The invention relates to a processing plant and a process for producing bales of amorphous and semi-crystalline polyolefins and to the bales so produced. The invention provides easily dispersible bales of pellets made from an amorphous and/or semicrystalline polyolefin.
For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

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— with international search report
PROCESS FOR PRODUCING BALES OF AMORPHOUS AND SEMI-
CRystALLINE POLYOLEFINs AND BALES PRODUCED THEREBY

[0001] This application claims the benefit of U.S. Provisional Application
No. 60/435,045, filed December 20, 2002, the entire disclosure of which is hereby
incorporated herein by reference.

FIELD OF INVENTION

[0002] The invention relates to a processing plant and a process for producing bales of amorphous and semi-crystalline polyolefins and to the bales so produced.

BACKGROUND OF INVENTION

[0003] Easily dispersible bales (EDB) are desirable in rubber processing to facilitate the mechanical break-up of the bales under conditions of shear for compounding, for example in Banbury mixers.

[0004] The ease with which the bales can be broken up and the bulk density is an important performance criteria. Bales having a lower bulk density are easier to break up, but bales having higher bulk density are desirable to reduce production, storage and transportation costs. The bulk density of a rubber bale is calculated by dividing the bale weight by its volume (W x L x H), and is typically expressed in units of gram/cubic centimeters (g/cm³). A typical rubber bale bulk density will vary from as low as 0.45 g/cm³ to as high as the specific gravity of the rubber itself. For an ethylene-propylene (and optionally diene) polymer EP(D)M, the specific gravity is 0.86-0.88 g/cm³ at room temperature.

[0005] The bulk density should be distinguished from the particulate (pellet or crumb) density, which is the density of the individual particles. The particulate density reflects the level of porosity. Both aforementioned densities should be further distinguished from the density of the polymer, which is measured after any porosity has been removed (e.g. by compressing the molten pellets or crumbs).
Conventional rubber finishing processes generally produce crumb, which is generally highly porous. Crumb is a particulate that is typically produced by exploding wet polymer resin through the die plate of dewatering or drying extruders or by flashing a rubber latex in spray drying equipment. These particulates contain many voids, porosities and fines, with very irregular shapes and surfaces, and therefore exhibit low particulate density, the term "particulate density" meaning the density including spaces and voids. The fine particulates have a major drawback in that they are extremely difficult to handle due to their stickiness and low bulk density.

Bales with a low bulk density around 0.38 - 0.72 g/cm³ have been produced and then stored within supported cartons to avoid densification, as described in U.S. Patent No. 3,775,933. During manufacture, the production rate of a readily dispersible, or friable bale may be enhanced by reducing the weight of the bale and increasing the frequency of the baling cycle. However, a specially designed supported bale unit (SBU) may be needed to package the bale and to store and to ship the friable bale to customers without densification to prevent undesired compression that may undermine the friability. The SBU can be expensive and brings additional storage and package disposal costs for the users. U.S. Patent No. 3,775,933 discloses the use of porous crumbs to make bales with a density of less than 0.72 g/cm³ and a high ethylene content of above 79 wt%.

Partitioning agent(s) such as those described in U.S. Patent No. 4,207,218 may be applied to the rubber particulates before baling, and may assist in establishing and preserving friability. The rubber particulates are crumb or powder. Goodrich produces friable bales from rubber crumbs. The rubber crumbs are admixed with a solid anti-cake agent, which is non-soluble in the rubber, and then formed into friable bales. Goodrich describes coating rubber particulates of low density and compressing them into a friable bale having a bulk density of about 1.3 to 2.0 times that of the original particulate rubber. The rubber particulates range from powder to crumb with sizes ranging from 0.1 millimeter (mm) to 15 mm in diameter. Due to the irregular shape and surface of the powder and crumb, a dusting level of from 0.5 parts to about 20 parts per 100 parts of rubber particulate (weight) is applied.
EP0050039 forms bales with a bulk density of 0.4 to 0.6 g/cm³ from porous crumbs. US 4,822,545 uses blends to form pellets with an outer skin of a plastic polymer to resist agglomeration and produce free flowing pellets. U.S. Patent Nos. 5,098,635, 5,559,178 and European Patent No. 427,339 describe applying a spray coating composition comprising an inorganic partitioning agent, a thickener and a binding agent, water and water soluble anionic dispersant onto rubber extrudate as the extrudate is cut into pellets exiting a de-watering extruder. The water evaporates upon extrusion and the particles are porous. The blade of the extruder is adjusted to produce porous, substantially flat pellets. The pellets are then dried and compressed into bales.

Oil-extension into the virgin rubber during the manufacturing step can also assist in improving bale friability. For example, Vistalon TM 5730 made and sold by ExxonMobil Chemical Company is produced with 30 parts per hundred of rubber (phr) oil-extension with a bulk density of 0.70g/cm³ to 0.85 g/cm³. Oil-extension of hard-to-mix rubber (semi-crystalline or very high molecular weight rubber) decreases the apparent viscosity of the rubber, and therefore renders the bale easier to be dispersed with carbon black and additional oil and compounding ingredients using internal mixers, such as a Banbury mixer. However, the use of oil-extension delivers less rubber to the customer, adds cost, and can reduce the torque and the carbon black incorporation rate in the mixing equipment, which results in a longer mixing time to reach good carbon black dispersion.

In recent years, an improved process has been developed in the rubber industry, in which the rubber is directly recovered from its reaction medium (solution, slurry, or latex) through a direct devolatilization process, such as described in WO02/34795 and WO98/02467, both incorporated fully herein by reference. An advantage of such process is that dense pellets, without appreciable levels of voids and porosities, can be produced prior to baling. Depending on the method of density measurement the voids may be included to various extents from the density measurement. By dense pellets it is generally meant that the rubber particles, after extrusion through a cylindrical die, have a density of at least 90%, or at least 95%, of the density of the polyolefin. In other words, the density of the
pellet including any voids, etc., approximates that of the polymer itself. The dense pellets may have a spherical, ellipsoidal or cylindrical shape. However, bales made from dense pellets of the semi-crystalline grades or the high Mooney viscosity grades resulting from this devolatilization process, without dusting, are not easily dispersed and are very difficult to break apart in a mixer.

[0012] Certain embodiments of the invention may provide one or more of the following advantages: (1) a finishing process for a polymerization plant that has the flexibility to produce free flowing pellets or form the amorphous or semi-crystalline polyolefin resin into a bale of agglomerated polyolefin pellets, with a higher bulk density than the particulate density of any free-flowing form of the pellets, and (2) a bale that is easily dispersible with preferably low levels of partitioning or dusting agent.

SUMMARY OF INVENTION

[0013] The invention is a process for producing bales of polyolefin comprising (a) in a devolatilizing step, removing solvent from a molten polymer, said molten polymer comprising solvent and polymer, (b) pelletizing said devolatilized polymer in a cooling medium to produce dense pellets, (c) removing substantially all of said cooling medium from the dense pellets, (d) applying a partitioning agent to the dense pellets, and (e) compressing said dense pellets in a bale mould for a sufficient time, temperature and pressure to obtain a bale of pellets, said bale having a density of 0.45 g/cm$^3$ to 0.85 g/cm$^3$.

[0014] The term "dense pellets" is used herein to contrast the particulates making up the bale, which are produced by the prior removal of volatiles in advance of pelletization on the one hand and which produce particulates having low levels of or no porosity, from the particulates produced in the form of porous crumb by the sudden vaporization coincident with extrusion as produced in many conventional processes for making EPDM, which produce particulates having a high porosity. The term "polyolefin" covers polymers produced in a single polymerization and blends, provided that the pellets remain compressible and capable of forming a stable compressed bale in which individual pellets are adhered to one another. The term "partitioning agent" is not used herein to
indicate that the pellets are successfully partitioned and remain free-flowing, but rather to indicate that the adhesion between the pellets is reduced so as to facilitate the breaking up or dispersion of the bale back into its constituent pellets in the course of subsequent mixing and compounding steps.

[0015] The process of the invention may particularly involve any one or more of the following (a) in a devolatilizing step, removing solvent from a molten amorphous or semicrystalline polymer comprising solvent and polymer, (b) pelletizing the devolatilized polymer in a cooling medium at a temperature of from about 4°C to about 60°C to produce dense pellets having a density of at least 95% of the density of the amorphous or semi-crystalline polyolefin, (c) removing substantially all of the cooling medium from the dense pellets, (d) applying a partitioning agent to the dense pellets, and (e) compressing the dense pellets in a bale mould for a sufficient time, temperature and pressure to obtain a bale of pellets, said bale having a bulk density of 0.66 g/cm³ to 0.85 g/cm³. The bulk density is below the particulate density of the constituent pellets as a result of the voids remaining between the pellets.

[0016] In another aspect, the invention provides a processing plant for producing bales from pellets of polyolefin comprising (a) a devolatilizer capable of removing solvent from molten polymer comprising solvent and polymer, (b) a pelletizer in fluid communication with the devolatilizer, said pelletizer capable of concentrating the devolatilized polymer into dense pellets in a cooling medium, (c) a dewatering device in fluid communication with the pelletizer, said dewatering device capable of removing at least a portion of the cooling medium from the dense pellets to provide dewatered pellets, (d) a drying device adapted to receive the dewatered pellets from the dewatering device, said drying device capable of conditioning said dewatered pellets by removing substantially all the cooling medium from the dewatered pellets and heating the dewatered pellets to a baling temperature, (e) dusting equipment adapted to receive the conditioned pellets from the drying device, said dusting equipment capable of applying a partitioning agent to the conditioned pellets, and (f) a baler adapted to receive the conditioned pellets from the dusting equipment, said baler capable of compressing
the conditioned pellets into a bale having a density of from 0.45 g/cm³ to 0.85 g/cm³.

[0017] In another aspect, the invention provides a bale of agglomerated pellets, said bale having a density of from 0.45 g/cm³ to 0.85 g/cm³, said pellets having a particulate density in the range of about 0.85 g/cm³ to about 0.91 g/cm³ at room temperature, said pellets comprising an amorphous or semi-crystalline polyolefin having a crystallisation enthalpy of less than 80 J/g as measured by DSC, and said pellets being at least partially covered on their surface by a partitioning agent. The bale may be formed by the processes described above.

BRIEF DESCRIPTION OF DRAWINGS

[0018] Figure 1 is a schematic representation of one embodiment of the invention.

[0019] Figures 2-6 are graphs of the power in-take (as measured by the Banbury motor amps) of inventive EDBs and comparative examples, as a function of the mixing time.

DETAILED DESCRIPTION

[0020] The easily dispersed bales of the current invention may be produced from dense rubber pellets of amorphous or semi-crystalline polyolefins, preferably formed by a direct devolatilization process.

[0021] Amorphous or semi-crystalline polyolefins may have a crystallisation enthalpy in general lower than 100 J/g, or lower than 80 J/g, or lower than 60 J/g, as determined by DSC.

[0022] Direct devolatilization processes are polymerization finishing processes that include devices permitting substantially complete removal of solvent and/or unreacted comonomer, generally with the polymer in a molten condition, before the polymer is brought to ambient pressure. Examples of such devices include, but are not limited to vented extruders, vented devolatilization devices, spray ring pelletizers, and any other devices that produces discrete, and non-agglomerating pellets. An example of such a process that produces dense rubber pellets is described in WO02/34795, incorporated herein by reference.
However, dense rubber pellets or rods can also be produced by breaking up crumb bales from conventional rubber finishing processes and feeding the crumb bales through a pelletizing device, provided that the pelletizing conditions are appropriately selected to provide dense pellets.

[0023] Referring now to Figure 1, in an embodiment of the invention molten polymer 10 comprising melted polymer and solvent from a polymerization reaction enters the devolatilization equipment 30 where the solvent or diluent is removed by a process such as squeezing, evaporating or flashing. The solvent-free (or diluent-free) rubber 50 is then processed in a pelletizer 70 where it is extruded or pushed through a die plate with a cutting device to produce the dense pellets. The devolatilized polymer may be advanced to a pelletizer die by a screw. Pelletization is preferably conducted in a cooling medium 75, and the pelletized die outlet is accordingly located in the cooling medium. The strands emerging from the die are cut, with the cut pellets slurried in the cooling medium.

[0024] In one embodiment, chilled water is used as the cooling medium for pelletizing amorphous or low crystalline grades of resin. The water temperature should typically be lower than about 100°C, or lower than 60°C, and can even be as low as about 4°C. The lower temperatures are sometimes needed to prevent massing or agglomeration of sticky pellets in the pelletization process.

[0025] Typical pellet dimensions range from about 2 millimeters (mm) in diameter up to 20 mm in diameter and have a height or length ranging from about 2 mm to about 20 or 30 mm. Pellets having a higher length/diameter ratio are often called rods. As opposed to crumb, the pellets used in the current invention have a high density, which improves ease of handling. There are minimal airborne particulates when processing dense pellets because almost no fines are present. In addition, the regular shapes and sizes of the dense pellets also contribute to easier handling, as opposed to the irregularly shaped crumb.

[0026] An EP(D)M pellet typically has a particulate density of 0.86 g/cm³ to 0.88 g/cm³ at room temperature, and a plastomer pellet will have a particulate density that ranges between 0.85 g/cm³ and 0.91 g/cm³ at room temperature, depending on its co-monomer content.
[0027] An anti-sticking agent(s) 80 may be added to the cooling medium to eliminate risk of pellet agglomeration in the pelletization process. For example, calcium stearate, zinc stearate, magnesium stearate, sodium stearate aqueous suspension and mixtures thereof may be used. Many other anti-sticking agents, such as waxes, surfactants and dispersants can also be used.

[0028] In one embodiment, a very low level of calcium stearate slurry is added into the pellet cooling medium. The calcium stearate slurry coats the pellet surface and renders it much less sticky, and therefore allows the resin to advance through the underwater pelletizer and spin dryer without agglomeration, lumping and plugging. In this embodiment, the calcium stearate level in the pellets is very low; for example, the amount of calcium stearate in the pellets may be from 10 or 50 parts per million (ppm) to 60 or 300 ppm. In a particular aspect of this embodiment, the calcium stearate coating allows high heating in the later solvent and water removal steps without severe pellet agglomeration.

[0029] The cooling medium is then removed from the pellet slurry 85 in a dewatering device 90, such as a spin dryer. After the pellet slurry 85 has been de-watered, the pellets 95 are then further conditioned in drying equipment 110 to remove surface moisture and to attain a proper baling temperature. Non-limiting examples of drying equipment include a fluidized bed conveyor (FBC), or a tumble dryer, or other similar devices used by those skilled in the industry. Generally the residual volatile of rubber pellets are dried to below 0.6 wt%. In one embodiment, the pellets 95 are heated by air in the drying equipment 110 in a first zone with an air temperature of from about 30 ºC to about 100 or 110ºC, and in a second zone the pellet temperature brought to baling temperature.

[0030] In one embodiment, the conditioned pellets 115 are dusted with a partitioning agent in suitable dusting equipment 120 before conveying the pellets to the baling equipment 150. Many kinds of partitioning agents, i.e. dusting agents, can be applied. Calcium stearate powder is an example of such a partitioning agent. The dusting agent may be a single dusting agent or a combination of more than one type of dusting agents. The selection of dusting agent types, to a large extent, depends on the final application by the users, and can be selected by those of ordinary skill in the art. Typical partitioning agents
are metal salts of organic aliphatic acids like calcium stearate powder, talc, calcium carbonate, clay, and crystalline polyolefin powders, like low or high density polyethylene, polypropylene, ethylene-vinylacetate copolymers. To make an EDB of the current invention, only a minimum amount of dusting is necessary to promote proper breakup of the EDB in a Banbury mixer. Too little dusting agent on the pellets may cause the pellets to be bound tightly together and, thus, a mixing advantage may not achieved. Too much dusting agent on pellets may cause the bale to fall apart prematurely even before loading into the storage and shipping containers.

[0031] The level of dusting agent required will be a further function of the specific surface of the particulate rubber. If calcium stearate dust is used on EP(D)M pellets, the level of dusting may be from a lower level of 0.01, or 0.05 wt% to an upper level of 0.2, or 0.5, or 2 wt%. The dusting device 120 can be selected from a number of commercial dusting devices, including, for example, Kason Vibratory Conveyor, Hirshel mixer, Rotary Tumbler, roller mixer, or any vibratory or screw conveyors. The dusting device 120 is selected to achieve uniform distribution and coating of dust on pellets. For more amorphous rubber types, higher dusting level, i.e. up to about 2 wt%, may be desirable to make an EDB. Also, the particle sizes of the dusts could have an impact on how much dusting agent will be used on an EDB. Typical dust size varies from 1 to 100 microns. However, dust particle sizes could be as large as 500 to 1000 microns. Generally, the finer the dust particle, the lower the dust level needed to make EDB.

[0032] In the baling operation the dusted pellets 125 are compressed under controlled temperature and pressure conditions. In one embodiment, the pellet bale 175 has minimal rebound as the bale exits the baler cavity, and the dwell time in the baler is minimized in order to accelerate processing.

[0033] Bales 175 may be made on standard rubber baling equipment 150 with normal control parameters, such as baler cavity pressure, dwell time during which the pellets are compressed in the cavity, and sometimes height control of the pellet bale 175 within the cavity. The baling temperature may be less than 85°C, or less than 80°C, or less than 75°C, or less than 65°C, or less than 55°C.
The baling temperature may be above 30°C, or above 35°C, or above 40°C. The baling temperature may be controlled by adjusting the temperature of a cooling or heating medium, and/or adjusting the pellet residence time in the pellet conditioning equipment 110. For example, in one embodiment, the baling temperature can be controlled by adjusting the vibration angle of the FBC to increase or decrease residence time. The desired baling temperature can be determined by reference to the freezing point of the crystalline phase of the polymer. The higher the freezing point of the polymer, the higher the baling temperature should be. Usually the baling temperature is about 5°C to 10°C higher than the freezing point of the crystalline phase of the polymer measured by DSC on the cooling cycle. The minimum baling temperature depends on the type of rubber, its molecular composition and crystallinity. For example, for EP and EP(D)M rubber, the minimum baling temperature is above 35°C for the semi-crystalline grades. And typically the pellets are heated up to 45°C to 75°C before baling.

During the baling process, which traps the air voids between the compressed pellets in forming an EDB, the baler cavity pressure is suitably from 50 to 1000 psig. In one embodiment, the dwell time is from a lower limit of 5 or 10 seconds, to an upper limit of 20 or 60 seconds. The dwell time controls the bale height once the bale 175 is removed from the baler. If the pellet temperature is cooler, a longer dwell time will be required. Correspondingly, a higher baling temperature allows a shorter dwell time and provides a higher production rate. The bale weight can be from a lower limit of 15, or 20, or 25 Kg, to an upper limit of 35, or 40 Kg. The ultimate bale bulk density is from a lower limit of 0.45, 0.66, or 0.7 g/cm³, to an upper limit of 0.85 g/cm³. The bales 175 can then be transported by roller or belt conveyors, etc. to film wrappers, metal detector, check weighers, autoloaders, robots, etc. to be loaded into shipping containers. Where necessary, cold or chilled pellets are heated up to temperatures above the minimum baling temperature prior to baling in order to make the EDBs without exerting extremely high baler cavity pressures, or very long dwell times.

The EDBs are then processed and packaged by conventional means into the shipping boxes or containers. For example, the bales are wrapped in
plastic film and stacked one over another in a shipping container, optionally without the use of support bale units (SBU). The bales stacked on the lower level will be somewhat compressed but will not exceed the density of a dense bale rubber, and therefore will retain the easy mixing characteristic of the EDB. However, SBU packaging units may still be used with the dense pellet EDB's of this invention.

[0036] The dense pellet EDB's of this invention may have one or more of the following advantages: very little, if any, fines present in the pellet handling process, which eliminates the health and fouling issues associated with sticky crumb and fines; the dense pellet EDB's of this invention can be made with a high bulk density as compared to traditional friable bales, which typically require lower bulk density such as from 0.5 g/cm$^3$ to 0.65 g/cm$^3$; and high bulk density EDBs have higher bale weights (as compared to a bale of the same size), and therefore have a higher production rate for a given baler, compared to producing conventional friable bales. Additionally, the EDB's of some embodiments of this invention may also show a shorter mixing cycle and a more efficient mixing than the conventional friable bales due to the easy breakup of EDB's in Banbury mixers. This breakup results in a higher rubber apparent volume leading to a higher filling efficiency of the mixer chamber, thus generating higher shear, and a higher rubber specific surface resulting in a higher energy absorption and a faster carbon-black incorporation. Also, in some embodiments, the dense pellet EDB's of the current invention may be made with a lower dusting level, and cost savings, as compared to low-density rubber particulates.

[0037] The process steps described above can be altered by those of ordinary skill in the art without affecting the resultant EDB, and accordingly such alterations are intended to be encompassed by the scope of this invention. For example, the dusting may be performed prior to dewatering, and then the dusted pellets conditioned for further heating and drying. Dusting can also be done by spraying aqueous suspensions of the partitioning agents onto the pellets after the dewatering equipment and upon entering the drying (or conditioning) equipment where the dusted pellets are heated and dried.
EXAMPLES

[0038] The following examples illustrate how this process is applied to produce EDB with an ethylene-propylene-diene rubber, EP(D)M. The same process can of course be applied to other amorphous or semi-crystalline polymers having suitable compressibility.

[0039] The rubber or polymer density is measured according to ASTM D297 with voids eliminated from the measurement. The pellet or particulate density may be measured according to ASTM D 1505.

[0040] Dense pellets of an EPDM having 73 wt% ethylene, 5 wt% ethyldene norbornene (ENB) and a Mooney viscosity of 60 (1+4 @ 125 C) are produced from a devolatilization pilot unit. The pellet diameter is about 4-5 mm and the thickness is about 1-3 mm, with the pellet density about 0.87 g/cm³. The pellets are dusted with 0.15 wt% calcium stearate powder in a plastic bag with rapid tumbling motions. The dusted pellets are then heated up in an oven for one hour to various temperatures and miniature bales of 120 grams each are made in a laboratory baling device, with 81 psig cavity pressure, and 10 seconds dwell time. The final bulk density of the resultant EDB bales varies with the baling temperature: 0.69 g/cm³ at 40°C, 0.78 g/cm³ at 50°C, and 0.78 g/cm³ at 60°C.

[0041] Dense pellets of an EPDM having 68 wt% ethylene, 5 wt% ENB and a Mooney viscosity of 75 (1+4 @ 125 C) are produced from a devolatilization pilot unit. The pellet diameter is about 4-5 mm and the thickness is about 1-3 mm, with the pellet density about 0.87 g/cm³. The pellets are dusted with 0.15 wt% calcium stearate powder in a plastic bag with rapid tumbling motions. The dusted pellets are heated up in an oven for one hour at various temperatures and then removed from the oven, cooled down by about 5°C to 15°C before baling at 81 psig cavity pressure and 10 seconds dwell time. The final bulk density of the resultant EDB bales varies with the initial pellet temperature: 0.59g/cm³ when heated to 45°C and baled at 40°C, 0.71 g/cm³ when heated to 55°C and baled at 40°C, and 0.76 g/cm³ when heated to 65°C and baled at 40°C.

[0042] Dense pellets of Vistalon® 7000 (73 wt% ethylene content) are made by pelletizing a crumb bale in a pelletizing extruder. The pellet diameter is about 6 mm and the thickness is about 2 mm, with pellet density about 0.87 g/cm³.
The pellets are dusted with calcium stearate powder at room temperature between 0.1 wt% and 0.2 wt% level. Dusted pellets are heated up in an oven overnight to 70-74°C, and bales of 2.5 Kg weight are produced in a lab baling device, with baling conditions of 160-225 psig cavity pressure, 1 minute dwell time, and baling temperature varying from 66°C to 75°C. The resultant EDBs have bulk densities varying from 0.58 g/cm³ to 0.82 g/cm³. The mixing behavior of such bales at 0.71 g/cm³ and 0.78 g/cm³ bulk density with 0.1 wt% and 0.15 wt% calcium stearate dust are evaluated via an upside down mixing process in a 20 liter Fawcett mixer, and compared to conventional Vistalon™ 7000 friable bale (V-7000 FB, typical density of 0.55 g/cm³).

[0043] Mixing is effected with carbon black and oil according to the following recipe:

Compound recipe:

<table>
<thead>
<tr>
<th>Ingredient</th>
<th>PHR</th>
</tr>
</thead>
<tbody>
<tr>
<td>Polymer</td>
<td>100</td>
</tr>
<tr>
<td>FEF N-550</td>
<td>120</td>
</tr>
<tr>
<td>Furnace Black</td>
<td></td>
</tr>
<tr>
<td>Flexon 878</td>
<td>60</td>
</tr>
<tr>
<td>Stearic Acid</td>
<td>2</td>
</tr>
<tr>
<td>Sulfur</td>
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</tr>
</tbody>
</table>

[0044] The EDB's all show much higher power peaks resulting from the explosion of the bale in particulate pellets in the mixer chamber, a higher mixing energy as shown by a larger area below the mixing curves, and a somewhat shorter carbon black incorporation time (BIT), indicating the more efficient mixing behavior of the EDB bales. Figure 2 shows the power in-take (as measured by the Banbury motor amps) of EDBs as compared to Vistalon™ 7000 friable crumb bale (FB), as a function of the mixing time.

[0045] Dense pellets of Vistalon® 5600 (68.5 wt% ethylene) and of Vistalon® 7000 (73 wt% ethylene) are made by chopping rubber bales and extruding the rubber chips in an industrial size single screw extruder at a rate of 750-1000 Kg/hr. The molten rubber is extruded in strands using an 8-mm die (die temperature was about 210°C) and an underwater pelletizing unit. Die swell occurs, so that the pellets that emerge from the die have a diameter D = 12 mm. The cutter rate is adjusted to produce rod-like pellets having 1/1 and 0.5/1 L/D
ratio. The pellets are subsequently dried in a spin drier and coated with about 0.1 wt% calcium stearate powder.

The pellets are then heated in a pilot fluidized bed conveyor (FBC) and fed to a manually operated baling unit. Several passes are performed in the FBC to reach the desired pellet skin temperature. EDB pellet bales of different densities are achieved by changing pellet skin temperature and dwell time in the press. The pellet skin temperature is measured using a laser thermocouple. The inner bale temperature is measured by inserting a thermocouple inside the bale. The bale weight is about 17 Kg. The experimental conditions are summarized in Table 2.

The EDB pellet-bales are then mixed in a 20-liter Fawcett internal mixer with carbon black and oil via an upside-down process according to the following recipe:

Compound recipe:

<table>
<thead>
<tr>
<th>Ingredient</th>
<th>PHR</th>
</tr>
</thead>
<tbody>
<tr>
<td>Polymer</td>
<td>100</td>
</tr>
<tr>
<td>FEF N-550 (Furnace Black)</td>
<td>130</td>
</tr>
<tr>
<td>Flexon 876</td>
<td>70</td>
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<tr>
<td>Stearic Acid</td>
<td>2</td>
</tr>
<tr>
<td>Sulfur</td>
<td>0.1</td>
</tr>
</tbody>
</table>

The mixing curves are compared to those obtained with conventional Vistalon® 5600 and Vistalon® 7000 made in friable bales (FB) (having a typical bulk density of 0.55 g/cm³). The mixing curves show the power in-take (as measured by the Banbury motor amps) of as a function of the mixing time. In a first series of experiments, the rubber bales are conditioned at room temperature before mixing (21°C). In a second series of experiments, the rubber bales are conditioned at 4°C to simulate a storage temperature potentially occurring in the winter season.

Comparative mixing curves are illustrated in Figures 3-6, where it can be seen that the EDB pellet bales mix at least as efficiently as the friable bales. The friable bales are obtained conventionally from friable crumbs produced upon extrusion by the sudden evaporation of a volatile, generally water turning into vapor. The pellet bales may mix more efficiently as seen by a higher and
earlier power peak developing when the bale explodes in particulate pellets in the mixer, and in some instances a higher mixing energy highlighted by a higher area under the mixing curve.

[0050] To verify the quality of the mixing, some of the compound is extruded in the form of a flat strip of 30 mm width and 2 mm thickness, and having several meters length. Careful examination of the tape surface defects using a light microscope did not reveal any trace of undispersed or poorly dispersed polymer in the compound, thereby indicating a dispersion at least as good as obtained with the friable bales.
Table 1

Vistalon® 7000 EDB

pellet bales

<table>
<thead>
<tr>
<th>Dusting wt %</th>
<th>Dusting temp °C</th>
<th>Baling conditions</th>
<th>Bale DENSITY (g/cm³) versus time after molding</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Pressure in mold psig</td>
<td></td>
</tr>
<tr>
<td>0.1</td>
<td>68 +/- 1</td>
<td>1</td>
<td>225</td>
</tr>
<tr>
<td>0.1</td>
<td>70 +/- 1</td>
<td>1</td>
<td>210</td>
</tr>
<tr>
<td>0.1</td>
<td>70 +/- 1</td>
<td>1</td>
<td>210</td>
</tr>
<tr>
<td>0.1</td>
<td>72 +/- 1</td>
<td>1</td>
<td>210</td>
</tr>
<tr>
<td>0.1</td>
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<td>1</td>
<td>210</td>
</tr>
<tr>
<td>0.15</td>
<td>66 +/- 2</td>
<td>1</td>
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</tr>
<tr>
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<td>72 +/- 1</td>
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</tr>
<tr>
<td>0.2</td>
<td>75 +/- 1</td>
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<td>210</td>
</tr>
</tbody>
</table>

Pellets heated in oven for 16-20 hours
Pellet size: 6mm dia x 2 mm height
Mold and press at pellet temperature
Dust Agent = Calcium stearate
Table 2  EDB pellet bales made with large pellets

<table>
<thead>
<tr>
<th>Bale No</th>
<th>Grade</th>
<th>Pellet dimensions L/D cm</th>
<th>FBC temperature Zone 1/2/3 (°C)</th>
<th>Number of passes in FBC (*)</th>
<th>pellet skin temp (°C)</th>
<th>dwell time in press (min)</th>
<th>bale core temperature (°C)</th>
<th>bale density (after 72 hrs)</th>
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<tr>
<td>1</td>
<td>V5000</td>
<td>0.5/1</td>
<td>50/120/120</td>
<td>3</td>
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<td>4.5</td>
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<td>0.00</td>
</tr>
<tr>
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<td>V6000</td>
<td>0.5/1</td>
<td>50/120/120</td>
<td>3</td>
<td>70</td>
<td>4.5</td>
<td>48</td>
<td>0.82</td>
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<td>1/1</td>
<td>50/110/110</td>
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<tr>
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<tr>
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<td>3</td>
<td>70</td>
<td>4.5</td>
<td>48</td>
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</tr>
</tbody>
</table>

(*) residence time in FBC is 1.5 minute.

[0051] All patents, test procedures, and other documents cited herein, including priority documents, are fully incorporated by reference to the extent such disclosure is not inconsistent with this invention and for all jurisdictions in which such incorporation is permitted.

[0052] While the illustrative embodiments of the invention have been described with particularity, it will be understood that various other modifications will be apparent to and can be readily made by those skilled in the art without departing from the spirit and scope of the invention. Accordingly, it is not intended that the scope of the claims appended hereto be limited to the examples and descriptions set forth herein but rather that the claims be construed as encompassing all the features of patentable novelty which reside in the invention, including all features which would be treated as equivalents thereof by those skilled in the art to which the invention pertains.

[0053] When numerical lower limits and numerical upper limits are listed herein, ranges from any lower limit to any upper limit are contemplated.
CLAIMS

What is claimed is:

EP CLAIMS

1. A process for producing bales of polyolefin comprising:
   a) in a devolutilizing step, removing solvent from a mixture of solvent and polymer,
   b) pelletizing said devolutilized polymer in a cooling medium to produce dense pellets,
   c) removing substantially all of said cooling medium from said dense pellets, preferably with a density inclusive of any voids of at least 0.8 g/cm$^3$,
   d) applying a partitioning agent to said dense pellets, and
   e) compressing said dense pellets in a bale mould for a sufficient time, temperature and pressure to obtain a bale of pellets, said bale having a bulk density of 0.45 g/cm$^3$ to 0.85 g/cm$^3$, preferably at least 0.7 g/cm$^3$.

2. The process according to claim 1, in which said polyolefin comprises an amorphous or semi-crystalline polyolefin, preferably having a polymer density of from 0.85 to 0.91 g/cm$^3$, and especially from 0.86 to 0.88 g/cm$^3$, and wherein said dense pellets have a particulate density of at least 90%, and especially 95%, of the polymer density of said amorphous or semi-crystalline polyolefin.

3. The process according to claim 1 or claim 2, in which said partitioning agent is applied after said pelletizing step and before said compressing step.

4. The process according to any of the preceding claims in which said partitioning agent is applied after said step of removing substantially all
said cooling medium and before said compressing step and/or is applied as an anti-sticking agent to said devolatilized polymer in said cooling medium.

5. The process according to any of the preceding claims in which said partitioning agent is applied in an aqueous suspension and/or is applied as a powder.

6. The process according to any of the preceding claims in which said partitioning agent is selected from one of calcium stearate, zinc stearate, magnesium stearate, sodium stearate or mixtures thereof.

7. The process according to any of the preceding claims in which said partitioning agent is calcium stearate and is applied at a treat rate of about 10 parts per million to 300 parts per million of said devolatilized polymer.

8. The process according to any of the preceding claims in which said cooling medium is at a temperature of between 4°C and 100°C, preferably less than 60°C.

9. The process according to any of the preceding claims in which said pelletizing step provides pellets in the size range of 2 mm to 30 mm in diameter, preferably in the size range of 2 mm to 30 mm in diameter, and optionally 20 mm in length, and preferably from at least 6 mm in diameter and length, and especially at least 8 mm in diameter and length.

10. The process according to any of the preceding claims in which said pelletizing step provides pellets with a particulate density in the range of about 0.85 g/cm³ to about 0.91 g/cm³ when measured at room temperature, and preferably less than 0.89 g/cm³, and especially from 0.86 g/cm³ to 0.88 g/cm³.
11. The process according to any of the preceding claims in which said step of removing substantially all of the said cooling medium from said pellets comprises removing said cooling medium to a level below 0.6 wt%.

12. The process according to any of the preceding claims in which the cooling medium is water and said removing step comprises the steps of dewatering and drying said pellets.

13. The process of claim 12 in which said pellets are dewatered in a spin dryer and optionally dried in a fluidized bed conveyor and/or tumble dryer.

14. The process according to any of the preceding claims in which the polyolefin is a terpolymer of ethylene-propylene-diene and said calcium stearate is applied as a partitioning agent at a treat rate of from 0.01 wt% to 2 wt% based on the weight of the terpolymer.

15. The process according to any of the preceding claims in which the baling temperature is selected to be 5°C to 10°C higher than the freezing point of the crystalline phase of the polyolefin as measured by DSC; said step of compressing said pellets is at a pressure of from 50 to 1000 psig; said step of compressing said pellets is at a temperature of between about 30°C to 85°C, preferably less than 80°C; and/or, said step of compressing said pellets is at a dwell time of between about 5 seconds and 60 seconds.

16. The process according to any of the preceding claims in which devolatilized polymer is pelletized in a cooling medium at a temperature of between about 4°C to about 100°C, preferably less than 60°C, to produce dense pellets having a particulate density of at least 90 %, preferably at least 95%, of the density of said amorphous or semi-crystalline polyolefin, and said dense pellets are compressed in a bale mould for a sufficient time, temperature and pressure to obtain a bale of pellets, said bale having a bulk density of 0.66 g/cm³ to 0.85 g/cm³.
17. A processing plant for producing bales from pellets of polyolefin, said processing plant comprising:
   a) a devolatilizer capable of removing solvent from molten polymer comprising solvent and polymer,
   b) a pelletizer in fluid communication with said devolatilizer, said pelletizer capable of concentrating the devolatilized polymer into dense pellets in a cooling medium,
   c) a dewatering device in fluid communication with said pelletizer, said dewatering device capable of removing at least a portion of said cooling medium from said dense pellets to provide dewatered pellets,
   d) a drying device adapted to receive said dewatered pellets from said dewatering device, said drying device capable of conditioning said dewatered pellets by removing substantially all said cooling medium from the dewatered pellets and heating said dewatered pellets to a baling temperature,
   e) dusting equipment adapted to receive said conditioned pellets from said drying device, said dusting equipment capable of applying a partitioning agent to said conditioned pellets, and
   f) a baler adapted to receive said conditioned pellets from said dusting equipment, said baler capable of compressing the conditioned pellets into a bale having a bulk density of 0.45 g/cm³ to 0.85 g/cm³.

18. The processing plant according to claim 17, further comprising a means for applying a partitioning agent as an anti-sticking agent to said devolatilized polymer in said cooling medium.

19. The processing plant according to claim 17 or claim 18 in which said dewatering device is a spin dryer and said drying device is a fluidized bed conveyor and/or a tumble dryer.
20. A bale of agglomerated pellets, said bale having a bulk density of 0.45 g/cm$^3$ to 0.85 g/cm$^3$, said pellets having a particulate density in the range of about 0.85 g/cm$^3$ to about 0.91 g/cm$^3$ at room temperature, said pellets comprising an amorphous or semi-crystalline polyolefin having a crystallisation enthalpy of less than 80 J/g as measured by DSC, and said pellets being at least partially covered on their surface by a partitioning agent.

21. The bale according to claim 20 in which said bale is formed by the process of:
   a) in a devolatilizing step, removing solvent from a molten polymer comprising solvent and polymer,
   b) pelletizing said devolatilized polymer in a cooling medium to produce dense pellets,
   c) removing substantially all of said cooling medium from said dense pellets,
   d) applying a partitioning agent to said dense pellets, and
   e) compressing said dense pellets in a bale mould for a sufficient time, temperature and pressure to obtain said bale.

22. The bale according to claim 20 or claim 21 in which said partitioning agent is selected from one of calcium stearate, zinc stearate, magnesium stearate, sodium stearate or mixtures thereof and is preferably calcium stearate at a concentration of about 10 parts per million to 300 parts per million of said devolatilized polymer.

23. The bale according to claim 22 in which said polyolefin comprises an ethylene-propylene-diene terpolymer, and said calcium stearate content comprises 0.01 wt% to 2 wt% based on the weight of the terpolymer.
- 23 -

24. The bale according to any of claims 20 to 23 in which said dense pellets are in the size range of about 2 mm to about 20 mm in diameter, and about 2 mm to about 30 mm in length, preferably less than 2 mm in length, and preferably said bale has a residual volatile content of below 0.6 wt%.

US CLAIMS

25. A process for producing bales of polyolefin, the process comprising:
   a) removing solvent from a mixture of solvent and polymer to produce a devolatilized polymer,
   b) pelletizing said devolatilized polymer in a cooling medium to produce dense pellets,
   c) removing substantially all of said cooling medium from said dense pellets,
   d) applying a partitioning agent to said dense pellets, and
   e) compressing said dense pellets in a bale mould for a sufficient time, temperature and pressure to obtain a bale of pellets, said bale of pellets having a bulk density of from 0.45 g/cm\(^3\) to 0.85 g/cm\(^3\).

26. The process of claim 25, wherein said bale of pellets has a bulk density of from 0.66 g/cm\(^3\) to 0.85 g/cm\(^3\).

27. The process of claim 25, wherein said bale of pellets has a bulk density of from 0.7 g/cm\(^3\) to 0.85 g/cm\(^3\).

28. The process of claim 25, wherein said polyolefin has a polymer density of from 0.85 g/cm\(^3\) to 0.91 g/cm\(^3\).

29. The process of claim 25, wherein said polyolefin has a polymer density of from 0.86 g/cm\(^3\) to 0.88 g/cm\(^3\).

30. The process of claim 28, wherein said polyolefin comprises an amorphous or semi-crystalline polyolefin, and wherein said dense pellets have a
particulate density of at least 90% of the polymer density of said amorphous or semi-crystalline polyolefin.

31. The process of claim 28, wherein said polyolefin comprises an amorphous or semi-crystalline polyolefin, and wherein said dense pellets have a particulate density of at least 95% of the polymer density of said amorphous or semi-crystalline polyolefin.

32. The process of claim 25, further comprising applying an anti-sticking agent to said devolatilized polymer in said cooling medium.

33. The process of claim 32, wherein said anti-sticking agent is selected from calcium stearate, zinc stearate, magnesium stearate, sodium stearate, and mixtures thereof.

34. The process of claim 32, wherein said anti-sticking agent is calcium stearate and is applied at a treat rate of about 10 parts per million to about 300 parts per million of said devolatilized polymer.

35. The process of claim 25, wherein said cooling medium is at a temperature of from about 4°C to about 100°C.

36. The process of claim 25, wherein said cooling medium is at a temperature of from about 4°C to about 60°C.

37. The process of claim 25, wherein said pelletizing step produces pellets in the size range of about 2 mm to about 20 mm in diameter, and about 2 mm to about 30 mm in length.

38. The process of claim 25, wherein said pelletizing step produces pellets in the size range of about 2 mm to about 20 mm in diameter, and about 2 mm to about 20 mm in length.
39. The process of claim 25, wherein said pelletizing step produces pellets in the size range of about 6 mm to about 8 mm in diameter, and about 6 mm to about 8 mm in length.

40. The process of claim 25, wherein said pelletizing step produces pellets with a particulate density in the range of about 0.8 g/cm³ to about 0.91 g/cm³ when measured at room temperature.

41. The process of claim 25, wherein said pelletizing step produces pellets with a particulate density in the range of about 0.85 g/cm³ to about 0.89 g/cm³ when measured at room temperature.

42. The process of claim 25, wherein said pelletizing step produces pellets with a particulate density in the range of about 0.86 g/cm³ to about 0.88 g/cm³ when measured at room temperature.

43. The process of claim 25, wherein step c) comprises removing said cooling medium to a level below 0.6 wt%.

44. The process of claim 25, wherein step c) comprises removing said cooling medium to a level below 0.6 wt% by dewatering and drying said pellets.

45. The process of claim 44, wherein said pellets are dewatered in a spin dryer.

46. The process of claim 44, wherein said pellets are dried in a fluidized bed conveyor.

47. The process of claim 44, wherein said pellets are dried in a tumble dryer.
48. The process of claim 25, wherein said partitioning agent is applied after step b) and before step e).

49. The process of claim 25, wherein said partitioning agent is applied after step c) and before step e).

50. The process of claim 25, wherein said partitioning agent is applied in an aqueous suspension.

51. The process of claim 25, wherein said partitioning agent is applied as a powder.

52. The process of claim 25, wherein said partitioning agent comprises calcium stearate.

53. The process of claim 52, wherein said polyolefin is a terpolymer of ethylene-propylene-diene, and said partitioning agent is applied at a treat rate of from 0.01 wt% to 2 wt% based on the weight of the terpolymer.

54. The process of claim 25, wherein the baling temperature is about 5°C to 10°C higher than the freezing point of the crystalline phase of the polyolefin as measured by DSC.

55. The process of claim 25, wherein said step of compressing said pellets is at a pressure of from 50 psig to 1000 psig.

56. The process of claim 25, wherein said step of compressing said pellets is at a temperature of from about 30°C to about 85°C.

57. The process of claim 25, wherein said step of compressing said pellets is at a temperature of from about 30°C to about 80°C.
58. The process of claim 25, wherein said step of compressing said pellets is at a dwell time of from about 5 seconds to about 60 seconds.

59. A process for producing bales of amorphous or semi-crystalline polyolefin, the process comprising:
   a) removing solvent from a mixture of solvent and polymer to produce a devolatilized polymer,
   b) pelletizing said devolatilized polymer in a cooling medium at a temperature of from about 4 °C to about 60°C to produce dense pellets having a density of at least 95% of the density of said amorphous or semi-crystalline polyolefin,
   c) removing substantially all of said cooling medium from said dense pellets,
   d) applying a partitioning agent to said dense pellets, and
   e) compressing said dense pellets in a bale mould for a sufficient time, temperature and pressure to obtain a bale of pellets, said bale of pellets having a density of from 0.66 g/cm³ to 0.85 g/cm³.

60. The process of claim 59, further comprising applying an anti-sticking agent to said devolatilized polymer in said cooling medium.

61. The process of claim 60, wherein said anti-sticking agent is selected from calcium stearate, zinc stearate, magnesium stearate, sodium stearate, and mixtures thereof.

62. The process of claim 60, wherein said anti-sticking agent is calcium stearate and is applied at a treat rate of about 10 parts per million to 300 parts per million of said devolatilized polymer.

63. The process of claim 59, wherein said pelletizing step produces dense pellets in the size range of about 2 mm to about 20 mm in diameter, and about 2 mm to about 20 mm in length.
64. The process of claim 59, wherein said pelletizing step produces dense pellets in the size range of about 6 mm to about 8 mm in diameter, and about 6 mm to about 8 mm in length.

65. The process of claim 59, wherein said pelletizing step produces pellets with a particulate density in the range of about 0.8 g/cm$^3$ to about 0.91 g/cm$^3$ when measured at room temperature.

66. The process of claim 59, wherein said pelletizing step produces pellets with a particulate density in the range of about 0.85 g/cm$^3$ to about 0.89 g/cm$^3$ when measured at room temperature.

67. The process of claim 59, wherein said pelletizing step produces pellets with a particulate density in the range of about 0.86 g/cm$^3$ to about 0.88 g/cm$^3$ when measured at room temperature.

68. The process of claim 59, wherein step c) comprises removing said cooling medium to a level below 0.6 wt%.

69. The process of claim 59, wherein step c) comprises removing said cooling medium to a level below 0.6 wt% by dewatering and drying said pellets.

70. The process of claim 69, wherein said pellets are dewatered in a spin dryer.

71. The process of claim 69, wherein said pellets are dried in a fluidized bed conveyor.

72. The process of claim 69, wherein said pellets are dried in a tumble dryer.
73. The process of claim 59, wherein said partitioning agent is applied after step b) and before step e).

74. The process of claim 59, wherein said partitioning agent is applied after step c) and before step e).

75. The process of claim 59, wherein said partitioning agent is applied in an aqueous suspension.

76. The process of claim 59, wherein said partitioning agent is applied as a powder.

77. The process of claim 59, wherein said partitioning agent comprises calcium stearate.

78. The process of claim 77, wherein said polyolefin is a terpolymer of ethylene-propylene-diene, and said partitioning agent is applied at a treat rate of from 0.01 wt% to 2 wt% based on the weight of the terpolymer.

79. The process of claim 59, wherein the baling temperature is about 5°C to 10°C higher than the freezing point of the crystalline phase of the polyolefin as measured by DSC.

80. The process of claim 59, wherein said step of compressing said pellets is at a pressure of from 50 psig to 1000 psig.

81. The process of claim 59, wherein said step of compressing said pellets is at a temperature of from about 30°C to about 85°C.

82. The process of claim 59, wherein said step of compressing said pellets is at a temperature of from about 30°C to about 80°C.
83. The process of claim 59, wherein said step of compressing said pellets is at a dwell time of from about 5 seconds to about 60 seconds.

84. A processing plant for producing bales from pellets of polyolefin, said processing plant comprising:

a) a devolatilizer capable of removing solvent from molten polymer comprising solvent and polymer,

b) a pelletizer in fluid communication with said devolatilizer, said pelletizer capable of concentrating the devolatilized polymer into dense pellets in a cooling medium,

c) a dewatering device in fluid communication with said pelletizer, said dewatering device capable of removing at least a portion of said cooling medium from said dense pellets to provide dewatered pellets,

d) a drying device adapted to receive said dewatered pellets from said dewatering device, said drying device capable of conditioning said dewatered pellets by removing substantially all of said cooling medium from the dewatered pellets and heating said dewatered pellets to a baling temperature,

e) dusting equipment adapted to receive said conditioned pellets from said drying device, said dusting equipment capable of applying a partitioning agent to said conditioned pellets, and

f) a baler adapted to receive said conditioned pellets from said dusting equipment, said baler capable of compressing the conditioned pellets into a bale having a bulk density of 0.45 g/cm$^3$ to 0.85 g/cm$^3$.

85. The processing plant of claim 84, further comprising a means for applying an anti-sticking agent to said devolatilized polymer in said cooling medium.
86. The processing plant of claim 84, wherein said dewatering device is a spin dryer.

87. The processing plant of claim 84, wherein said drying device is a fluidized bed conveyor.

88. The processing plant of claim 84, wherein said drying device is a tumble dryer.

89. A bale of agglomerated pellets, said bale having a bulk density of from 0.45 g/cm\(^3\) to 0.85 g/cm\(^3\), said pellets having a particulate density in the range of about 0.85 g/cm\(^3\) to about 0.91 g/cm\(^3\) at room temperature, said pellets comprising an amorphous or semi-crystalline polyolefin having a crystallisation enthalpy of less than 80 J/g as measured by DSC, and said pellets being at least partially covered on their surface by a partitioning agent.

90. The bale of agglomerated pellets of claim 89, wherein said bale is formed by the process of:
   a) in a devolatilizing step, removing solvent from a molten polymer comprising solvent and polymer to produce devolatilized polymer,
   b) pelletizing said devolatilized polymer in a cooling medium to produce dense pellets,
   c) removing substantially all of said cooling medium from said dense pellets,
   d) applying a partitioning agent to said dense pellets, and
   e) compressing said dense pellets in a bale mould for a sufficient time, temperature and pressure to obtain said bale.

91. The bale of agglomerated pellets of claim 90, wherein said partitioning agent is selected from calcium stearate, zinc stearate, magnesium stearate, sodium stearate, and mixtures thereof.
92. The bale of agglomerated pellets of claim 90, wherein said partitioning agent comprises calcium stearate at a concentration of about 10 parts per million to 300 parts per million of said devolatilized polymer.

93. The bale of agglomerated pellets of claim 90, wherein said polyolefin is a terpolymer of ethylene-propylene-diene, and wherein said partitioning agent comprises calcium stearate and is applied at a treat rate of from 0.01 wt% to 2 wt% based on the weight of the terpolymer.

94. The bale of agglomerated pellets of claim 90, wherein said dense pellets are in the size range of about 2 mm to about 20 mm in diameter, and about 2 mm to about 20 mm in length.

95. The bale of agglomerated pellets of claim 90, wherein said dense pellets are in the size range of about 6 mm to about 8 mm in diameter, and about 6 mm to about 8 mm in length.

96. The bale of agglomerated pellets of claim 90, wherein said bale has a residual volatile content of below 0.6 wt%.
Figure 2  
Vistalon® 7000 Pellet-bale mixing  
20 Liter Banbury

![Graph of Vistalon® 7000 Pellet-bale mixing showing motor amps against mixing time from ram down (sec). The graph includes various data points and labels such as Carbon black ingestion and BIT (C-black incorporation time).]

Figure 3  
V 5600R mixing curves  
bales conditioned at 21 C

![Graph of V 5600R mixing curves showing motor amps against mixing time from ram down (s). The graph includes data points for different bales and conditions.]

Figure 4
V 5600\textsuperscript{th} mixing curves
bales conditioned at 4 deg. C

Figure 5
V 7000\textsuperscript{th} mixing curves
bales conditioned at 21 C
Figure 6
V 7000R mixing curves
bales conditioned at 4 deg. C

- V 7000 FB
- Bale 6
- Bale 7

motor amps

mixing time from ram down (s)
INTERNATIONAL SEARCH REPORT

A. CLASSIFICATION OF SUBJECT MATTER

IPC(7) : B29C 47/00; C08I 3/12
US CL. : 264/115,123,141,142; 425/6,308,406; 428/407

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 264/115,123,141,142; 425/6,308,406; 428/407

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base searched other than minimum documentation to the extent that such documents are included in the fields searched

C. DOCUMENTS CONSIDERED TO BE RELEVANT

<table>
<thead>
<tr>
<th>Category</th>
<th>Citation of document, with indication, where appropriate, of the relevant passages</th>
<th>Relevant to claim No.</th>
</tr>
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Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier application or patent published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search

01 April 2004 (01.04.2004)

Date of mailing of the international search report

26 APR 2004

Authorized officer

Mary Lynn F. Theisen

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