

ADDITIONAL TESTS

Hereinbelow, the amorphous of Compound (1) explained in Reference Example 1 of the present application and the crystal of the present invention are each respectively abbreviated to "A-Amor." and "A-Crys.".

[Additional Test 1: Production of Flowable Agent Containing A-Amor.]

1-1. Production of Slurry Containing Grinded A-Amor.

1 pts. mass of sodium salt of methylnaphthalene sulfonic formaldehyde polycondensate (Tradename: Supragil MNS/90, available from Solvay), 0.5 pts. mass of a silicone type defoaming agent (Tradename: KM-73, available from Shin-Etsu Chemical Co., Ltd.), 0.25 pts. mass of citric acid (available from Showa Kako Corp.) and 7 pts. mass of propylene glycol (available from ADEKA Corporation) are dispersed in 35.96 pts. mass of water. Further, 5.19 pts. mass of A-Amor. (Purity: 96.4%) is dispersed in the obtained dispersion, and is subjected to wet grinding by a sand grinder (available from Imex Co., Ltd.) with Φ 0.8 mm to Φ 1.2 mm of glass beads, thus obtaining 50 pts. mass of grinded slurry.

1-2. Production of Dispersion Medium

3.0 pts. mass of carboxymethyl-cellulose sodium (Tradename: Cellogen 5A, available from DKS Co. Ltd.), 1.5 pts. mass of refined bentonite (Tradename: KunipiaF, available from Kunimine Industries Co., Ltd.) and 0.1 pts. mass of 1,2-benzisothiazolin-3-one (Tradename: Proxel GXL(S), available from Lonza Japan) are dispersed in 45.4 pts. mass of water, thereby obtaining 50 pts. mass of dispersion medium.

1-3. Production of Flowable Agent Containing A-Amor.

The 50 pts. mass of grinded slurry obtained in Additional Test 1-1 and the 50 pts. mass of dispersion medium obtained in Additional Test 1-2 are mixed, thereby obtaining 100 pts. mass of a flowable agent containing A-Amor.

[Additional Test 2: Production of Flowable Agent Containing A-Crys.]

2-1. Production of Slurry Containing Grinded A-Crys.

The same production method used in Additional Test 1-1 is applied, except: water is changed to 36.04 pts. mass; and A-Amor. is changed to 5.11 pts. mass of A-Crys. (Purity: 97.9%). Through this production method, 50 pts. mass of grinded slurry is obtained.

2-2. Production of Flowable Agent Containing A-Crys.

The 50 pts. mass of grinded slurry obtained in Additional Test 2-1 and the 50 pts. mass of dispersion medium obtained in Additional Test 1-2 are mixed, thereby obtaining 100 pts. mass of a flowable agent containing A-Crys.

[Additional Test 3: Confirming Crystallinity of Flowable Agent Containing A-Amor.]

Setting crystallinity of A-Crys. in a flowable agent obtained in Additional Test 2 at 100%, crystallinity of A-Amor. in the flowable agent obtained in Additional Test 1 is measured according to the measuring conditions and measuring methods outlined below.

- Measuring Conditions:

- X-ray Diffractometers: X' Pert PRO MPD, available from PANalytical
- Measuring Principle: Capillary Transmission Method
- X-ray Source: Cu K-alpha ray
- Measuring Range: 2-30° (2θ)

- Measuring Methods:

The A-Amor. used in Additional Test 1 is directly filled in a glass capillary (Φ 0.7 mm). A diffraction spectrum for the sample is then measured according to the above measuring conditions. A-Crys. used in Additional Test 2, the flowable agent obtained in Additional Test 1 and the flowable agent obtained in Additional Test 2 are measured by the same methods.

FIG. 1 indicates each chart of diffraction patterns that have been obtained. As to the 4 diffraction peaks derived from Compound (1) in the obtained diffraction patterns, considering each area value of the flowable agent containing A-Amor. and the flowable agent containing A-Crys., the crystallinity (%) of the flowable agent comprising A-Amor. is calculated according to Equation (1) below. Each result is shown in Table 1.

EQUATION (1)

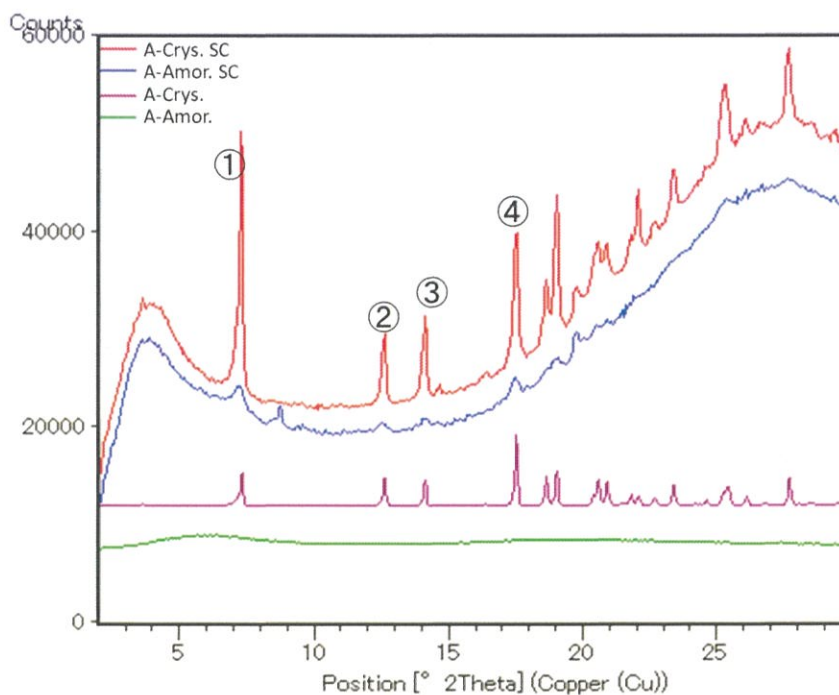
Crystallinity (%) of Flowable agent A-Amor. = Area value of Flowable agent containing A-Amor.
/ Area value of Flowable agent containing A-Crys. $\times$ 100

[Table 1]

Peak No.	Position [$^{\circ}$ 2theta]	Area Value of Flowable Agent containing A-Amor.	Area Value of Flowable Agent containing A-Crys.	Crystallinity [%] of Flowable Agent containing A-Amor.
①	7.3	670	4600	15
②	12.6	210	1430	15
③	14.1	260	1660	16
④	17.5	540	2320	23

Based on the mean of the crystallinity values represented by the 4 peaks in Table 1, it is confirmed that “17%” of A-Amor. in the flowable agent obtained in Additional Test 1 has been crystallized.

[FIG. 1]



[Additional Test 4: Herbicidal Effect Test to Upland Weeds By Foliar Application]

Sterilized diluvial soil is put in a plastic pot having a dimension of 9 cm in length, 14 cm in width and 8 cm in depth. Seeds of common ragweed and velvetleaf are each planted in the diluvial soil in separate spots. After covering the seeds with soil up to a height of 1.5 cm above

the soil surface, plants have been grown in a greenhouse having a temperature of 20°C to 30°C. After the plants have been growing for 15 days, each flowable agent obtained in Additional Test 1 and Additional Test 2 is diluted so as to become a predetermined dosage. Then, as an adjuvant, any one of 0.05% (Volume ratio) of Tween20 (Tradename), 0.5% (Volume ratio) of Actirob B (Tradename) and 0.1 % (Volume ratio) of Tris(2-ethylhexyl) phosphate (TEHP) is added and broadcasted to the stem and leaves with a small sprayer (Agent treatment). 30 days after the agent treatment is applied, the herbicidal effect on each plant is visually observed and evaluated along an index of 0 (No Effect) to 100 (Completely Killed). Those results are shown in both Table 2 and Table 3.

TABLE 2

	Compound (1)	Common Ragweed	
Adjuvant	g/ha	Crystal	Amorphous
TEHP 0.1%	12.5	60	60
	25	90	75
	50	93	100

TABLE 3

	Compound (1)	Velvetleaf	
Adjuvant	g/ha	Crystal	Amorphous
Tween20 0.05%	12.5	80	65
	25	95	75
	50	95	90
Actirob B 0.5%	12.5	80	60
	25	85	80
	50	95	85

Based on Table 2 and Table 3, compared with the amorphous of Compound (1) explained in Reference Example 1 of the present application, it is determined that the crystal of the present invention shows effective herbicidal activities more than twice as high as those of the amorphous of Compound (1) ^{Note 1}.

Note 1: The index level being able to confirm effective herbicidal activities is 80 or more.