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(54) **Title**: A PROCESS FOR CRYSTALLIZING POLYESTER CHIPS BY IR LAMPS

(57) **Abstract**: A process for crystallizing polyester chips having amorphousness greater than 95 % is disclosed. The process comprises: − providing polyester chips; − heating the polyester chips by a plurality of infrared lamps while being conveyed on a conveyor; and − collecting the infrared heated polyester chips from one operative end of the conveyor; wherein at least one factor selected from a group consisting of the speed of conveyance, the heat emission rate of said infrared lamps, the number of the infrared lamps and the residence time of the polyester chips on the conveyor, is controlled during conveyance, to obtain polyester chips having a degree of crystallization up to 50%.

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A PROCESS FOR CRYSTALLIZING POLYESTER CHIPS BY IR LAMPS

FIELD

The present disclosure relates generally to a process for crystallizing polyester resins and chips made therefrom, and more particularly to a process for crystallization of polyester resins and chips made therefrom using infrared heating lamps.

BACKGROUND

In today's world, thermoplastic and products made therefrom have become essential part of our life. This is evident from the use of thermoplastics in clothes, vehicles, appliances, etc. The significance gained by thermoplastics may be attributed to the properties like good mechanical strength, low density and high formability to name a few.

Typically, 'thermoplastics' are materials that flow upon heating and harden when cooled. Various useful goods including, but not limited to, containers for storing food, beverages, and other liquids as well as synthetic fibers may be manufactured using thermoplastics by using a wide variety of techniques, such as injection moulding, thermoforming, blow moulding, rotational moulding, and the like. Further, thermoplastics have the potential to be recycled by re-melting them to form new articles, or burnt and they can be used to generate electrical energy.

In particular, thermoplastics like polyesters have become essential commodities, whose manufacture is well known and established. Typical examples of polyesters are polyethylene terephthalate (PET), polyethylenenaphthalate (PEN), and similar polymers and copolymers. The polyesters such as PET may exist both in amorphous and semi-crystalline forms, the amorphous PET is transparent whereas the crystalline PET is opaque.

In accordance with the conventional process, the PET is formed by the process of esterification of terephthalic acid and ethylene glycol in a reaction vessel to form a mixture. The step of esterification may or may not include catalyst. The mixture is then heated to increase polymerization. The resulting mixture is then subjected to poly-condensation in a melt at elevated temperatures in the presence of an appropriate catalyst. The polymer is extruded directly from the poly-condensation reactor into strands. The hot, extruded strands

are contacted with cool water prior to chopping into chips, dried, and stored into silos prior to crystallization.

In particular, in accordance with the conventional process, two major and distinct process steps are involved in the production of high molecular weight polyesters, viz., the steps of melt polymerization and solid-state polymerization (SSP). In the step of melt polymerization for producing polyester having a high intrinsic viscosity (IV), a pre-polymer polyester having an IV of about 0.4 dL/g to 0.65 dL/g is produced in the form of chips.

The pre-polymer polyester so obtained is amorphous in nature. This pre-polymer polyester is subjected to solid-state polymerization, wherein the molecular weight of the polyester can be augmented further. In the step of solid-state polymerization, the polyester with desired intrinsic viscosity can be produced. However, before the step of solid-state polymerization the pre-polymer polyester has to be crystallized in a crystallizer. This step of crystallization is essential as direct solid-state polymerization of the pre-polymer polyester results in sintering and/or lump formation and hence to avoid the sintering and/or lump-formation in the pre-polymer polyester during the solid-state polymerization step, the pre-polymer polyester is subjected to crystallization.

In accordance with the conventional process, the pre-polymer polyester, which may have crystallinity of about 0 % to 5 %, is subjected to the process of crystallization, wherein the crystallinity of the pre-polymer polyester is increased up to 45% before it is subjected to the solid-state polymerization.

In accordance with the conventional process, the pre-polymer polyester is subjected to crystallization, wherein the temperature during the process of crystallization is in the range from 140°C to 210°C. The temperature depends on the co-monomer contents in the polyester resin.

In accordance with the conventional process, the residence time for achieving the desired crystallinity in the pre-polymer polyester is in the range of 45 minutes to 90 minutes. The pre-polymer polyester is required to be heated throughout this time interval in the temperature range of 140°C to 210°C.

Further, in order to crystallize the pre-polymer polyester, it is necessary to provide a series of process equipments such as pre-crystallizers, crystallizers, and the like that inevitably

consumes huge plant space and incurs large capital expenditure and operating expense for the process equipments.

Thus, it is apparent that the conventional crystallization process as described herein above, is time consuming and along with being energy inefficient.

Further, it is observed that, if the co-monomer content is increased for specialty application, the crystallization rate of the pre-polymer polyester is reduced, thereby increasing the crystallization time.

In summary, the overall output of the plant is affected by the crystallization process.

Hence, there is a need for improving the crystallization process and provide a process that is time and energy efficient. Further, there is a need for providing a crystallization process that enhances the overall plant output and hence is economically viable. Still further, there is a need for providing a crystallization process that substantially reduces plant space requirement and does not incur large capital expenditure and operating expense.

OBJECTS

Some of the objects of the present disclosure, which at least one embodiment herein satisfies, are as follows.

It is an object of the present disclosure to ameliorate one or more problems of the prior art or to at least provide a useful alternative.

An object of the present disclosure is to provide a process for crystallization of polyester that consumes less time.

Another object of the present disclosure is to provide a process for crystallization of polyester that is energy efficient.

Still another object of the present disclosure is to provide a process for crystallization of polyester that enhances the overall output of the plant.

Yet another object of the present disclosure is to provide an apparatus for crystallization of polyester that reduces the overall time for crystallization and at the same time is energy efficient.

Still another object of the present disclosure is to provide an apparatus for crystallization of specialty polyesters containing higher co-monomer contents (such as Isopthalic acid, naphthalene dicarboxylic acid, higher DEG, Polyethylene Glycol, Isosorbide, Penta Spiro Glycol, MP Diol Glycol (2-methyl-1,3-propanediol), Neo Pentyl Glycol, Propane Diol, Cyclohexanedimethanol (CHDM), Butane Diol and the like aliphatic and aromatic glycols which reduces the overall crystallization) without increasing the crystallization time and at the same time is energy efficient.

Yet another object of the present disclosure is to provide a process and an apparatus for crystallization of polyester that does not require huge plant space.

Still another object of the present disclosure is to provide a process and an apparatus for crystallization of polyester that does not incur large capital expenditure and operating expense.

Other objects and advantages of the present disclosure will be more apparent from the following description, which is not intended to limit the scope of the present disclosure.

SUMMARY

In accordance with the embodiments of the present disclosure, a process for crystallizing polyester chips having amorphousness greater than 95 % is disclosed. The process comprises the following steps:

- providing the polyester chips;
- heating the polyester chips by a plurality of infrared lamps while being conveyed on a conveyor; and
- collecting the infrared heated polyester chips from one operative end of said conveyor;

wherein at least one factor selected from a group consisting of the speed of conveyance, the heat emission rate of the infrared lamps, the number of the infrared lamps and the residence time of the polyester chips on the conveyor; is controlled during conveyance, to obtain polyester chips having a degree of crystallization up to 50%.

BRIEF DESCRIPTION OF ACCOMPANYING DRAWINGS

The disclosure will now be described with reference to the accompanying non-limiting drawing:

Figure 1 illustrates an apparatus for crystallizing polyester chips having amorphousness greater than 95 % in accordance with the present disclosure.

DETAILED DESCRIPTION

The disclosure will now be described with reference to the accompanying embodiments which do not limit the scope and ambit of the disclosure. The description provided is purely by way of example and illustration.

The embodiments herein and the various features and advantageous details thereof are explained with reference to the non-limiting embodiments in the following description. Descriptions of well-known components and processing techniques are omitted so as to not unnecessarily obscure the embodiments herein. The examples used herein are intended merely to facilitate an understanding of ways in which the embodiments herein may be practiced and to further enable those of skill in the art to practice the embodiments herein. Accordingly, the examples should not be construed as limiting the scope of the embodiments herein.

The description of the specific embodiments will so fully reveal the general nature of the embodiments herein that others can, by applying current knowledge, readily modify and/or adapt for various applications such specific embodiments without departing from the generic concept, and, therefore, such adaptations and modifications should and are intended to be comprehended within the meaning and range of equivalents of the disclosed embodiments. It is to be understood that the phraseology or terminology employed herein is for the purpose of description and not of limitation. Therefore, while the embodiments herein have been described in terms of preferred embodiments, those skilled in the art will recognize that the embodiments herein can be practiced with modification within the spirit and scope of the embodiments as described herein.

Conventional crystallization process for crystallizing pre-polymer polyester is time consuming and energy inefficient. Further, in order to crystallize the pre-polymer polyester, it

is necessary to provide a series of process equipments such as pre-crystallizers, crystallizers, and the like that inevitably consumes huge plant space and incurs large capital expenditure and operating expense for the process equipments. Hence, in order to overcome the afore-stated drawbacks, the present disclosure envisages a process for crystallizing polymer chips and in particular a process for crystallization of polyester resins or polyester chips using infrared heating.

Typically, the process for crystallization of the polyester chips is carried out before the polyester chips are sent to solid-state polymerization to avoid sintering and/or lump formation in the step of solid-state polymerization of the polyester chips.

In accordance with the present disclosure, a process for crystallizing polyester chips having amorphousness greater than 95 % is disclosed.

Figure 1 illustrates an apparatus 100 for crystallizing polyester chips having amorphousness greater than 95 % in accordance with the present disclosure. The apparatus 100 includes a container 10, a conveyor 20, a set of pulleys P1 and P2, a plurality of infrared lamps 30, a chamber c, a scrapper knife 40, an operative end o, a container 60 and a control panel 80.

The process comprises the following steps:

- providing polyester chips PC;
- introducing the polyester chips PC from the container 10 onto the conveyor 20, and heating the polyester chips PC by the plurality of infrared lamps 30 while being conveyed on the conveyor 20; and
- collecting the infrared heated polyester chips CP from the operative end o into the container 60 of the conveyor 20;

wherein at least one factor selected from a group consisting of the speed of conveyance, the heat emission rate of the infrared lamps 30, the number of the infrared lamps 30 and the residence time of the polyester chips PC on the conveyor 20, is controlled during conveyance, to obtain polyester chips having a degree of crystallization up to 50%.

In accordance with the present disclosure, the infrared lamps 30 are fitted on either side of the conveyor 20, and disposed in proximity to the conveyor 20, for heating the polyester chips PC. Moreover, the infrared lamps 30 are placed in the chamber c.

In accordance with the present disclosure, the scrapper knife 40 is adapted to facilitate scrapping of the crystallized chips on the conveyor belt 20 into free flowing chips to avoid lump formation on the belt.

In accordance with the present disclosure, the polyester chips PC are Polyethylene terephthalate (PET) chips.

In accordance with the present disclosure, length of the conveyor 20 is dependent upon throughput required. Typically, the length of the conveyor 20 in the experimental set up is 1 meter.

In accordance with the present disclosure, the speed of the conveyor 20 is very low. More specifically, the speed of the conveyor 20 varies with the % crystallinity requirement of the polyester chips PC. Typically, in the experimental set up it is 0.5 meters per min (mpm).

In accordance with the present disclosure, the polyester chips PC are laid down on the conveyor 20 in a single or maximum 2 layers of the polyester chips.

In accordance with the present disclosure, the conveyor 20 is an endless conveyor belt, that is, a conveyor in the form of a continuous belt traveling around the set of pulleys P1 and P2. Moreover, the speed of the conveyor 20 can be controlled by a speed controller (not shown in Figure 1) which is configured on the control panel 80.

In accordance with the present disclosure, the polyester chips PC are introduced into the conveyor 20 at a room temperature.

In accordance with the present disclosure, the polyester chips PC being introduced into the conveyor 20 have the shape chosen from a group consisting of rectangular, planar, spherical, and cylindrical or any other regular or irregular shape.

In accordance with the present disclosure, the infrared lamps 30 span the entire length of the conveyor 20.

In accordance with the present disclosure, the conveyor 20 is divided into a number of zones and the infrared lamps 30 are fitted in each zone accordingly as per temperature requirement. Particularly, the conveyor 20 is divided into two zones for achieving maximum temperature of 250°C. Moreover, the required temperature can be controlled by controlling the intensity of the infrared lamps 30 in each zone as per the % crystallinity of the polyester chips PC to be

achieved without lump formation. Typically, quartz infrared emitters having heating power of 6kW are used in the process of the present disclosure.

In accordance with the present disclosure, the polyester chips PC are heated by the infrared lamps 30 in the temperature range of 80 °C to 230 °C, depending upon the co-monomer content in the polyester chips PC.

In accordance with the present disclosure, the polyester chips PC are heated by the infrared lamps 30 for a time span of a few seconds subjected to the constraint that the crystallinity of the polyester chips PC after heating reaches at most up to 50 %.

In accordance with the present disclosure, the residence time of the polyester chips PC on the conveyor 20 is in the range of 5 to 900 seconds, to achieve the required % crystallinity of the polyester chips PC and to increase throughput of the process of the present disclosure at a desired crystallization temperature without lump formation.

In accordance with the present disclosure, a desired crystallinity of the polyester chips PC can be achieved by heating the polyester chips PC using the infrared lamps 30 in just a few seconds as compared to several minutes when heated in accordance with the conventional process, thereby reducing the process time substantially. The residence time required for achieving the desired crystallinity in accordance with the conventional process and the present disclosure is tabulated in Table 1 and Table 2.

Generally, the conventional process consists of a series of pre-crystallizers and crystallizers having different temperature zones.

Table 1:

Equipment	Residence time (minute)	Average outlet temperature (°C) of polyester chips	% crystallinity
Pre-crystallizer	12 - 15	195 - 198	40 - 42
Crystallizer-1	35	205 – 208	42 - 46

Crystallizer-2	35	209 – 210	46 - 50

(The data tabulated in Table 1 is for the polyester chips containing 1.8% of co-monomer, particularly isopthalic acid).

Table 2:

	1.8% of isopthalic acid	4.5% of isopthalic acid
IR Exposure	% Crystallinity	% Crystallinity
160°C/ 20 seconds	38.1	35.2
180°C/ 20 seconds	40.8	38.1
200°C/ 20 seconds	42.5	39.6

(The data tabulated in Table 2 is a pilot scale experimental data for crystallizing the amorphous polyester chips having 1.8% and 4.5% of co-monomer, particularly isopthalic acid).

From Table 1 and Table 2, it can be confirmed that the residence time required to achieve the desired crystallinity in accordance with the conventional process is greater than that of the process of the present disclosure. Hence, throughput of the plant increases substantially as the time required for crystallizing the polyester chips before solid-state polymerization has shrunk to a few seconds, even with the addition of the co-monomer instead of several minutes. This reduction in time is further beneficial for better color retention and lower acetaldehyde generation in the polyester resin in the solid-state polymerization step along with a decrease in the residence time in the solid-state polymerization step.

In accordance with the present disclosure, the length of the conveyor 20 and span of the infrared lamps 30 is such that the crystallinity of the chips increases up to 50%.

In accordance with the present disclosure, the crystallized polyester chips CP are sent to solid-state polymerization, wherein the sintering and/or lump formation is reduced and/or eliminated.

In accordance with the present disclosure, the energy required for heating the chips by the infrared lamps 30 is low as compared with the conventional process.

In accordance with the present disclosure, the plant space required to install the conveyor 20 along with the infrared lamps 30 is substantially low as compared with the conventional crystallization apparatus.

In accordance with the present disclosure, the capital expenditure and the operating expenses for operating the conveyor 20 along with the infrared lamps 30 is substantially low as compared with the conventional crystallization apparatus.

TECHNICAL ADVANCES AND ECONOMICAL SIGNIFICANCE

The present disclosure described herein above has several technical advantages including, but not limited to, the realization of a process for crystallizing polyester and/or polyester chips by IR lamps, that:

- is time and energy efficient;
- enhances the overall output of the plant;
- does not require huge plant space; and
- does not incur large capital expenditure and operating expense.

Throughout this specification the word "comprise", or variations such as "comprises" or "comprising", will be understood to imply the inclusion of a stated element, integer or step, or group of elements, integers or steps, but not the exclusion of any other element, integer or step, or group of elements, integers or steps.

The use of the expression "at least" or "at least one" suggests the use of one or more elements or ingredients or quantities, as the use may be in the embodiment of the disclosure to achieve one or more of the desired objects or results.

Any discussion of documents, acts, materials, devices, articles or the like that has been included in this specification is solely for the purpose of providing a context for the disclosure. It is not to be taken as an admission that any or all of these matters form a part of the prior art base or were common general knowledge in the field relevant to the disclosure as it existed anywhere before the priority date of this application.

The numerical values mentioned for the various physical parameters, dimensions or quantities are only approximations and it is envisaged that the values higher/lower than the

numerical values assigned to the parameters, dimensions or quantities fall within the scope of the disclosure, unless there is a statement in the specification specific to the contrary.

While considerable emphasis has been placed herein on the components and component parts of the preferred embodiments, it will be appreciated that many embodiments can be made and that many changes can be made in the preferred embodiments without departing from the principles of the disclosure. These and other changes in the preferred embodiment as well as other embodiments of the disclosure will be apparent to those skilled in the art from the disclosure herein, whereby it is to be distinctly understood that the foregoing descriptive matter is to be interpreted merely as illustrative of the disclosure and not as a limitation.

CLAIMS:

1. A process for crystallizing polyester chips having amorphousness greater than 95 %, said process comprising the following steps:
   - providing polyester chips;
   - heating said polyester chips by a plurality of infrared lamps while being conveyed on a conveyor; and
   - collecting said infrared heated polyester chips from one operative end of said conveyor;

   wherein at least one factor selected from a group consisting of the speed of conveyance, the heat emission rate of said infrared lamps, the number of said infrared lamps and the residence time of said polyester chips on said conveyor; is controlled during conveyance, to obtain polyester chips having a degree of crystallization up to 50%.

2. The process as claimed in claim 1, wherein the plurality of infrared lamps is disposed in proximity to said conveyor, for heating said polyester chips.
3. The process as claimed in claim 1, wherein said infrared lamps are fitted on either side of the conveyor.
4. The process as claimed in claim 1, wherein said polyester chips are laid down on said conveyor in at least one of a single layer and a double layer.
5. The process as claimed in claim 1, wherein a shape of said polyester chips is at least one of spherical, cylindrical, rectangular and planar.
6. The process as claimed in claim 1, wherein said infrared lamps span entire length of said conveyor belt.
7. The process as claimed in claim 1, wherein said polyester chips are heated by the infrared lamps in a temperature range from 80°C to 230°C.
8. The process as claimed in claim 1, wherein the conveyor is an endless conveyor belt and equipped with a speed controller, the infrared lamps are fitted on either side of the conveyor and the residence time of said polyester chips on said conveyor is configured to range between 5 to 900 seconds.

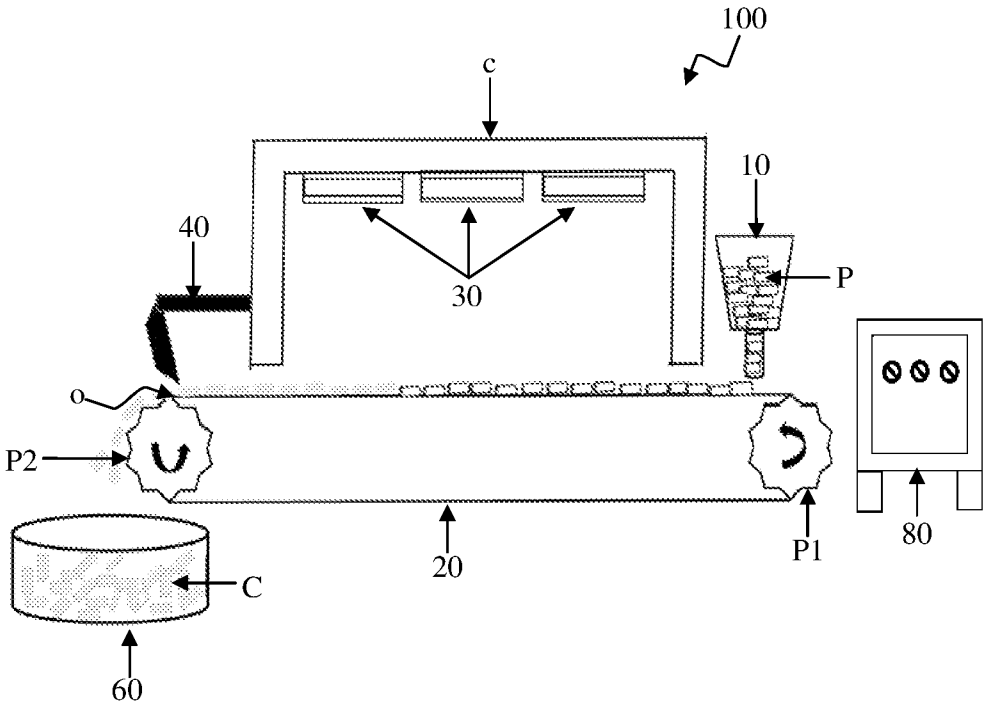

Figure 1