

Title Method of preparing granular decabromodiphenyl ethane or decabromodiphenyl ether

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Priority

Abstract

This invention relates to a method for preparing granular 1,2-bis(pentabromophenyl) ethane or decabromodiphenyl ether, by weight percent, the greater than or equal to 800 meshes 88%-96%'s 1,2-bis(pentabromophenyl) ethane or decabromodiphenyl ether; 2-7%'s α crystal form nucleating agent; 0.5-3% dispersant; 0.1%-1.5%'s coupling agent of organic titanate and 0.1-2%'s antioxidant are placed in high speed agitator in performing high speed stirring to 70°C-110°C, when stirring to dust free, pouring the mixed material then further adding to extruder for fusing, plasticizing, extruding, then pelletizing or pelleting, and making into with low dosage, an universal type, the material physical chemistry performance influence minor granular

1,2-bis(pentabromophenyl) ethane or decabromodiphenyl oxide, and can reduce dust pollution, improve labour environment, and can be used as general purpose plastics and engineering plastics.

What is claimed is:

1. one method for preparing granular 1,2-bis(pentabromophenyl) ethane or decabromodiphenyl ether, which is characterized in that: (weight percent) the greater than or equal to 800 meshes 88%~96%'s 1,2-bis(pentabromophenyl) ethane or decabromodiphenyl ether; 2 ~7%'s α crystal form nucleating agent; 0.5 ~3% dispersant; 0.1%~1.5%'s coupling agent of organic titanate and 0.1 ~2%'s antioxidant are placed in high speed agitator in performing high speed stirring to 70°C ~110°C, when stirring to dust free, pouring the mixed material then further adding to extruder for fusing, plasticization, extruding, then pelletizing or pelleting.

2, according to claim 1 said 1,2-bis(pentabromophenyl) ethane or decabromodiphenyl ether granular preparation method, which is characterized in that: said dispersants include polyethylene wax or/and diffusion oil.

3, according to claim 1 said granular 1,2-bis(pentabromophenyl) ethane or decabromodiphenyl ether's preparing method, which is characterized in that: said extruder's length-diameter ratio is 20 ~56.

4, according to claim 2 said granular 1,2-bis(pentabromophenyl) ethane

or decabromodiphenyl ether's preparing method, which is characterized in that: the extruder material cylinder six zone controlled temperature at 100 °C ~180°C therebetween.

5, according to claim 1 said granular 1,2-bis(pentabromophenyl) ethane or decabromodiphenyl ether's preparing method, which is characterized in that: the cotruder material cylinder six zone controlled temperature at 120 °C ~170°C.

Specification

Method for preparing granular 1,2-bis(pentabromophenyl) ethane or decabromodiphenyl ether

Technical field

This invention relates to a method for preparing 1,2-bis(pentabromophenyl) ethane or decabromodiphenyl oxide powder into the form of particle .

Background

At present, with building plastic material and common civil with plastic's mass application, which plastic material in flame retarding property requirements are also higher and higher, in order to adapt national economy development needs, more and more plastic are required to achieve definite flame retarding grade. The plastic requirements additive besides liquid or warm molten, the required additive material grain size very fine, to obtain relatively good product performance. As fire-barrier

material preferably commonly-used flame retarding synergist-1,2-bis(pentabromophenyl) ethane or decabromodiphenyl ether fineness generally at above 800 meshes, if 1,2-bis(pentabromophenyl) ethane or decabromodiphenyl ether grade the higher, its grain size also the smaller, hence the very fine 1,2-bis(pentabromophenyl) ethane or decabromodiphenyl oxide powder adding to the resin base body material performing mixing, pelletizing process, can produce large quantity of dust pollution and noise, work environment relatively adverse. Adding 1,2-bis(pentabromophenyl) ethane or decabromodiphenyl ether is heavy metal oxide, it also can do harm to human body 's health.

As everyone knows, 1,2-bis(pentabromophenyl) ethane or decabromodiphenyl ether is inorganic material, only adding above 20% high molecular polymer carrier, be able to obtain granular flame retarding mother material. But manufactured flame retarding mother material due to reducing 1,2-bis(pentabromophenyl) ethane or decabromodiphenyl ether content, to make fire-barrier material's flame retarding property reduced. And the flame retarding mother material also contain such as polyolefin (PO), polystyrene (PS), ABS resin carrier, but due to differential resins with different compatibility, hence concrete carrier resinous species is determined by fire-barrier material, otherwise can only form mechanical blending material, resulting in the fire-barrier material physical chemistry performance greatly reduced. This also makes

obtained granular 1,2-bis(pentabromophenyl) ethane or decabromodiphenyl ether only can be prepared into proprietary flame retarding mother material, it only is directed towards a certain base body material speciality, so that has the disadvantages of scope of application is narrow, large dosage. While conventional plastics mother material in inorganic material content is no less than 80%, generally there is no sufficient content resin acting as mother material carrier, hence in 1,2-bis(pentabromophenyl) ethane or decabromodiphenyl ether content over 80%, generally it is impossible to obtain high-performance, low cost plastics mother material, which is also powder-like 1,2-bis(pentabromophenyl) ethane or decabromodiphenyl ether cannot by additive make into granule, to eliminate dust pollution reasons.

Contents of the invention

The purpose of the invention is producing one 1,2-bis(pentabromophenyl) ethane or decabromodiphenyl ether content is higher than 85%, with low dosage, an universal type, minor influence on base body material physical chemistry characteristics granular 1,2-bis(pentabromophenyl) ethane or decabromodiphenyl ether preparing method.

The invention to achieve said purpose technical scheme is: one method for preparing granular 1,2-bis(pentabromophenyl) ethane or decabromodiphenyl ether, which is characterized in that: (weight percent) the greater than or equal to 800 meshes 88%~96%'s

1,2-bis(pentabromophenyl) ethane or decabromodiphenyl ether; 2 ~7%'s α crystal form nucleating agent; 0.5 ~3% dispersant; 0.1%~1.5%'s coupling agent of organic titanate and 0.1 ~2%'s antioxidant placed in high speed agitator in performing high speed stirring to 70°C ~110°C, when stirring to dust free, pouring the mixed material then further adding to extruder for fusing, plasticization, extruding, then pelletizing or pelleting.

Said dispersants include polyethylene wax or/and diffusion oil.

Said screw extruder's length-diameter ratio is 20 ~56.

The screw extruder material cylinder six zone controlled temperature at 100°C ~180°C therebetween.

The cotruder material cylinder six zone controlled temperature at 120°C ~170°C.

The invention adopting said technical scheme has the following advantages:

1. the invention flame retarding masterbatch 1,2-bis(pentabromophenyl) ethane or decabromodiphenyl ether's inorganic material content is greater than or equal to 90%'s, by adding low melting point dispersants and coupling agent of organic titanate, in high speed agitator high temperature in the mixing process, promote inorganics material curing, simultaneously low melting point compatibilizer and coupling agent of organic titanate molten to make inorganics material therebetween,

inorganic material and additive therebetween produce definite adhesive function, then by extruder heating and screw cutting, mixing and compressing action, to make the above adhesive function further enhance, to make inorganics material to obtain large viscosity, may be achieved by die extruding pelleting or drawn wire to make into granular flame retarding mother material. Thus breaking the conventional in inorganic material content is no less than 80%, high load inorganic material due to carrier proportion of resin present deficiency while cannot perform adherent problem, and the problem of long time polymeric material industries high load inorganic material with a little carrier performing blending, cannot obtain high-performance, low cost mother material problems mentioned above.

2 , the invention powder 1,2-bis(pentabromophenyl) ethane or decabromodiphenyl ether and a low-melting dispersant and coupling agent of organic titanate mixing and fusing, after by screw machine and extruding out pelletizing or pelleting to make into granules, directly acting as granular main fire retarding agent, in adding in the resin base body material, by extruder extruding is performed, as granular 1,2-bis(pentabromophenyl) ethane or decabromodiphenyl ether as adhesion compound molten, hence in fire-barrier material still has original powder-like 1,2-bis(pentabromophenyl) ethane or decabromodiphenyl ether effect, by mixing action, can make resin base

body material to form good dispersity. Thereby greatly reducing dust pollution, improve labour environment, which has significant social effect on other inorganic material making into granular inorganic material.

3, the invention powder 1,2-bis(pentabromophenyl) ethane or decabromodiphenyl oxide by low melting point dispersant and coupling agent of organic titanate mixing and fusing, because without resin carrier, hence not exist with fire-barrier material therebetween produce eutectic problem, so that it can be used in multiple general purpose plastics and engineering plastics, scope of application is extensive. Besides the 1,2-bis(pentabromophenyl) ethane or decabromodiphenyl oxide content of over 85%, to make flame retarding system effective flame retarding elementary bromine relatively high content, so that in low dosage precondition, fire-barrier material can be up to relatively high flame retarding property.

4, the inventive producing mechanical is impeller and screw extruder, which extruder can adopt common cotruder, single-screw extruder, reciprocating screw extruding machine and roller type extruder etc., and producing mechanical ordinary, easy to obtain, technical process of production is simple, labor intensity is lower, with relatively strong operability.

5, the invention adopts the α crystal form nucleating agent, that is in molten material is added grain of crystallization, raise material

crystallizing temperature, and since there exists the crystal nucleus, makes rate of crystallization and degree of crystallinity greatly raised, and is refined material base body crystal grain, to make material base body with processing cooling pre-high temperature stage is due to the nucleating agent to make material starting crystallizing, to make material base body during the cooling stage crystallization more completely, which can effectively avoid the material post-moulding occur recrystallization, to make product size stability increasing, and the said process can ensure material size stability, dimensional accuracy and suffaceness. In addition, by adding α crystal form nucleating agent, material base body crystallizing time mostly generate fine crystal grain, avoiding large-grained appears, which has positive effect on improve material creep resistance, shock strength and other some mechanical property, the material physical chemistry performance influence is small. Besides the invention added dispersant and coupling agent of organic titanate, not only in the preparation process produce bonding action, and when mixed with material base body, also can play a dispersion and surface modification action, hence generally has little effect on material physical chemistry characteristics.

Detailed description of the preferred embodiment

By the following given inventive concrete embodiment may further clearly understand the invention.

Example 1

The greater than 800 purpose 88%'s 1,2-bis(pentabromophenyl) ethane, 7%'s α crystal form nucleating agent: 0.5% diffusion oil and 2% polyethylene wax built dispersant, 1%'s coupling agent of organic titanate and 1.5%'s antioxidant placed in homogenizer high speed stirring to $75^{\circ}\text{C} \pm 2^{\circ}\text{C}$, approximately 25 min, when stirring to dust free, pouring the mixed material adding to length-diameter ratio is 44 cotruder, cotruder material cylinder six zone controlled temperature are respectively region I 128°C , II zone 134°C , III region 146°C , IV zone 158°C , V region 168°C , VI zone 170°C , material in cotruder material cylinder fusing, plasticization, extruding, then flour-milling pelletizing or brace pelleting or cooling pelletizing, and making into granular 1,2-bis(pentabromophenyl) ethane flame retarding mother material.

Example 2

The 1250 purpose 90%'s 1,2-bis(pentabromophenyl) ethane, 5%'s α crystal form nucleating agent; 2.5% polyethylene wax and 1.5%'s coupling agent of organic titanate and 1%'s antioxidant placed in homogenizer high speed stirring until $82^{\circ}\text{C} \pm 2^{\circ}\text{C}$, approximately 25 min, when stirring to dust free, pouring the mixed material adding to length-diameter ratio is 56 single-screw extruder, single-screw extruder material cylinder six zone controlled temperature are respectively region I 130°C , II zone 135°C , III region 140°C , IV zone 150°C , V region 160°C ,

VI zone 170°C, material in single-screw extruder material cylinder fusing, plasticization, extruding, then flour-milling pelletizing or brace pelleting or cooling pelletizing, and making into a granular 1,2-bis(pentabromophenyl) ethane flame retarding mother material.

Example 3

The 1250 purpose 92%'s 1,2-bis(pentabromophenyl) ethane, 4%'s α crystal form nucleating agent; 1% diffusion oil and 1.5% polyethylene wax built dispersant, 1%'s coupling agent of organic titanate and 0.5%'s antioxidant placed in homogenizer to mix rapidly and $89^{\circ}\text{C} \pm 2^{\circ}\text{C}$, approximately 30 min, when stirring to dust free, pouring the mixed material adding to length-diameter ratio is 20 reciprocating screw extruding machine, reciprocating screw extruding machine charging spout six zone controlled temperature are respectively region I 122°C, II zone 128°C, III region 136°C, IV zone 146°C, V region 154°C, VI zone 160°C, material in reciprocating screw extruding machine charging spout fusing, plasticization, extruding, then flour-milling pelletizing or brace pelleting or cooling pelletizing, and making into a granular 1,2-bis(pentabromophenyl) ethane flame retarding mother material.

Example 4

At 800 purpose 94%'s 1,2-bis(pentabromophenyl) ethane, 3%'s α crystal form nucleating agent; 0.5% diffusion oil and 1.5%'s coupling agent of organic titanate and 1%'s antioxidant placed in homogenizer high

speed stirring to $95^{\circ}\text{C} \pm 2^{\circ}\text{C}$, approximately 30 min, when stirring to dust free, pouring the mixed material adding to length-diameter ratio is 36 cotruder, extruder material cylinder six zone controlled temperature are respectively region I $110^{\circ}\text{C} \pm 2^{\circ}\text{C}$, II region 115°C , III region 122°C , IV zone 130°C , V region 142°C , VI zone 154°C , material in cotruder material cylinder molten, plasticization, extruding, then flour-milling pelletizing or brace pelleting or cooling pelletizing, and making into a granular 1,2-bis(pentabromophenyl) ethane flame retarding mother material.

Example 5

The 1250 purpose 96%'s 1,2-bis(pentabromophenyl) ethane, 2%'s α crystal form nucleating agent; 0.8 diffusion oil and 1% polyethylene wax built dispersant, 0.1%'s coupling agent of organic titanate and 0.1%'s antioxidant placed in homogenizer to mix rapidly $102^{\circ}\text{C} \pm 2^{\circ}\text{C}$, approximately 35 min, when stirring to dust free, pouring the mixed material adding to length-diameter ratio is 32 cotruder, cotruder material cylinder six zone controlled temperature are respectively region I 100°C , II zone 110°C , III region 115°C , IV zone 125°C , V region 135°C , VI zone 150°C , and the mixed material in cotruder material cylinder molten, plasticization, extruding, then flour-milling pelletizing or brace pelleting or cooling pelletizing, and making into a granular 1,2-bis(pentabromophenyl) ethane flame retarding mother material.

Decabromodiphenyl ether can according to the above embodiment replacement 1,2-bis(pentabromophenyl) ethane can be made into granular decabromodiphenyl ether's flame retarding mother material, but not limited more embodiment.

The invention to obtain granular 1,2-bis(pentabromophenyl) ethane or changing decabromodiphenyl oxide, and granular antimony trioxide mixed formula see table 1, table 2, table 3 is flame retarding ABS and flame retarding HIPS material behavior to comparative sheet.

Table 1 test materials formula (unit: phr)

	Resin material	antimony trioxide	decabromodiphenyl ether	1,2-bis(pentabromophenyl) ethane
Formula 1	ABS 83.3	4.2	12.5	
Formula 2	ABS 83.3	4.2		12.5
Formula 3	HIPS 83.3	4.2	12.5	
Formula 4	HIPS 83.3	4.2		12.5

Table 2 flame retarding ABS performance comparative sheet

items	unit	standard	common flame retarding ABS	formula 1	formula 2
tensile strength	MPa	GB/T1040	42.5	42	42.1
elongation at break	%	GB/T1040	20	25	26
simple beam impact strength non-notch	KJ/m2	GB/T1043	58.4	83.5	63.5
simple beam impact strength notch	KJ/m2	GB/T1043	7.9	8.8	11.4
cantilever impact strength non-notch	KJ/m2	GB/T1843	8.3	7.5	9.8
average shrinkage rate	%	HG-112	0.71	0.58	0.58
combustion performance	UL94	ANSI/UL94	V-0	V-0	V-0

Table 3 flame retarding HIPS performance comparative sheet

items	unit	standard	common flame retarding ABS	formula 3	formula 4
tensile strength	MPa	GB/T1040	18.7	22.7	22.6
elongation at break	%	GB/T1040	42	45	46
simple beam impact strength non-notch	KJ/m2	GB/T1043	59.9	63.7	71.4
simple beam impact strength notch	KJ/m2	GB/T1043	8.3	8.8	9.8
cantilever impact strength non-notch	KJ/m2	GB/T1843	7.5	7.5	8.3
average shrinkage rate	%	HG-112	0.77	0.55	0.55
combustion performance	UL94	ANSI/UL94	V-0	V-0	V-0

By said Table 2 and Table 3 contrast shows that, the invention-mentioned granular 1,2-bis(pentabromophenyl) ethane or decabromodiphenyl ether with different resin base body mixing, its flame retarding property are can achieve V-0 grade, tensile strength, Cantilever impact strength non-notch with common fire-barrier material and compared are unbiased, its elongation at break of raising, reduces average shrinkage rate, and can properly increase fire-barrier material ground mechanical property.