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CA 2143626 C 2004/12/28

(11)(21) 2 143 626

(12) BREVET CANADIEN
CANADIAN PATENT

(13) C

(86) Date de dépôt PCT/PCT Filing Date: 1993/08/31
(87) Date publication PCT/PCT Publication Date: 1994/03/17
(45) Date de délivrance/Issue Date: 2004/12/28
(85) Entrée phase nationale/National Entry: 1995/02/28
(86) N° demande PCT/PCT Application No.: US 1993/008004
(87) N° publication PCT/PCT Publication No.: 1994/005432
(30) Priorité/Priority: 1992/09/01 (P 42 29088.0) DE

(51) Cl.Int.^{6}/Int.Cl.^{6} C08J 3/205

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(54) Titre : GRANULES EN THERMOPLASTIQUE CONTENANT UN LUBRIFIANT; METHODE DE FABRICATION ET UTILISATIONS DE CES GRANULES

(54) Title: LUBRICANT-CONTAINING PELLETS OF THERMOPLASTICS PROCESS FOR PREPARING SAME AND USE THEREOF

(57) Abrégé/Abstract:
The present invention relates to lubricant-containing pellets of thermoplastic resins, which pellets are characterized in that the lubricant is present thereon in the form of a film firmly adhering to the surface of the pellets, and to a process for their production and their use in injection molding machines.

PCT WORLD INTELLECTUAL PROPERTY ORGANIZATION
International Bureau

INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification 5 : **B05D 1/28** 2143626 **A1**

(11) International Publication Number: **WO 94/05432**

(43) International Publication Date: 17 March 1994 (17.03.94)

(21) International Application Number: PCT/US93/08004

(22) International Filing Date: 31 August 1993 (31.08.93)

(30) Priority data:
P 42 29 088.0 1 September 1992 (01.09.92) DE

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(81) Designated States: CA, JP, US, European patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).

Published
With international search report.

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(57) Abstract

The present invention relates to lubricant-containing pellets of thermoplastic resins, which pellets are characterized in that the lubricant is present thereon in the form of a film firmly adhering to the surface of the pellets, and to a process for their production and their use in injection molding machines.

TITLE

LUBRICANT-CONTAINING PELLETS OF THERMOPLASTICS PROCESS FOR PREPARING SAME AND USE THEREOF

The present invention relates to lubricant-containing pellets of thermoplastics, and especially those pellets which contain the lubricant in the form of a film firmly adhering to the surface of the pellets.

The invention further relates to processes for the production of these lubricant-containing pellets of thermoplastics and to the use thereof, more particularly for injection molding.

Articles made of thermoplastic materials are conventionally produced from pellets of the thermoplastic resins by injection molding. Auxiliary agents (lubricants) are added to the pellets to achieve trouble-free processing. These agents control the rheology of the melt, and often they involve further positive effects for processing and/or for the optimization of the final product.

Small injection molding machines with short dwell times are especially susceptible to malfunctions occurring in the screw region where the polymer begins to melt. It has been shown that these malfunctions can be eliminated, if suitable lubricants are present in sufficient amounts on the pellets. This cannot be accomplished by a lubricant incorporated in the thermoplastic resin; the lubricant must rather be present on the outer surface of the pellets in order to be effective on the machine walls.

The lubricants generally used for pellets of thermoplastic resins are products of fat chemistry, mostly those of stearin, olein, glycerol; waxes such as montan wax and polyolefin wax; and metal stearates such as aluminum stearate, calcium stearate, zinc stearate and magnesium stearate.

An amount of from 0.01 to 0.5% by weight of said metal stearates in the form of a finely divided powder is usually added to the pellets of thermoplastics and is admixed in continuous mixers or double-cone batch blenders, wherein the metal stearate powder becomes attached to the surface of the pellets. However, the adhesion to the pellets of the metal stearates powder is insufficient so that upon shipment and, more particularly upon the use of pneumatic (pressurized air) conveying systems, a separation of the metal stearate powder occurs. Due to this peel-off, not only the effective lubricant content on the pellet surface is reduced, resulting in a loss in functionality of the pellet-lubricant adduct, but also the released powder

causes process-technological drawbacks such as soiling the shipment containers and plugging the filters of conveyor units. In addition, the deposition on the floors of the production plant constitutes a particular source of danger, since areas covered with metal stearates are extremely slippery.

Therefore, the desire has existed for a long time to provide pellets of thermoplastics with a firmly adhering film that has the same lubricant properties as the pellets provided with powdery metal stearates without the disadvantage of the powder coming off.

The manufacture of such film-coated pellets of thermoplastic resins is relatively simply to accomplish for most of the organic materials by way of an application of the film material in the molten state, for example by tumbler coating. Molten solutions of metal stearates can also be applied to pellets to form lubricant coatings (cf. European Patent Specification 0 280 221 B1), however, such coatings are sometimes found to be irregular or uneven, or do not adhere as tightly to the pellets as the lubricant of this invention.

Another approach for firmly linking metal stearate lubricants to the pellets consists of including the lubricants in the pellets. However, thereupon it was found that, mainly in small injection molding machines with short dwell times, the pellets including the lubricant resulted in malfunctions of the feeding process. Thus, a trouble-free functionality of the lubricant-pellet adduct is ensured only if the lubricant will become effective prior to the pellet being melted, thus, if the lubricant is available on the surfaces of the pellets.

Therefore, it was the object of the present invention to provide pellets of thermoplastic resins, to which pellets the lubricant has been firmly bonded, while the action of the lubricant is not adversely affected thereby.

Surprisingly it was found that it is possible to provide lubricant-containing-pellets of thermoplastic resins wherein the lubricant is present on the surface of the pellets in the form of a firmly adhering lubricant film using a coating process as defined hereinafter. Moreover it was found that a particularly good result is achieved where the lubricant is comprised predominantly (on a weight basis) of an aluminum salt of a fatty acid.

As used herein, the term "firmly adhering film" refers to a continuous film which is essentially free from gaps or holes. The firmly

adhering film of this invention is thereby distinguishable from lubricant coatings of the prior art, which tend to resemble powder coatings. This distinction is visible when viewing the coated pellets under a microscope. The firmly adhering film of the present invention will appear somewhat as a varnish on the pellet. It will not easily be visible apart from the fact that light reflects off it somewhat differently as compared to an uncoated pellet. Conversely, the coatings of the prior art will appear under the microscope as discontinuous coatings, *i.e.*, as dusts or very finely divided powders.

The term "is comprised predominantly (on a weight basis) of an aluminum salt of a fatty acid" means that where the lubricant contains more than one component, an aluminum salt of a fatty acid is the component present in the greatest amount on a weight basis.

Preferred according to the present invention are pellets having a lubricant content of from 0.01 to 0.5% by weight, and especially from 0.05 to 0.15% by weight.

The lubricants of the lubricant-containing pellets of thermoplastic resins according to the invention consist preferably of from 75 to 100% by weight of an aluminum salt of a fatty acid and of from 25 to 0% by weight of fat chemistry products, especially fatty acids and fatty alcohols; waxes, especially montan wax and polyolefin wax; and esters and/or metal salts (apart from aluminum) of fatty acids, especially sodium-, magnesium-, calcium- and zinc-stearate, stearyl stearate, and the lithium, odium, and calcium salts of montanic acid. Where the lubricants (other than the metal salts of fatty acids) are in the form of a powder, the powder is preferably milled to a particle size of less than about 1 micron.

According to present invention preferred is a lubricant comprising 75 to 100% by weight, and especially from 90 to 100%, of an aluminum stearate, and especially aluminum distearate.

According to the present invention, the thermoplastic resin may be any melt-processable solid thermoplastic material. The preferred thermoplastic resins include polyesters, polyacetals, polyacrylates, polyamide and/or copolymers or mixtures containing said thermoplastic resins, among which polyamides and copolymers containing polyamides are more preferred. The polyamides of the present invention have been selected from Nylon 6, Nylon 6/6, Nylon 6/9, Nylon 6/10, Nylon 6/12, Nylon 11, Nylon 12

and Nylon 12/12, among which copolymers containing polyamides of adipic acid, hexamethylene diamine and/or Caprolactam are preferred.

According to the present invention the lubricant-containing pellets of thermoplastic resins may contain up to 70% by weight of additives and filler materials, including stabilizers, and especially antioxidants and UV absorbents; plasticizers; fillers and minerals, especially talc, chalk, kaolin, wollastonite, slate meal, mica powder and baryte; pigments and dyes; reinforcing agents and impact modifiers, especially glass fibers; flame retardants and nucleating agents, especially salts, talc, HT-Nylon, fluorocarbon compounds.

The invention further relates to a process for the preparation of pellets of thermoplastic resins, which process is characterized in that

(A) the uncoated pellets of thermoplastic resins and the lubricant are combined and mixed at a temperature below the melting point of the lubricant; and

(B) the resulting pellet-lubricant adducts are then kept in motion so that they are subjected to a sufficient number of mutual impacts and shear forces whereby a firmly adhering lubricant film is formed on the pellets.

In a preferred embodiment of the process according to the invention, combining and mixing the pellets with the lubricant is carried out at a temperature of from 20°C to 50°C, and especially at a temperature of from 40°C to 50°C. Thereby it is ensured that by means of the process the pellets of thermoplastic resins can be coated without any thermal-oxidative damage (discoloration) in the presence of air.

According to the present invention, in a preferred process embodiment the lubricant is an aluminum salt of a fatty acid, preferably an aluminum stearate, more preferably aluminum distearate, and the pellets are of polyamide, and preferably are Nylon 6/6.

Any device in which the pellets are subjected to the above-described motions is suitable to be used in the process according to the invention. In a preferred embodiment of the process according to the invention the process is operated in a continuous mode (continuous process). It is particularly preferred that the process step (B) takes place in a dense phase conveying system as described in Example 2.

The present invention further relates to the use of the lubricant-containing pellets of thermoplastic resins for the manufacture of thermoplastic resin-based articles.

The lubricant-containing pellets of thermoplastic resins show the same functional properties as the pellets provided with powdery lubricants; however, they do not tend to have the lubricant coming loose therefrom. Moreover, the process according to the invention allows a lubricant film to be applied onto thermolabile pellets of thermoplastic resins, and especially onto pellets.

In another embodiment of the present invention, it is possible to provide lubricant containing pellets of thermoplastic resins wherein at least two lubricant layers are present. a first lubricant layer is present on the surface of the pellets in the form of a firmly adhering lubricant film using a coating process as defined herein above. At least one additional lubricant layer which is comprised of a lubricant having a melting point of 140°C or less is present on the surface of the first lubricant layer. Suitable lubricants for the additional lubricant layer(s) are known i the art and include, but are not limited to, montan wax, polyolefin wax, paraffin, N-stearyl eruceamide, and metal stearates. Such additional lubricant layer(s) can be applied by conventional techniques known in the art such as melt spray and tumbling coating techniques.

The present invention is further illustrated by the following Examples:

EXAMPLES

Example 1 (Coating Test)

A reactor comprising a 100 mm in diameter vessel capable of being rotated and performing gyroscopic motions is charged with 100g of Nylon 6/6 and 0.1 g of powdery lubricant; then, mixing is carried out at room temperature of 60 rpm.

The film forming property of various lubricants at a stirring period of 15 minutes is shown in Table I.

Table I

Example	Lubricant	Remarks
A	Aluminum Distearate	Film Formation
B	Aluminum Tristearate	Firm Formation
C	Stearol Stearate	Partial Film Formation

Example 2 (Continuous Coating)

For continuously coating polyamide pellets with aluminum distearate at a temperature of from 40°C to 50°C, the pellets of Nylon 6/6 as available after drying and having a regular oval-cylindrical shape are supplied to a continuous mixer, into which at the same time aluminum distearate is introduced in a controlled amount. In the mixer the stearate present as a fine powder is uniformly powdered onto the pellet surfaces.

Through an exit port in the bottom of the mixer the powder-coated pellets are passed through a commercially available dense phase conveying system, (sold, *e.g.*, by the companies WAESCHLE (D-7980 Ravensburg), GERICKE (D-7703 Rielasingen) and BUEHLER (CH-9240 Uzwil). The conveying system will typically be 200 to 350 m in length, preferably about 300 m. At the end of the conveying system the powdered layer, which initially is loosely adhered to the surface, becomes a firmly adhering uniform coating. The product is thereby essentially free from aluminum distearate dust.

Example 3 (Adhesion Test)

An amount of 2.5g of Nylon 6/6 pellets coated with 0.1% by weight of aluminum distearate (Example 2) and an amount of 2.5g of conventional Nylon 6/6 pellets containing 0.1% by weight of aluminum distearate (lubricant in powder form) each are washed in 40 ml of Freon® 113 for 10 seconds at a speed of the stirring rod of 88 rpm. After the washing liquid is poured off, the pellets are dried, and the aluminum distearate content is determined.

As a result of the determination, the Nylon 6/6 pellets prepared according to the conventional method (only passed through a continuous mixer) has a residual aluminum distearate content which was by 30% lower.

In the pellets coated by means of the process according to the present invention, 95% to 97% of the aluminum distearate added are present as a firmly adhered film.

Example 4 (Use Test)

Using a small molding machine (NETSTAL AG., Schweiz, Model N 350/150 S with a standard NETSTAL low compression (2.6) screw), the screw retraction time (SRC) is measured at a hold up time of three minutes for each of the Nylon 6/6 pellets treated with 0.1% by weight of aluminum distearate as specified in Table II. The results are also set forth in Table II.

Table II

Example	Sample	SRC (Seconds)
D	Melt-incorporated	10.4 to 20.2
E	Powder-coated	10.1 to 14.5
F	Film-coated/Example 2	9.0 to 10.6

CLAIMS

1. A process for the preparation of lubricant-containing pellets of thermoplastic resins, characterized in that:

(a) the uncoated pellets of thermoplastic resins and a lubricant comprised of a metal salt of a fatty acid are combined and mixed at a temperature from 20°C to 50°C;

(b) the resulting pellet-lubricant adducts are then kept in motion in a dense phase conveying system so that they are subjected to a sufficient number of mutual impacts and shear forces whereby a firmly adhering lubricant film is formed on the pellets.

2. The process according to Claims 1 wherein the process is a continuous process and the process step (B) is carried out in a dense phase conveying system.

3. The process according to Claim 1, characterized in that at least one additional lubricant layer is applied to the surface of the firmly adhering lubricant film.

4. Lubricant-containing pellets of thermoplastic resins obtained by a process according to any one of Claims 1-3.

5. Lubricant-containing pellets of thermoplastic resins prepared in accordance with the process of Claim 3, said lubricant-containing pellets being characterized in that they contain at least two lubricant layers in which

(a) the first lubricant layer is comprised predominately, on a weight basis, of an aluminum salt of a fatty acid and is present on the surface of the pellets in the form of a firmly adhering film; and

(b) the second lubricant layer is comprised of a lubricant having a melting point of 140°C or less and is present on the surface of the first lubricant layer.