound (b-1) and 0.2% of compound (b-9)	15	132.0%	145.6%	174.5%	142.9%	100.0%	111.0%
No treatment	-	31	2.23g	10.3g	7.3	0.38g	5.57g

#### Preparation Example 1 (extruded granule)

To a mixture of 3 parts by weight of a compound (a), 0.1 part by weight of a compound (b-2), 30 parts by weight of sodium bentonite (montmorillonite), 1 part by weight of sodium tripolyphosphate, 3 parts by weight of ligninsulfonate and 61.4 parts by weight of calcium carbonate was added 25 parts by weight of water containing 0.5 part by weight of dialkylsulfosuccinic acid sodium salt and 1 part by weight of polycarboxylic acid sodium salt and the mixture was kneaded well, and processed into granules of 500 to 1700  $\mu\text{m}$  by an extrusion type granulator and dried at 80°C to give granules.

#### Preparation Example 2 (extruded granule)

To a mixture of 3 parts by weight of a compound (a), 0.1 part by weight of a compound (b-2), 30 parts by weight of sodium bentonite (montmorillonite), 2 parts by weight of sodium hexametaphosphate and 60.2 parts by weight of clay was added 22 parts by

weight of water containing 0.2 part by weight of dialkylsulfosuccinic acid sodium salt, 4 parts by weight of polyoxyalkylene tristyrylphenyl ether sulfuric acid sodium salt and 0.5 part by weight of polycarboxylic acid sodium salt and the mixture was kneaded well, and processed into granules of 500 to 1700  $\mu\text{m}$  by an extrusion type granulator and dried at 80°C to give granules.

#### Preparation Example 3 (fine granule)

73.2 Parts by weight of clay mineral particles having a particle size distribution of 0.2 to 0.7 mm was charged in a vessel rotation type mixer, and 15 parts by weight of a compound (a) and 0.3 part by weight of a compound (b-2) were sprayed together with 10 parts by weight of a liquid diluent under rotation, and 1.5 parts by weight of white carbon was added, to obtain fine particles.

#### Preparation Example 4 (flowable formulation)

A mixture of 6 parts by weight of a compound (a), 0.1 part by weight of a compound (b-2), 10 parts by weight of propylene glycol, 5 parts by weight of polyoxyalkylene tristyrylphenyl ether, 1 part by weight of Xanthan gum, 0.1 part by weight of silicone oil emulsion and 77.8 parts by weight of water was stirred well, then, milled using DYNO-MILL (type KDL), to give suspension concentrate.

## CLAIMS

1. A herbicidal composition with reduced phytotoxicity, containing, as a combination,
  - (a) 2-{2-chloro-4-mesy-3-[(tetrahydrofuran-2-ylmethoxy)methyl]benzoyl}-cyclohexan-1,3-dion; and
  - (b) at least one compound selected from the group consisting of benzoisothiazolinone derivatives, benzamide derivatives, benzothiazole derivatives, benzoxazolinone derivatives, benzoxazine derivatives, thiazinyl, isoprothiolane, hexamethylenetetramine, potassium oxalate and vanillin derivatives.
  
2. The herbicidal composition with reduced phytotoxicity according to Claim 1, wherein the compound (b) as defined in Claim 1 is at least one compound selected from the group consisting of 1,2-benzoisothiazolin-3-one and a sodium salt thereof, probenazole, saccharin, phthalimide, benzamide, 2-hydroxybenzothiazole, 2-benzoxazolinone, 2H-1,4-benzoxazin-3(4H)-one, isatic anhydride, thiazinyl, isoprothiolane, hexamethylenetetramine, potassium oxalate, vanillin, vanillic acid, vanillyl alcohol and ethylvanillin.