

SUPERINTENDENCIA DE INDUSTRIA Y COMERCIO



No. 14-122897- -00007-0000

Señor Superintendente
SUPERINTENDENCIA DE INDUSTRIA Y COMERCIO
E. S. D.

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Evento : 001
Actuación : 412

Referencia : Expediente No. **14.122.897**
Asunto : Solicitud de patente: **"PROCESO PARA PRODUCIR UNA FORMULACIÓN PESTICIDA EN SUSPENSIÓN DE UN HIDRATO DE SULFONILUERA"**
Solicitante : SUMITOMO CHEMICAL COMPANY, LIMITED
Fecha de solicitud : 26 de Diciembre de 2011

I. RECURSO VÍA GUBERNATIVA

Yo, Luz Clemencia DE PÁEZ, en mi condición de apoderada de la sociedad **SUMITOMO CHEMICAL COMPANY, LIMITED** solicitante de la patente de la referencia, por el presente escrito atentamente manifiesto a usted que debidamente notificada de la Resolución No. **72862** del 1 de Diciembre de 2014, interpongo en su contra el recurso de **REPOSICIÓN**, para que sea revocada y en consecuencia se conceda el privilegio de patente de invención para **"PROCESO PARA PRODUCIR UNA FORMULACIÓN PESTICIDA EN SUSPENSIÓN DE UN HIDRATO DE SULFONILUERA"** a favor de **SUMITOMO CHEMICAL COMPANY, LIMITED**.

II. OPORTUNIDAD DE LA IMPUGNACIÓN

La Resolución No. **72862** del 1 de Diciembre de 2014, con fecha de publicación listado del 9 de Diciembre de 2014, fecha de vencimiento del 9 de Enero de 2015, y fecha del término legal 19 de Enero de 2015.

III. LA DECISIÓN RECURRIDA

"EXCELENCIA LEGAL EN UN MUNDO SIN FRONTERAS"

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- 3.1 Mediante la Resolución objeto de la presente impugnación, el Superintendente de Industria y Comercio decidió:
- 3.1.1 Denegar la patente de invención a la creación titulada: **“PROCESO PARA PRODUCIR UNA FORMULACIÓN PESTICIDA EN SUSPENSIÓN DE UN HIDRATO DE SULFONILUERA”**.
- 3.2 En la parte motiva de la providencia, se mencionaron los siguientes fundamentos:
- 3.2.1 Mediante escrito radicado bajo el No. 14-122897-00004-0000 del 26 septiembre de 2014, la solicitante respondió oportunamente el requerimiento formulado y presentó nuevas reivindicaciones 1 a 6 que reemplazan las originalmente presentadas. Se aceptó el nuevo capítulo reivindicatorio.
- 3.2.2 El Examinador concluye que as reivindicaciones 1 a 6, no cumplen con el requisito de nivel inventivo.

IV. OBJETO DEL RECURSO

De acuerdo con los argumentos que expongo a continuación, solicito atentamente a su Despacho lo siguiente:

1. Revocar la Resolución No. **72862** del 1 de Diciembre de 2014.
2. Conceder el privilegio de patente de invención para **“PROCESO PARA PRODUCIR UNA FORMULACIÓN PESTICIDA EN SUSPENSIÓN DE UN HIDRATO DE SULFONILUERA”** a favor de **SUMITOMO CHEMICAL COMPANY, LIMITED**.

V. FUNDAMENTOS DEL RECURSO

Respetuosamente me permito disentir de lo expuesto por ese Despacho en la Resolución objeto del presente recurso, toda vez que la presente solicitud sí cumple con los requerimientos de novedad y nivel inventivo exigidos en la Decisión 486, como se demostrará a continuación:

5.1 La presente solicitud sí tiene nivel inventivo

A pesar de lo anterior y con el fin de demostrar que la presente solicitud cumple no sólo con el requerimiento de novedad sino también con el requerimiento de nivel inventivo, se pone de presente lo siguiente:

El artículo 18 de la Decisión 486 señala lo siguiente:

***“Artículo 18.-** Se considerará que una invención tiene nivel inventivo, si para una persona del oficio normalmente versada en la materia técnica correspondiente, esa invención no hubiese resultado obvia ni se hubiese derivado de manera evidente del estado de la técnica”.*

Consecuentemente, una invención se considerará inventiva si la misma no hubiese resultado obvia a partir del arte previo, para una persona del oficio normalmente versada en la materia. Esto a su vez implica que el Examinador a cargo de esta evaluación debe colocarse en el momento exacto en el que el inventor buscaba resolver el problema técnico contemplado en la solicitud y para el cual poseía la literatura y el conocimiento disponibles en dicho momento.

Siguiendo entonces los lineamientos de la Jurisprudencia aplicable al análisis del nivel inventivo de una solicitud de patente, se considera que una anterioridad o una combinación de dos anterioridades puede afectar dicho requerimiento de patentabilidad, si una persona medianamente versada en la materia encuentra enseñanzas o sugerencias claras que, a partir de un conocimiento medio en dicho campo, le permitan llegar de manera obvia a la invención reivindicada. Adicionalmente, se considera que una mejora sustancial, un efecto inesperado o una ventaja con respecto al arte previo es un indicio de nivel inventivo, por cuanto aporta un salto tecnológico en la regla técnica.

Respecto a lo anterior, el Tribunal de Justicia de la Comunidad Andina ha expuesto en la Interpretación Prejudicial del proceso 13-IP-2010:

“En este orden de ideas, se puede concluir que una invención goza de nivel inventivo cuando a los ojos de un experto medio en el asunto de que se trate, se necesita algo más que la simple aplicación de los conocimientos técnicos en la materia para llegar a ella, es decir, que de conformidad con el estado de la técnica el invento no es consecuencia clara y directa de dicho estado”.

[señalado fuera del texto]

Por lo anterior, se entiende que para que una persona medianamente versada en la materia pudiera derivar de manera obvia una invención, requeriría simplemente aplicar sus conocimientos técnicos. En contraste, todo aquello que va más allá de la simple aplicación de los conocimientos técnicos y que no ha sido revelado en el estado de la técnica, debe ser considerado inventivo.

A este respecto, respetuosamente manifestamos nuestro desacuerdo con el análisis llevado a cabo por el Examinador y con las conclusiones del mismo y solicitamos atentamente la reconsideración de las razones por las cuales se decidió negar la patente, con base en la siguiente información:

El problema planteado en la presente solicitud consiste en la necesidad de proporcionar compuestos derivados de sulfonilurea con actividad herbicida y formulaciones en suspensión que los contienen, en las cuales no se produzca un aumento del tamaño de partícula del herbicida durante el almacenamiento. Este problema es el que precisamente soluciona la presente invención con el proceso acá descrito toda vez que se proporciona un hemi-hidrato con las características arriba señaladas y deseadas.

Sin embargo, de acuerdo con las afirmaciones realizadas por el señor Examinador, se formaría un hemihidrato en un proceso para producir una formulación en suspensión como se describe en el Ejemplo de Producción Comparativo 1 (Formulación de suspensión (9)), y por lo tanto el crecimiento de partículas no debería ocurrir en la formulación de suspensión (9) tal y como sucede en las otras formulaciones en suspensión que son obtenidas en los Ejemplos de Producción 3-10 (Formulaciones de suspensión (1) a (8)). Sin embargo, se observó un crecimiento significativo de las partículas en la formulación de suspensión (9) como se muestra en el Ejemplo Experimental 1 de la presente solicitud. Es evidente que dichos resultados son aparentemente incompatibles con la opinión del señor Examinador.

Incluso si un hidrato se forma en un proceso de formulación, hay una diferencia significativa en el crecimiento de partículas entre un proceso que comprende la adición del compuesto de fórmula (I), incluyendo un hidrato del mismo y un proceso que comprende la adición de un compuesto no hidrato de fórmula (I), es decir, sin incluir un hidrato del mismo. Por lo tanto, queda en evidencia, una vez más que la afirmación señalada por el señor Examinador respecto al nivel inventivo, es errónea.

El señor Examinador también menciona que la memoria descriptiva de la presente solicitud no proporciona un avance técnico real obtenido por la invención reivindicada. Sin embargo, la evaluación de los cambios en el tamaño de las partículas en las formulaciones en suspensión que se muestran en el Ejemplo Experimental 1 de la presente solicitud, es la evaluación de la estabilidad de almacenamiento de las formulaciones en suspensión.

La ley de Stokes, ampliamente conocida en el estado de la técnica, presenta una relación entre el tamaño de las partículas y la velocidad de sedimentación de las partículas.

$$V_t = \{(\rho_p - \rho) \cdot G \cdot D_p^2\} / 18\mu$$

donde

V_t : Velocidad final de sedimentación (m/s);

ρ_p : Densidad de la partícula (kg/m³);

ρ : Densidad del agua (kg/m³);

G : Aceleración de la gravedad (m/s²);

μ : Viscosidad total del sistema; y

D_p : Diámetro de partícula (m).

En la fórmula, ρ_p y ρ depende del tipo y cantidad de componentes y G es un número constante. La velocidad de sedimentación de las partículas varía en función de los cambios en D_p y μ . Específicamente, la velocidad de sedimentación es proporcional al cuadrado de D_p e inversamente proporcional a μ .

En la formulación de la suspensión comparativa (Formulación de suspensión (9)) de la presente solicitud, se observó el aumento de D_p (diámetro medio en volumen en este caso). Por lo tanto, se asume que la velocidad de sedimentación de las partículas aumenta drásticamente durante el almacenamiento. No se observó el aumento del tamaño de partícula después de una semana de almacenamiento a 50°C. Sin embargo, el diámetro medio en volumen de las partículas se incrementó en alrededor de 6,75 veces después de dos semanas de almacenamiento a 50°C, y por cerca de 5,7 veces después de una semana de almacenamiento a 60°C, en comparación con el que se obtiene inmediatamente después de la producción. Debido al aumento significativo del tamaño de partícula, el aumento de la velocidad de sedimentación de las partículas se calcula aproximadamente como por 45,6 veces (= el cuadrado de 6,75 veces) después de un

almacenamiento de dos semanas en 50°C, y por 32,5 veces (= el cuadrado de 5,7 veces) después de una semana de almacenamiento a 60°C, en comparación con el que se obtiene inmediatamente después de la producción. Así, es claro que con el proceso acá reivindicado se soluciona el problema técnico planteado en la presente solicitud y por lo tanto, respetuosamente se solicita a su Despacho otorgar la presente invención teniendo en cuenta que soluciona un problema técnico real y el avance que aporta frente a las enseñanzas del estado de la técnica.

En las formulaciones en suspensión de acuerdo con la presente invención, el aumento de la velocidad de sedimentación de las partículas se considera que está impedido por el uso del compuesto de fórmula (I), incluyendo un hidrato del mismo. Incluso en la formulación de suspensión (8) que contiene el compuesto de fórmula (I) incluyendo sólo 0,2% de un hemihidrato del mismo, el aumento de la velocidad de sedimentación de las partículas se supone que es sólo alrededor de 2,4 veces (= el cuadrado de 1,56 veces) después de dos semanas de almacenamiento a 50°C, y cerca de 2,8 veces (= el cuadrado de 1,67 veces) después de una semana o dos de almacenamiento a 60°C. En la formulación de suspensión (1) que contiene sólo un hemihidrato del compuesto de fórmula (I), no se observó el aumento del tamaño de partícula, es decir, la velocidad de sedimentación de las partículas se considera que no presenta cambios en absoluto, después de dos semanas de almacenamiento a 50°C o 60°C.

Como se mencionó anteriormente, el ejemplo experimental 1 de la presente solicitud muestra que la velocidad de sedimentación de las partículas en las formulaciones en suspensión de acuerdo con la presente invención es al menos 1/16 a 1/19 en comparación con la formulación de la suspensión comparativa. Por lo tanto, es evidente que en el capítulo descriptivo se describe un avance técnico real obtenido por la presente invención.

Además, los datos experimentales adicionales muestran una relación entre el tamaño de las partículas y la estabilidad de las formulaciones. La prueba adicional es una prueba de simulación que utiliza suspensiones que contienen partículas no relacionadas con la presente invención. En la prueba, se observaron los cambios en la estabilidad de las suspensiones por las diferencias de tamaño de partícula.

Cada suspensión contiene partículas que tienen un tamaño medio de partícula de 1.8 a 3.6 micras se centrifugó a 800 rpm y se midió una cantidad residual de la

suspensión en un tubo de centrifugación. Cuanto mayor sea el tamaño de partícula, mayor es la cantidad residual de la suspensión en un tubo. Así que, la fluidez de la suspensión se reduce y por lo tanto la suspensión tiende a mantenerse en un tubo. La mayor cantidad residual de la suspensión en un tubo genera un buen estado de dispersión que es difícil mantener. Por lo tanto, es evidente que el tamaño de partícula más grande genera un estado de dispersión deficiente y disminuye la estabilidad de la suspensión.

Así, en los argumentos arriba presentados se pueden identificar claramente dos indicios de presencia de nivel inventivo de acuerdo con el numeral 2.13.5.3.1 de la "GUÍA PARA EXAMEN DE SOLICITUDES DE PATENTE DE INVENCION Y MODELO DE UTILIDAD" de su despacho:

- Superación de dificultades no resueltas por las técnicas rutinarias, ya que son evidentes las mejoras en la evaluación de la estabilidad de almacenamiento de las formulaciones en suspensión, teniendo en cuenta el tamaño y la velocidad de sedimentación de la partícula.
- Efecto técnico sorprendente, ya que la velocidad de sedimentación de las partículas en las formulaciones en suspensión de acuerdo con la presente invención es al menos 1/16 a 1/19 en comparación con la formulación de la suspensión comparativa

Por lo tanto, se le debe otorgar una compensación a mi representada que demuestra nuevos hechos (efectos inesperados) a las enseñanzas descritas en el estado de la técnica y que contribuyen con el desarrollo de la tecnología.

En consecuencia, un técnico con conocimientos medios en la materia no estaba en condiciones de plantearse el problema, tampoco de resolverlo en la forma en que se reivindica y tampoco de prever el resultado, por lo que la presente solicitud posee la altura inventiva para que se le conceda el privilegio solicitado.

Así, la presente invención es inventiva toda vez que los ejemplos y la descripción tal como fue originalmente presentada muestran claramente la mejora aportada al estado del arte por las formulaciones en suspensión obtenidas con el proceso acá reivindicado en

términos de estabilidad durante el almacenamiento y la velocidad de sedimentación de las partículas en dichas formulaciones. Adicionalmente, es claro al señalar que el efecto (estabilidad, tamaño de partícula y velocidad de sedimentación) de las formulaciones en suspensión obtenidos con el proceso acá reivindicado no podía haber sido esperado por un técnico medio a partir de las enseñanzas de los documentos citados tal como se demostró previamente, en consonancia con los indicios de nivel inventivo que menciona tanto el Manual Andino de Patentes como el "INSTRUCTIVO EXAMEN DE SOLICITUDES DE PATENTES DE INVENCION Y MODELO DE UTILIDAD" de su Despacho, la invención tiene nivel inventivo.

Conclusión

El documento D1, el documento D2, o su combinación no resultan anterioridades válidas capaces de afectar el nivel inventivo de la invención de la presente solicitud.

En consecuencia, el mérito investigativo de la presente invención ha sido reconocido por otras oficinas de patentes del mundo, las cuales han otorgado patentes para invenciones equivalentes. Tal es el caso de Estados Unidos, patente No. US8907085 B2.

A este respecto, deseamos poner de presente que si bien cada país es autónomo de conceder o negar una solicitud luego de la realización del estudio de fondo, es claro que el hecho de que la novedad y el nivel inventivo de una invención hayan sido reconocidos por varias oficinas a nivel mundial, es un indicio de la patentabilidad de la misma toda vez que (i) el estudio de novedad y nivel inventivo en todas las Oficinas de Patentes del mundo es realizado por personas con conocimientos técnicos en el área de interés que son consideradas personas idóneas para emitir conceptos sobre la patentabilidad de una invención y que (ii) los resultados de los exámenes de novedad o de patentabilidad efectuados respecto a solicitudes extranjeras pueden ser reconocidos como suficientes para acreditar el cumplimiento de las condiciones de patentabilidad de la invención, tal como se señala en el artículo 46 de la Decisión 486:

"Artículo 46.- La oficina nacional competente podrá requerir el informe de expertos o de organismos científicos o tecnológicos que se consideren idóneos, para que emitan

opinión sobre la patentabilidad de la invención. Asimismo, cuando lo estime conveniente, podrá requerir informes de otras oficinas de propiedad industrial.

De ser necesario, a efectos del examen de patentabilidad y a requerimiento de la oficina nacional competente, el solicitante proporcionará, en un plazo que no excederá de 3 meses, uno o más de los siguientes documentos relativos a una o más de las solicitudes extranjeras referidas total o parcialmente a la misma invención que se examina:

a) copia de la solicitud extranjera;

b) copia de los resultados de exámenes de novedad o de patentabilidad efectuados respecto a esa solicitud extranjera;

(...)

*La oficina nacional competente podrá **reconocer los resultados de los exámenes referidos en el literal b) como suficientes para acreditar el cumplimiento de las condiciones de patentabilidad de la invención***” (Negrilla fuera del texto).

VI. PETICIÓN

Por todo lo anterior, solicito a ese Despacho lo siguiente:

7.1 Que al considerar las razones precedentes se revoque la decisión contenida en la Resolución No. **72862** del 1 de Diciembre de 2014, en cuanto resuelve negar el privilegio de patente de invención.

7.2. Que se otorgue el privilegio de patente de invención para **“PROCESO PARA PRODUCIR UNA FORMULACIÓN PESTICIDA EN SUSPENSIÓN DE UN HIDRATO DE SULFONILUERA”** a favor de **SUMITOMO CHEMICAL COMPANY, LIMITED.**

VII. NOMBRE Y DIRECCIÓN

El nombre y la dirección del solicitante recurrente son los siguientes:

SUMITOMO CHEMICAL COMPANY, LIMITED.

27-1, Shinkawa 2-chome, Chou-ku, Tokio, 104-8260

Tokio,

Japón

VIII. ANEXOS

- Datos experimentales adicionales.
- Patente No. US8907085 B2.

IX. NOTIFICACIONES

Atentamente manifiesto a usted que recibiré notificaciones en la Secretaría de su Despacho o en mi oficina de abogada situada en la Carrera 4ª. No. 72-35, piso 4º, de esta ciudad de Bogotá, D.C.

Señor Superintendente,


LUZ CLEMENCIA DE PAEZ

C.C. 35.456.344 de Usaquén

T.P.A. No. 23.555

COLO-17818-0801-002-3
CPT\CPT\ID-276893\WPC17818
No. M-2015-276893\CPT

PRUEBAS EXPERIMENTALES ADICIONALES

Método

Los procedimientos 1) a 6) fueron llevados a cabo y se obtuvieron los valores de (a) hasta (e), para así calcular la relación de depósito y la proporción residual de concentrados en suspensión de acuerdo con las siguientes ecuaciones A) y B):

- 1) Un peso de un tubo de centrifugación de vidrio de 50 ml es medido. (a)
- 2) Cerca de 40g de un concentrado en suspensión es pesado y cargado con precisión en el tubo. (b)
- 3) El extremo abierto del tubo se invierte verticalmente para descargar el concentrado en suspensión hasta que ninguna gota caiga dentro de los siguientes 10 segundos. Luego, el tubo se pesa. (c)
- 4) El concentrado en suspensión descargado, se recarga en el tubo, y luego el tubo se pesa. (d)
- 5) El tubo es centrifugado a 800 rpm durante 180 o 360 min, y la cantidad de sobrenadante (longitud de sobrenadante/longitud total) se mide.
- 6) El concentrado en suspensión se descarga de una manera similar a la descrita en el punto 3), y luego el tubo se pesa. (e)
- 7) El estado separado después de la centrifugación y el estado precipitado (por ejemplo, la presencia o ausencia de apelmazamiento, dureza, espesor) son observados después de la descarga.

A) Relación de Depósito (Antes de la centrifugación, %) = {(c)-(a)}/(b) x 100

B) Relación Residual (%) = {(e) - (a)} / {(d) - (a)} x 100

Resultados

Los resultados se presentan en la siguiente tabla A:

Tamaño medio de partícula		3.6	3.2	2.8	2.2	1.8
Relación Residual *1)	0 min ^{*2)}	1.6%	1.2%	1.2%	1.5%	1.8%
	180 min ^{*2)}	2.8%	2.4%	2.1%	1.9%	2.0%
	360 min ^{*2)}	3.2%	2.8%	2.3%	1.9%	1.9%

*1): Cantidad residual de concentrado en suspensión (que se adjunta a la superficie interior) en el tubo

2): Tiempo de centrifugación (minutos) a 800 rpm

De los resultados mostrados en la Tabla A, es evidente que cuanto mayor sea el tamaño de partícula, mayor es la cantidad residual de concentrado en la suspensión. Así que, la fluidez de concentrado en suspensión se disminuye y por lo tanto el concentrado en suspensión tiende a mantenerse en un tubo. Por lo tanto, se hace difícil mantener un buen estado de dispersión en el concentrado de suspensión.

CPT\CPTID-276892\WPC17818



(12) **United States Patent**
Yanagisawa et al.

(10) **Patent No.: US 8,907,085 B2**
(45) **Date of Patent: Dec. 9, 2014**

- (54) **HYDRATE OF SULFONYLUREA COMPOUND, PROCESS FOR PRODUCING THE SAME AND SUSPENSION FORMULATION CONTAINING THE SAME**
- (75) Inventors: **Yu Yanagisawa**, Osaka (JP); **Daisaku Kamo**, Takarazuka (JP)
- (73) Assignee: **Sumitomo Chemical Company, Limited**, Tokyo (JP)
- (*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 0 days.
- (21) Appl. No.: **13/334,278**
- (22) Filed: **Dec. 22, 2011**
- (65) **Prior Publication Data**
US 2012/0165196 A1 Jun. 28, 2012
- (30) **Foreign Application Priority Data**
Dec. 27, 2010 (JP) 2010-289619
- (51) **Int. Cl.**
C07D 487/04 (2006.01)
- (52) **U.S. Cl.**
USPC **544/236**
- (58) **Field of Classification Search**
USPC 544/236; 514/252.05
See application file for complete search history.

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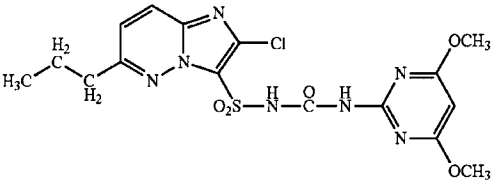
Primary Examiner — Paul V. Ward

(74) Attorney, Agent, or Firm — Birch Stewart Kolasch & Birch, LLP

(57) **ABSTRACT**

The present invention relates to a hydrate of sulfonylurea compound represented by formula (I):

(I)



having herbicidal activity, a process for producing the same, a suspension formulation containing the same, and the like.

4 Claims, No Drawings

1

HYDRATE OF SULFONYLUREA COMPOUND, PROCESS FOR PRODUCING THE SAME AND SUSPENSION FORMULATION CONTAINING THE SAME

FIELD OF THE INVENTION

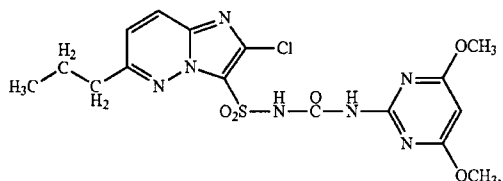
The present application is filed claiming the priority of the Japanese Patent Application No. 2010-289619 (filed on Dec. 27, 2010), the entire contents of which are herein incorporated by the reference.

The present invention relates to a hydrate of sulfonylurea compound having herbicidal activity, a process for producing the same, a suspension formulation containing the same, and the like.

BACKGROUND OF THE INVENTION

Recently, there is a need for a formulation that allows a herbicidal compound to be sprayed in a more simple way in order to control weeds because of the aging or decreasing population of farmers, and the like. Regarding a solid and poorly water-soluble herbicidal compound, for example, suspension formulations containing said compound, which can be sprayed directly from levee into paddy field under flood irrigation, have been developed for practical use.

EP 1466527 A1 discloses a sulfonylurea compound represented by formula (I):



which has herbicidal activity, and a suspension formulation containing said compound.

SUMMARY OF THE INVENTION

In suspension formulations containing a herbicidal compound, the herbicidal compound is generally suspended in water. Such suspension formulations can be produced by, generally, mixing a finely-ground, solid and poorly water-soluble herbicidal compound, a thickening agent, a surfactant and water, and, if necessary, an agricultural formulation additive.

In view of formulation stability such as suspension stability, herbicidal activity, spraying operability, and the like, it is advantageous that solid particles comprising a herbicidal compound in a suspension formulation have a small particle size (about 10 μm or less) immediately after the production of the suspension formulation as well as when using the suspension formulation after storage.

In suspension formulations containing a sulfonylurea compound represented by formula (I), solid particles comprising the sulfonylurea compound generally tend to grow (i.e., the particle size thereof tends to increase) in the suspension formulations over time during the storage period. If suspension formulations after storage are applied, there are sometimes problems that (i) the nozzle of sprayer used is clogged up by the solid particles, (ii) the herbicidal activity is not sufficiently obtained, and (iii) a large amount of precipitate is formed after long storage.

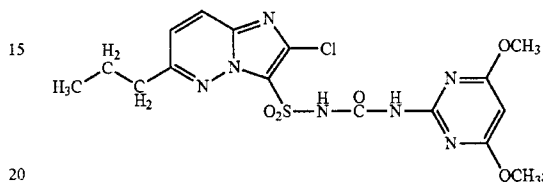
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In these circumstances, the present inventors have conducted intensive studies and finally found a novel form of the sulfonylurea compound, which can make solid particles comprising the sulfonylurea compound difficult to grow in the suspension formulation, thereby reaching the present invention.

Namely, the present invention provides the followings:

1. A hydrate (hereinafter sometimes referred to as "the present inventive hydrate") of a sulfonylurea compound represented by formula (I) (hereinafter sometimes referred to as "the present compound"):

(I)



2. The hydrate according to the above item 1, wherein the hydrate is a hemihydrate (hereinafter sometimes referred to as "the present inventive hemihydrate");
3. A suspension formulation (hereinafter sometimes referred to as "the present inventive suspension formulation") obtainable by mixing the hydrate according to the above item 1 or 2, a thickening agent, a surfactant and water;
4. A suspension formulation obtainable by mixing the hydrate according to the above item 1 or 2, a thickening agent, a surfactant, an agricultural formulation additive and water.

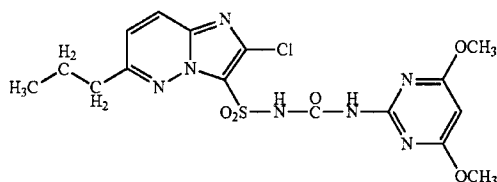
EFFECT OF THE INVENTION

According to the present invention, a novel form of the sulfonylurea compound represented by formula (I) (i.e., the present compound), which can make solid particles comprising the present compound difficult to grow in a suspension formulation containing the present compound, and a suspension formulation having superior stability such that the growth of solid particles comprising the present compound (i.e., the increasing of the particle size) is hardly occurred in the suspension formulation over time during the storage period, can be provided.

DETAILED DESCRIPTION OF THE EMBODIMENTS

The present inventive hydrate refers to a hydrate of a sulfonylurea compound represented by formula (I) (i.e., the present compound):

(I)



Preferred examples of the hydrate include a hemihydrate (i.e., $\frac{1}{2}$ hydrate).

The present inventive hydrate may be in a mixture with a nonhydrate of the sulfonylurea compound represented by formula (I) (i.e., the present compound). Preferred examples

of the mixture include those containing the present inventive hydrate (preferably, consisting essentially only of the present inventive hemihydrate) in an amount of not less than 0.2% by weight based on the total amount of the mixture, and more preferred are those containing the present inventive hydrate (preferably, consisting essentially only of the present inventive hemihydrate) in an amount of not less than 0.8% by weight based on the total amount of the mixture. The upper limit of the proportion of the present inventive hydrate is not particularly limited, but may be not more than 100% by weight based on the total amount of the mixture.

In addition, the present inventive hydrate present in a mixture with a nonhydrate of the present compound includes crystals comprising a nonhydrate of the present compound and the present inventive hydrate (preferably, the present inventive hemihydrate).

The present inventive hydrate can be produced by mixing a nonhydrate of sulfonylurea compound represented by formula (I) (i.e., the present compound) and water, and then stirring the resultant mixture (hereinafter sometimes referred to as "the present inventive production process No. 1").

In addition, the present inventive hydrate can be produced by dissolving a nonhydrate of sulfonylurea compound represented by formula (I) (i.e., the present compound) into a water-containing organic solvent, and then recrystallizing the mixture to obtain crystals (hereinafter sometimes referred to as "the present inventive production process No. 2").

In the present inventive production process No. 1, the present inventive hydrate can be obtained, for example, by (a) mixing a nonhydrate of the present compound with water, and then stirring the resultant mixture, and (b) subjecting the mixture to recrystallization, and then collecting the precipitated crystals by filtration and drying the collected residue under reduced pressure.

The temperature in the stirring of the "mixture of a nonhydrate of the present compound and water" (stirring temperature) in the present inventive production process No. 1 is, for example, from 5° C. to 95° C., preferably from 20° C. to 60° C.

The time in the stirring of the "mixture of a nonhydrate of the present compound and water" (stirring time) in the present inventive production process No. 1 is, for example, 7 days or more. Such process is suitable, for example, when producing the present inventive hydrate (preferably, the present inventive hemihydrate) in a mixture with a nonhydrate of the present compound.

In addition, a production process wherein the stirring time is set for not less than 14 days is suitable, for example, when producing the present inventive hydrate consisting essentially only of the present inventive hemihydrate, but not a mixture with a nonhydrate of the present compound.

The amount of "water" to be used in the present inventive production process No. 1 is preferably an amount not causing any trouble in the stirring of the mixture, and is, for example, from 10 mL to 100 mL, more preferably from 10 mL to 60 mL, relative to 1 g of a nonhydrate of the present compound.

In the present inventive production process No. 1, the present inventive hydrate can be produced in a shorter time by adding the present inventive hydrate produced separately as a seed crystal to a mixture of a nonhydrate of the present compound with water, and then stirring the mixture. The amount of the "hydrate of the present invention produced separately" to be added as a seed crystal is preferably not less than 8 parts by weight relative to 92 parts by weight of a nonhydrate of the present compound.

In the present inventive production process No. 2, the present inventive hydrate can be obtained, for example, by (a)

mixing a nonhydrate of the present compound with a water-containing organic solvent, and then stirring the resultant mixture to dissolve the nonhydrate into the organic solvent, (b) ice-cooling the resultant solution, and (c) subjecting the solution to recrystallization, and then collecting the precipitated crystals by filtration and drying the collected crystals under reduced pressure.

Preferably, the production process No. 2 may comprise (a) mixing a nonhydrate of the present compound with a water-containing organic solvent, and then stirring the resultant mixture with warming to dissolve the nonhydrate into the organic solvent and allowing the resultant solution to cool at room temperature, (b) ice-cooling the cooled solution, and (c) subjecting the solution to recrystallization, and then collecting the precipitated crystals by filtration and drying the collected crystals under reduced pressure.

In the step (a), after mixing a nonhydrate of the present compound with a water-containing organic solvent, the temperature of "stirring the resultant mixture to dissolve the nonhydrate into the organic solvent" (stirring temperature) is, for example, from 60° C. to 80° C.

In the step (a), after mixing a nonhydrate of the present compound with a water-containing organic solvent, the time of "stirring the resultant mixture to dissolve the nonhydrate into the organic solvent" (stirring time) is, for example, from 1 to 2 hours.

The "organic solvent" to be used in the present inventive production process No. 2 includes, for example, hydrophilic organic solvents, and the like. Preferred examples thereof include tetrahydrofuran (THF), acetonitrile, methanol, ethanol, dimethylsulfoxide, t-butanol, isopropyl alcohol, and the like, and more preferred examples include tetrahydrofuran (THF), acetonitrile, and the like.

The "water-containing organic solvent" to be used in the present inventive production process No. 2 includes, for example, those having a water content of not less than 1 part by weight relative to 99 parts by weight of the organic solvent. Specific examples thereof include water-containing tetrahydrofuran having a water content of about 1% by weight, water-containing acetonitrile having a water content of about 15% by weight, and the like.

The amount of "water-containing organic solvent" to be used in the present inventive production process No. 2 may be, for example, an amount capable of dissolving the present compound and precipitating the crystals by recrystallization. Specifically, when tetrahydrofuran or acetonitrile is used as the organic solvent, the amount is, for example, from 10 mL to 25 mL relative to 1 g of a nonhydrate of the present compound.

In the present inventive production process No. 2, the present inventive hydrate can be produced in a shorter time by adding the present inventive hydrate produced separately as a seed crystal to a mixture of a nonhydrate of the present compound with a water-containing organic solvent, stirring the mixture to dissolve the nonhydrate into the organic solvent, allowing the solution to cool at room temperature, and then ice-cooling the cooled solution. The preferred amount of the "hydrate of the present invention produced separately" to be added as a seed crystal is, for example, not less than 8 parts by weight relative to 92 parts by weight of a nonhydrate of the present compound.

In the production of the present inventive hydrate, the conversion from a nonhydrate of the present compound to the present inventive hydrate (including the present inventive hydrate present in a mixture with a nonhydrate of the present compound) may be monitored by various analytical methods

such as infrared absorption spectrum analysis, water content measurement, thermal analysis, X-ray powder diffraction analysis, and the like.

Specifically, for example, when the "increase in size of the peak corresponding to water molecule" or "shift in the peak" in a test sample is observed by infrared absorption spectrum analysis or the "increase in water content" in a test sample is observed by water content analysis, with reference to a nonhydrate of the present compound as a control sample, it is judged that a nonhydrate of the present compound has been converted to the present inventive hydrate (including the present inventive hydrate present in a mixture with a nonhydrate of the present compound).

As to thermal analysis, when an endotherm peak is detected in the vicinity of 136° C. and the decrease in weight is measured, for example, by thermogravimetric analysis (TG)/differential thermal analysis (DTA), it is judged that a nonhydrate of the present compound has been converted to the present inventive hydrate (including the present inventive hydrate present in a mixture with a nonhydrate of the present compound).

When a peak such as $2\theta=9.6^\circ, 11.0^\circ$ is detected by X-ray powder diffraction (Cu-K α) analysis, it is judged that a nonhydrate of the present compound has been converted to the present inventive hydrate (including the present inventive hydrate present in a mixture with a nonhydrate of the present compound).

The anhydrate of the sulfonylurea compound represented by formula (1) to be used for producing the present inventive hydrate is produced, for example, by a method as described in paragraphs [0173] and [0174] of EP 1466527 A1 (compound No. 36).

Hereinafter, the present inventive suspension formulation will be explained.

The present inventive suspension formulation can be obtained by mixing the present inventive hydrate, a thickening agent, a surfactant and water, and, if necessary, an agricultural formulation additive. Namely, the present inventive suspension formulation is a herbicidal composition obtained by dispersing or dissolving the present inventive hydrate, a thickening agent and a surfactant, and, if necessary, an agricultural formulation additive into water.

In the suspension formulation, solid particles comprising the present compound are dispersed in water in the form of fine particles. The average particle size of the fine particles is, for example, not more than 10 μm , preferably 0.2 μm to 5 μm .

When the present inventive hydrate is present in a mixture with a nonhydrate of the present compound, the amount of the present inventive hydrate in the mixture is, for example, not less than 0.2% by weight and less than 100% by weight relative to the amount of the nonhydrate of the present compound. Namely, the proportion (weight ratio) of a nonhydrate of the present compound to the present inventive hydrate is, for example, from 99.8:0.2 to 0:100, preferably from 99.2:0.8 to 0:100.

The content of the present inventive hydrate (including the present inventive hydrate present in a mixture with a nonhydrate of the present compound) in the present inventive suspension formulation is, for example, from 0.5% by weight to 50% by weight, preferably from 1% by weight to 40% by weight.

The content of thickening agent in the present inventive suspension formulation is, for example, from 0.01% by weight to 5% by weight, preferably from 0.1% by weight to 3% by weight.

The content of surfactant in the present inventive suspension formulation is, for example, from 0.1% by weight to 10% by weight.

The water content in the present inventive suspension formulation is, for example, from 30% by weight to 90% by weight, preferably from 50% by weight to 80% by weight.

The present inventive suspension formulation may contain other pesticidal compounds, in addition to a pesticidal compound included in the present inventive hydrate (including the present inventive hydrate present in a mixture with a nonhydrate of the present compound). Examples of the "other pesticidal compound" include simetryn, dymron, propanil, mefenaset, phentolazamide, ethobenzanide, swep, oxadiclefone, oxadiazolone, pyrazolate, prodiamine, cafenstrole, pentoxazone, clomeprop, pyriphthalide, benzobicyclon, bromobutide, pyraclostrobin, imazosulfuron, sulfosulfuron, and the like.

When the "other pesticidal compound" is contained in the present inventive suspension formulation, the amount of all the pesticidal compounds in the present inventive suspension formulation [i.e., the total amount of a pesticidal compound included in the present inventive hydrate (including the present inventive hydrate present in a mixture with a nonhydrate of the present compound) and the "other pesticidal compound"] is, for example, 0.5% by weight to 50% by weight, preferably 1% by weight to 40% by weight.

Examples of the thickening agent to be used in the present inventive suspension formulation include xanthan gum, guar gum, gum arabic, casein, dextrin, carboxymethyl cellulose or a salt thereof (for example, sodium salt, calcium salt, etc.), sodium carboxymethylstarch, sodium alginate, hydroxyethyl cellulose, carboxyethyl cellulose, methyl cellulose, hydroxypropyl cellulose, hydroxyethyl cellulose, polyvinyl alcohol, polyacrylic acid or a derivative thereof, montmorillonite, talc, saponite, and the like.

In the present invention, carboxymethyl cellulose is generally used in the form of a salt. The preferred carboxymethyl cellulose salt to be used in the present inventive suspension formulation includes, for example, those having a relatively low viscosity when being dissolved in water, specifically a viscosity of from 10 mPa·s to 100 mPa·s (type B viscometer, 60 rpm, 25° C.) in 2% by weight aqueous solution.

Typical examples of carboxymethyl cellulose salt include those commercially available, such as CELLOGEN 6A (manufactured by Dai-ichi Kogyo Seiyaku Co., Ltd.), CELLOGEN 7A (manufactured by Dai-ichi Kogyo Seiyaku Co., Ltd.), CMC DAICEL 1110 (manufactured by Daicel Chemical Industry, Ltd.) and CMC DAICEL 1210 (manufactured by Daicel Chemical Industry, Ltd.).

Examples of the surfactant to be used in the present inventive suspension formulation include anionic surfactants such as polyoxyethylene arylphenyl ether phosphoric acid salts (e.g., NEWKALGEN FS-3EG, NEWKALGEN FS-3PG, manufactured by Takemoto Oil & Fat Co., Ltd.), alkylsulfuric acid salts (e.g., MONOGENY-500, manufactured by Dai-ichi Kogyo Seiyaku Co., Ltd.), polyoxyethylene arylphenyl ether sulfuric acid salts (e.g., AGRISOL FL-2017, manufactured by Kao Corporation), polyoxyalkylene arylphenyl ether sulfuric acid salts (e.g., NEWKALGEN FS-7, manufactured by Takemoto Oil & Fat Co., Ltd.), dioctylsulfosuccinic acid salts (e.g., NEOCOL YSK: manufactured by Dai-ichi Kogyo Seiyaku Co., Ltd., SANMORIN OT70: manufactured by Sanyo Chemical Industries, Ltd.), and the like; and nonionic surfactants such as sucrose fatty acid esters (e.g., NEWKALGEN FS-100, manufactured by Takemoto Oil & Fat Co., Ltd.), polyoxyethylene polyoxypropylene block polymers (e.g., NEWPOL PE68: manufactured by Sanyo Chemical Indus-

tries, Ltd.), polyoxyalkylene polyalkylene polyamine (e.g., NEWKALGEN D-3020, manufactured by Takemoto Oil & Fat Co., Ltd.), polyoxyethylene alkylphenyl ether (e.g., NEWKALGEN D-410, manufactured by Takemoto Oil & Fat Co., Ltd.), polyoxyethylene aryl phenyl ether formaldehyde condensates (e.g., NEWKALGEN E-300, manufactured by Takemoto Oil & Fat Co., Ltd.), and the like.

The present inventive suspension formulation may contain two or more surfactants as mentioned above. In this case, it is preferable to use a combination of at least one anionic surfactant and at least one nonionic surfactant.

The water to be used in the present inventive suspension formulation may be those used for conventional herbicidal composition, for example, tap water, well water, ion-exchanged water, and the like.

The agricultural formulation additive to be used in the present inventive suspension formulation includes anti-freezing agents, pH adjusters, antifoaming agents, preservatives, and the like.

Examples of the "anti-freezing agent" include ethylene glycol, diethylene glycol, glycerin, propylene glycol, and the like.

Examples of the "pH adjuster" include citric acid monohydrate, sorbic acid, potassium sorbate, and the like.

Examples of the "antifoaming agent" include silicone-based antifoaming agents, and the like.

Examples of the "preservative" include butylparaben (n-butyl para-hydroxybenzoate), sorbic acid, potassium sorbate, and the like.

The content of the agricultural formulation additive in the present inventive suspension formulation may vary depending on the amount of the present inventive hydrate (including the present inventive hydrate present in a mixture with a nonhydrate of the present compound) to be used, the kind of the surfactant, and the like.

The content of the anti-freezing agent in the present inventive suspension formulation is, for example, 1% by weight to 20% by weight, preferably 3% by weight to 12% by weight.

The content of the pH adjuster in the present inventive suspension formulation is, for example, 0.01% by weight to 5% by weight, preferably 0.5% by weight to 3% by weight.

The content of the antifoaming agent in the present inventive suspension formulation is, for example, 0.05% by weight to 0.5% by weight, preferably 0.05% by weight to 0.3% by weight.

The content of the preservative in the present inventive suspension formulation is, for example, 0.01% by weight to 3% by weight, preferably 0.01% by weight to 1.5% by weight.

The present inventive suspension formulation may be formulated, for example, by the following processes.

<Formulation Process No. 1>

A process comprising mixing the present inventive hydrate (including the present inventive hydrate present in a mixture with a nonhydrate of the present compound), thickening agent, a surfactant and water, and optionally the "other pesticidal compound" and, if necessary, an agricultural formulation additive, and then thoroughly stirring and mixing the mixture, for example, by a high speed stirrer, and finely-grinding and dispersing the mixture, for example, by a wet-pulverizer such as dynamill and micro-fluidizer.

<Formulation Process No. 2>

A process comprising finely-grinding the present inventive hydrate (including the present inventive hydrate present in a mixture with a nonhydrate of the present compound) by a dry-pulverizer such as jetmizer, adding the hydrate together with other components to water, and then stirring and mixing

the mixture, for example, by a high speed stirrer for about 30 to 90 minutes to disperse the mixture.

The present inventive suspension formulation can be used by spraying itself or, if desired, after dilution with water, according to known methods. For example, the present inventive suspension formulation can be sprayed directly from levee into paddy field under flood irrigation and the like. In this case, a vessel containing the present inventive suspension formulation is shaken slightly before use, and then the formulation is sprayed in portions along levee. When the present inventive suspension formulation is sprayed after dilution with water, the water dilution is sprayed by using known spraying devices on the surface of soil, stem or leaves, or the like in paddy field, dry field, orchard, turf, non-cultivated field, and the like. In addition, the water dilution can be used in a seed treatment, nursery box treatment, and the like.

EXAMPLES

The present invention is further described below in detail with reference to examples. The present invention is not limited to the examples.

For water content analysis, CA-07 type water-content measuring instrument (manufactured by Mitsubishi Chemical Corporation) was used.

For infrared absorption spectrum analysis, Fourier transform infrared spectrophotometer, Varian Fast Image 670-IR (manufactured by Varian Inc.) was used. The measurement conditions were as follows:

Detector: DTGS

Scans: 32

Scan speed: 5 kHz

For thermal analysis, TG/DTA6200R (manufactured by SII NanoTechnology Inc.) was used.

For X-ray powder diffraction (Cu-K α) analysis, powder X-ray diffractometer RINT2500V (manufactured by Rigaku Corporation) was used. The measurement conditions were as follows:

Target: Cu- α

Voltage: 40 kV

Current: 300 mA

For proton nuclear magnetic resonance spectrum analysis, DPX300 (manufactured by Bruker Corporation) was used. Tetramethylsilane was used as an internal standard substance, and all delta values were represented as ppm.

Production Example 1

To 217.8 g of the sulfonylurea compound represented by formula (I) (i.e., a nonhydrate of the present compound, hereinafter sometimes referred to as "sample A") produced by a method described in paragraphs [0173] and [0174] of EP 1466527 A1) (Compound No. 36) was added 2413 mL of tetrahydrofuran (THF) having a water content of 1.0% by weight. The resultant mixture was stirred at 70°C. for 1 hour to dissolve the sample A (i.e., a nonhydrate of the present compound) into the above THF. The resultant solution was allowed to cool to room temperature, and then the cooled solution was ice-cooled. After ice-cooling, the crystals precipitated by recrystallization were collected by filtration, and the collected crystals were dried under reduced pressure to obtain 121.8 g of dried crystals (i.e., the present inventive hemihydrate, hereinafter sometimes referred to as "sample B").

As test samples, the sample A and the sample B were subjected to proton nuclear magnetic resonance spectrum analysis (referred to as "H-NMR" in Table 1), infrared

absorption spectrum analysis (referred to as "IR" in Table 1), water content measurement (referred to as "water content" in Table 1), X-ray powder diffraction (Cu-K α) analysis (referred to as "XRD(2 θ)" in Table 1), and thermal analysis (referred to as "TG/DTA" in Table 1). As a result, some characteristic values were obtained (see Table 1).

The proton nuclear magnetic resonance spectrum of the sample A was identical with that of the sample B. On the other hand, peak corresponding to water molecule in infrared absorption spectrum and water content measured by water content analysis were different between the sample A and the sample B.

The sample A and the sample B had different characteristics in terms of crystal diffraction pattern by X-ray powder diffraction (Cu-K α) analysis.

Further, the sample A and the sample B were different in endotherm peaks observed by thermal analysis. Specifically, the sample B had an endotherm peak at 136° C., in addition to an endotherm peak at 208° C. as observed in the sample A. TG was decreased by 1.9%. Said value was identical with the theoretical water content, 1.9%, when the present compound is a hemihydrate. Therefore, it was confirmed that the sample A was in the form of a nonhydrate, and the sample B was in the form of a hemihydrate.

TABLE 1

	Sample A	Sample B
¹ H-NMR (DMSO-d ₆ , δ)	0.70 (3H, t, J = 7.3 Hz), 1.4-1.5 (2H, m), 2.6-2.7 (2H, m), 3.97 (6H, s), 6.08 (1H, s), 7.57 (1H, d, J = 9.4 Hz), 8.26 (1H, d, J = 9.4 Hz), 10.68 (1H, brs), 13.4-13.5 (1H, m)	0.70 (3H, t, J = 7.3 Hz), 1.4-1.5 (2H, m), 2.6-2.7 (2H, m), 3.97 (6H, s), 6.08 (1H, s), 7.57 (1H, d, J = 9.4 Hz), 8.26 (1H, d, J = 9.4 Hz), 10.68 (1H, brs), 13.4-13.5 (1H, m)
IR	3649 cm ⁻¹	3638 cm ⁻¹
Water content	0.06%	1.90%
XRD(2 θ)	6.2 8.6 9.1 13.7 15.4	9.6 11.0
TG/DTA	208° C. (endotherm)	136° C. (endotherm, decrease of TG: 1.9%) 208° C. (endotherm)

Production Example 2

Firstly, 852 mg of the sample A (i.e., a nonhydrate of the present compound) was mixed with 50 mL of a deionized water, and then the resultant mixture was started to stir by a stirrer at room temperature. Two (2) days, 7 days and 14 days after the start of stirring, each 10-mL portion of the resultant mixture was collected. The collected mixtures were independently subjected to filtration, collected the precipitated crystals obtained by recrystallization, and then dried the collected crystals under reduced pressure to obtain dried crystals. These crystals were classified into three types depending on the stirring time, i.e., 107 mg of dried crystals (hereinafter sometimes referred to as "sample C") for the portion collected after 2 days, 143 mg of dried crystals (hereinafter sometimes referred to as "sample D") for the portion collected after 7 days, and 144 mg of dried crystals (hereinafter sometimes referred to as "sample E") for the portion collected after 14 days.

Each of the thus obtained samples was subjected to X-ray powder diffraction (Cu-K α) analysis. As a result, it was con-

firmed that (i) the sample C was a nonhydrate, (ii) the sample D was the present inventive hemihydrate present in a mixture of a nonhydrate (a mixture of a hemihydrate and a nonhydrate, containing 9.1% by weight of the hemihydrate), and (iii) the sample E was the present inventive hemihydrate (the present inventive hydrate consisting essentially only of the present inventive hemihydrate).

Some of the present inventive hemihydrates in a mixture with a nonhydrate, which are different from the sample D as described in the above (ii), are shown in Table 2 along with the "sample symbol" and the "content (% by weight) of hemihydrate in the mixture".

TABLE 2

Sample symbol	Content (% by weight) of hemihydrate in the mixture
Sample G	7.9
Sample H	5.7
Sample I	5.6
Sample J	0.9
Sample K	0.5
Sample L	0.3
Sample M	0.2

Production Example 3

A mixture obtained by mixing 1.8 parts by weight of the sample B (i.e., the present inventive hemihydrate) as produced in Production Example 1, 0.1 parts by weight of sorbic acid, 0.3 parts by weight of a silicone-based antifoaming agent (Antifoam E-20, manufactured by Kao Corporation), 0.5 parts by weight of sucrose fatty acid ester (NEWKALGEN FS-100, manufactured by Takemoto Oil & Fat Co., Ltd.), 1.5 parts by weight of polyoxyethylenearyl phenyl ether phosphoric acid salt (NEW KALGEN FS-3PG, manufactured by Takemoto Oil & Fat Co., Ltd.), 0.35 parts by weight of sodium montmorillonite (KUNIPIA F, manufactured by Kuminine Industries Co., Ltd.), and 30.45 parts by weight of ion-exchanged water, and they were mixed and dispersed, then, wet-pulverized using Dynomill KDL (manufactured by Shinmaru Enterprises Corporation) to obtain an aqueous suspension (1) of the sample B.

On the other hand, 1.0 part by weight of sodium carboxymethylcellulose (CELLOGEN 7A, manufactured by Dai-ichi Kogyo Seiyaku Co., Ltd.), 0.65 parts by weight of sodium montmorillonite (KUNIPIA F, manufactured by Kuminine Industries Co., Ltd.), and 0.1 parts by weight of xanthan gum (Rhodopol 23, manufactured by Rhodia Nicca) were added to 56.65 parts by weight of ion-exchanged water, dissolved and dispersed to obtain a solution (1) of thickening agent. Then, 58.4 parts by weight of the thus obtained solution (1) of thickening agent, 35 parts by weight of the aqueous suspension (1) of the sample B as obtained above, and 6.6 parts by weight of propylene glycol were mixed to become 100 parts by weight in total, and then stirred and mixed the resultant mixture to obtain a suspension formulation of the present invention (hereinafter sometimes referred to as "suspension formulation (1)") containing the sample B (i.e., the present inventive hemihydrate) in an amount of 1.8% by weight.

Production Example 4

A suspension formulation of the present invention (hereinafter sometimes referred to as "suspension formulation (2)") was produced in the same manner as Production Example 3, except for using the sample G (the present inven-

11

tive hemihydrate present in a mixture with a nonhydrate as shown in Table 2) instead of the sample B (i.e., the present inventive hemihydrate).

Production Example 5

A suspension formulation of the present invention (hereinafter sometimes referred to as “suspension formulation (3)”) was produced in the same manner as Production Example 3, except for using the sample H (the present inventive hemihydrate present in a mixture with a nonhydrate as shown in Table 2) instead of the sample B (i.e., the present inventive hemihydrate).

Production Example 6

A suspension formulation of the present invention (hereinafter sometimes referred to as “suspension formulation (4)”) was produced in the same manner as Production Example 3, except for using the sample I (the present inventive hemihydrate present in a mixture with a nonhydrate as shown in Table 2) instead of the sample B (i.e., the present inventive hemihydrate).

Production Example 7

A suspension formulation of the present invention (hereinafter sometimes referred to as “suspension formulation (5)”) was produced in the same manner as Production Example 3, except for using the sample J (the present inventive hemihydrate present in a mixture with a nonhydrate as shown in Table 2) instead of the sample B (i.e., the present inventive hemihydrate).

Production Example 8

A suspension formulation of the present invention (hereinafter sometimes referred to as “suspension formulation (6)”) was produced in the same manner as Production Example 3, except for using the sample K (the present inventive hemihydrate present in a mixture with a nonhydrate as shown in Table 2) instead of the sample B (i.e., the present inventive hemihydrate).

Production Example 9

A suspension formulation of the present invention (hereinafter sometimes referred to as “suspension formulation (7)”) was produced in the same manner as Production Example 3, except for using the sample L (the present inventive hemihydrate present in a mixture with a nonhydrate as shown in Table 2) instead of the sample B (i.e., the present inventive hemihydrate).

Production Example 10

A suspension formulation of the present invention (hereinafter sometimes referred to as “suspension formulation (8)”) was produced in the same manner as Production Example 3, except for using the sample M (the present inventive hemihydrate present in a mixture with a nonhydrate as shown in Table 2) instead of the sample B (i.e., the present inventive hemihydrate).

Comparative Production Example 1

A comparative suspension formulation (hereinafter sometimes referred to as “suspension formulation (9)”) was pro-

12

duced in the same manner as Production Example 3, except for using the sample A (i.e., a nonhydrate of the present compound) instead of the sample B (i.e., the present inventive hemihydrate).

Test Example 1

Particle Size Measurement

For the suspension formulations (1) to (9), the particle size (volume median diameter (μm)) of solid particles comprising the present compound in each suspension formulation was measured by using a laser diffraction type particle size distribution measurement apparatus (HEROS & RODOS, manufactured by Japan Laser Corp., measurement conditions: focus length 20 mm, dispersing medium is ion-exchanged water) (i) immediately after the production, and (ii) after storage in a certain condition. The results are shown in Tables 3 and 4.

TABLE 3

Suspension formulation	(1)	(2)	(3)	(4)	(5)
Immediately after production	2.0	1.9	2.0	1.9	1.8
50° C., 1 week	1.9	2.2	2.5	2.4	2.6
50° C., 2 weeks	1.9	2.2	2.5	2.4	2.6
60° C., 1 week	2.0	2.2	2.5	2.5	2.7
60° C., 2 weeks	2.0	2.3	2.5	2.5	2.7

TABLE 4

Suspension formulation	(6)	(7)	(8)	(9)
Immediately after production	2.0	1.8	1.8	2.0
50° C., 1 week	2.9	2.9	2.8	2.8
50° C., 2 weeks	2.9	2.9	2.8	13.5
60° C., 1 week	3.2	3.0	3.0	11.4
60° C., 2 weeks	3.2	3.0	3.0	11.3

INDUSTRIAL APPLICABILITY

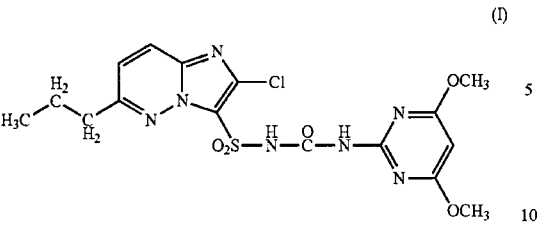
According to the present invention, a novel form of the sulfonylurea compound represented by formula (I) (i.e., the present compound), which can make solid particles comprising the present compound difficult to grow in a suspension formulation containing the present compound, and a suspension formulation having superior stability such that the growth of solid particles comprising the present compound (i.e., the increasing of the particle size) is hardly occurred in the suspension formulation over time during the storage period, can be provided.

What is claimed is:

1. A process for producing a suspension formulation, which comprises mixing a hemihydrate of a sulfonylurea compound represented by formula (I):

13

14



a thickening agent, a surfactant and water.

2. A suspension formation obtained by mixing the hemi-
hydrate according to claim 1, the thickening agent, the sur-
factant and water.

3. A suspension formulation obtained by mixing the hemi-
hydrate according to claim 1, the thickening agent, the sur-
factant, an agricultural formulation additive and water.

4. The process according to claim 1, which comprises
mixing the hemihydrate of the sulfonylurea compound rep-
resented by formula (I), the thickening agent, the surfactant,
an agricultural formulation additive and water.

* * * * *