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(54) Title: AN EFFICIENT AND COST-EFFECTIVE PROCESS FOR PRODUCTION OF COLOURED THERMOPLASTIC POLYMER

(57) **Abstract:** An efficient and cost-effective process for the production of coloured thermoplastic polyester; the process comprising extrusion of at least one thermoplastic polyester with at least one inherently coloured polyester as a master batch. Uniformly coloured thermoplastic polyester, the polyester comprising at least one thermoplastic polyester and at least one inherently coloured polyester as master batch; said inherently coloured polyester comprises at least one chromophoric co-monomer such as carboxyl terminated chromophoric co-monomer, particularly metallo phthalocyanine tetracarboxylic acid, in the polyester backbone, said metal is selected from copper, cobalt, nickel, iron, calcium, barium, zinc or vanadyloxy or any other transition metal. The uniformly coloured polyester is further spinned into fibers or filaments having superior mechanical properties and improved spinnability besides wash fastness, rubbing fastness, sublimation fastness, light fastness, tenacity, % elongation, no extractible matter or no leaching of colour or uniform colouring. The uniformly coloured polyester is also spinned and blow molded into containers or bottles or films having uniform colouring and no extractible matter or no leaching of colour.

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TITLE OF THE INVENTION

An efficient and cost-effective process for production of coloured thermoplastic polymer

Technical field :

The present invention relates to an efficient and cost-effective process for producing coloured thermoplastic polyesters using inherently coloured polyesters as masterbatch compounds.

The invention also relates to coloured thermoplastic polyester comprising at least one thermoplastic polyester and at least one inherently coloured polyester as masterbatch, produced by the above process.

The present invention also relates to inherently coloured polyester which is used as masterbatch and is to be loaded in lesser amount to produce coloured polyester.

The present invention also relates to use of the above process and coloured polyester to produce coloured filament yarns, fibers, molded articles such as containers, bottles, films etc.

BACKGROUND OF THE INVENTION

Commonly coloured polyester have been utilized in innumerable applications, including but not limited to, fibers, filament yarns, container, bottles, films, casing, molded articles, etc. Numerous types of colouring agents have been utilized to produce coloured polyesters, including but not limited to, pigments, dyestuff, polymeric colourant and the like.

There are a number of techniques to add colour to the polyester, especially fibers or filament yarns or fabrics. One of the technique is dyeing either individual fibers before they are formed into yarns or yarns before they are formed into fabrics or woven or knitted fabrics. The term "dye" generally describes a colourant that is soluble in the material being coloured and the term "pigment" is used to describe insoluble colourants. In the case of dyeing of polymer fibres / filaments or fabrics, a wide set of needs exist. The disadvantages like air and water pollution are generally associated with various colouring processes. Moreover, polyester requires high pressure and temperature for the homogeneous dispersion of dyes. The dyeing procedure in itself is not eco-friendly and it also

involves extensive use of water, the carrier that gets polluted and hence need to be treated before it can be released outside.

In another technique, the above colouring agents have been added in the polyester by precolouring or masterbatch process in order to produce coloured polyester, particularly in pellet format. Such pellet generally includes other additives like antistatic agents or plasticizer, etc depending upon the end applications. Based on the melting mechanism, the colouring agent is usually introduced to the extrusion screw or other equipment. From here, the molten plastic can be extruded, injected etc and finally molded into articles. Uniform colouration needs thorough mixing of the colourant and plastic during melting phase.

However the problem of staining exists in the above traditional method. In most of the traditional methods, initially colourant is mixed with polyester along with other necessary additives which causes staining on the equipment parts. The resultant stains must be cleaned, more particularly when the polyester is to be coloured with different colour than that of previous. Thus the cleaning process adds considerable cost to the final product and also results into loss of valuable time required for cleaning further increasing the selling price of the product. This factor also affects the production efficiency of the process and hence of the manufacturing plant.

Alternatively other methods, such as injection molding have been used to avoid such problems. In the injection molding process, the coloring agents can be introduced during the actual injection molding step. However, this process is acceptable for low color loading applications. The number of problems associated with the injection molding especially at high color loading are poor color dispersion, processing difficulties, extended cycle times, screw slippages, and inconsistent injection pressures. However, they have proven to be less desirable due to difficulties in producing uniform colorations within the target polyester articles.

Further coloured polyesters prepared according to the prior art always have problems of leaching of colour or presence of extractable.

As such, other alternatives are necessary, both to avoid the difficult injection molding coloring procedures (which result in uniformity and IV problems) and high staining, and thus costly traditional precoloring or masterbatch processes. Any alternative methods must not deleteriously affect the IV of the resin itself. To date, no such viable alternatives have been developed or

followed for the production of coloured polymer as per the present invention to obviate the above mentioned problems associated with the prior art.

In our earlier Patent Application No. 931/MUM/2005, we have disclosed a process for making inherently coloured polyester fibers or filaments with controlled branching or even linear structures comprising the above chromophoric co-monomer in their backbone and inherently coloured polyester thereof.

The inherently coloured polyester prepared according to our above mentioned patent application in the pellet form containing chromophoric co-monomer in the range of 1 to 10 % which can be used as masterbatch for colouring the thermoplastic polymer by extrusion where very less colour loading is required and we can still achieve uniform distribution of colour by extrusion and avoid the staining of the equipment.

OBJECTS OF THE INVENTION:

An object of the present invention is to provide an efficient and cost-effective process for the production of coloured thermoplastic polyester by extrusion of at least one thermoplastic polyester and at least one inherently coloured polyester as masterbatch having superior mechanical properties.

Another object of the present invention is to provide an efficient and cost-effective process for the production of coloured thermoplastic polyester by extrusion of at least one thermoplastic polyester and at least one inherently coloured polyester as masterbatch in which the coloured thermoplastic polyester have no extractable coloured matter or problem of leaching.

Another object of the present invention is to provide an efficient and cost-effective process for the production of coloured thermoplastic polyester by extrusion of at least one thermoplastic polyester and at least one inherently coloured polyester as master batch, where the process achieves uniform distribution of colour in the thermoplastic polyester with very low loading of colour in the form of master batch.

Another object of the present invention is to provide an efficient and cost-effective process for the production of coloured thermoplastic polyester by extrusion of at least one thermoplastic polyester

and at least one inherently coloured polyester as master batch, where the process avoids staining of the equipment and no transition period required for two different colour batches.

Another object of the present invention is to provide an efficient and cost-effective process for the production of coloured thermoplastic polyester by extrusion of at least one thermoplastic polyester and at least one inherently coloured polyester as masterbatch, where the melt phase process does not adversely affect the molecular weight and the resulting properties of the polyester.

Another object of the present invention is to provide an efficient and cost-effective process for the production of coloured thermoplastic polyester by extrusion of at least one thermoplastic polyester and at least one inherently coloured polyester as master batch, where the melt phase of extrusion process does not affect the melt viscosity of the polymer which eases spinning stage.

Another object of the present invention is to provide an efficient and cost-effective process for the production of coloured thermoplastic polyester by extrusion of at least one thermoplastic polyester and at least one inherently coloured polyester as master batch, where the process is eco-friendly and safe.

Another object of the present invention is to provide coloured thermoplastic polyester comprising at least one thermoplastic polyester and at least one inherently coloured polyester as master batch, where the product is cost-effective.

Another object of the invention is to provide a master batch of inherently coloured polyester which is to be loaded in very less amount to produce coloured thermoplastic polyester and at the same time avoid staining of the equipment.

Another object of the invention is to provide use of the above process and coloured thermoplastic polyester prepared by the above process to produce coloured filament yarns, fibers, molded articles such as containers, bottles, films etc.

Another object of the invention is to provide coloured thermoplastic polyester fibers or filament yarns, where the fibers or filament yarns have superior mechanical properties and improved spinnability than that of fibers or filaments made from 100 % inherently coloured polyester.

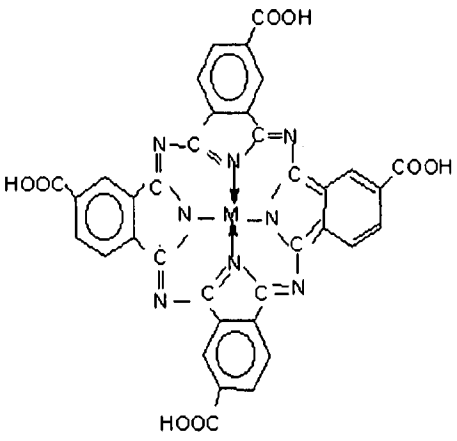
Another object of the invention is to provide films, particularly packaging film or bottles, particularly food grade bottles where the film or bottles have no extractible or problem of leaching of colour.

DETAILED DESCRIPTION OF THE INVENTION:

Inherently coloured polyester comprising 1 to 10% inherently coloured polyester is prepared according to the our earlier Patent Application No. 931/MUM/2005 and is used as a master batch in the present invention. Inherently coloured polyester comprising dicarboxylic acid monomer selected from pure terephthalic acid or isophthalic acid; diol monomer selected from ethylene glycol, 1:3 propane diol or 1:4 butane diol and ester of chromophoric co-monomer such as ester of metallo phthalocyanine tetracarboxylic acid; the metal is selected from copper, cobalt, nickel, iron or vanadyloxy or any other transition metal.

The chromophoric co-monomer is carboxyl terminated. Chromophoric co-monomer such as tetracarboxylic metallo phthalocyanine is used. The metal is, but not restricted to copper, cobalt, nickel, iron, calcium, barium, zinc, vanadyl oxy, or any other transition metal. One of the example of carboxyl terminated chromophore co-monomer is tetracarboxylic copper phthalocyanine. The co-monomers used here are indicative and those skilled in the art know that any other transition metal may be used to make the phthalocyanine and get the corresponding colour. Those skilled in the art also know that the above chromophoric co-monomer may have any of the transition metal as the central ion and may obtain the corresponding colour. The structural formula is given below:

Structural formula of metallo phthalocyanine tetra carboxylic acid



The term Inherently coloured polyester used here is intended to cover different coloured polyester prepared by changing the metal of the chromophoric co-monomer.

According to the invention there is provided an efficient and cost-effective process for the production of coloured thermoplastic polyester; the process comprising extrusion of at least one thermoplastic polyester with at least one inherently coloured polyester as a master batch; said inherently coloured polyester comprises at least one chromophoric co-monomer such as carboxyl terminated chromophoric co-monomer, particularly metallo phthalocyanine tetracarboxylic acid in the polyester backbone, said metal is selected from copper, cobalt, nickel, iron, calcium, barium, zinc or vanadyloxy or any other transition metal.

The above method is either a batch process or a continuous process. In the batch process the thermoplastic polyester and the inherently coloured polyester as a master batch are added in the form of chips for extrusion, the master batch is gravimetrically fed at any time during the extrusion while in the continuous process, the thermoplastic polyester and the inherently coloured polyester as a master batch are fed to the extruder from a continuous polymerization plant, the master batch is gravimetrically added by a transfer line injection at any time during the extrusion but before the melt spinning.

The extrusion is carried out at temperature in the range of 275-285° C in the nitrogen atmosphere.

The above process further comprises melt spinning the above extrudate to obtain coloured fiber or filament by conventional technique.

The above process further comprises injection molding of the above extrudate followed by blow molding to obtain molded articles such as containers, bottles or films by conventional technique.

According to the invention there is also provided uniformly coloured thermoplastic polyester, the polyester comprising at least one thermoplastic polyester and at least one inherently coloured polyester as a master batch; said inherently coloured polyester comprises at least one chromophoric co-monomer such as carboxyl terminated chromophoric co-monomer, particularly metallo

phthalocyanine tetracarboxylic acid, in the polyester backbone, said metal is selected from copper, cobalt, nickel, iron, calcium, barium, zinc or vanadyloxy or any other transition metal.

The thermoplastic polyester is selected from polyethylene terephthalate, polybutylene terephthalate, polytrimethylene terephthalate or co-polyesters thereof. The master batch of inherently coloured polyester comprising chromophoric monomer in the range of 1 to 10%. The concentration of chromophoric co-monomer in the coloured thermoplastic polyester is at least 0.01 % by weight of the coloured thermoplastic polyester. One can alter the concentration of chromophoric co-monomer in the coloured thermoplastic polyester to achieve desired shade as per the requirement of the end application. Uniformly coloured thermoplastic polyester is further converted into fibers, filament yarns, molded articles such as containers or bottles or films.

According to the invention there is also provided the coloured fibers or filament yarns comprising the above coloured thermoplastic polyester and prepared by the above process. The fibers or filaments have superior mechanical properties and improved spinnability

According to the invention there is also provided the molded articles such as containers or bottles or films comprising the above coloured thermoplastic polyester and prepared by the above process. The containers or bottles or films have no extractible or problem of leaching of colour.

Coloured thermoplastic polyester obtained by the above processes is characterized by measurement of Intrinsic viscosity, uniformity in colour, wash fastness, rubbing fastness, sublimation fastness , light fastness, no leaching of the colour or no extractable in the processed articles.

The fibers or filament yarns produced from the coloured thermoplastic polyester of the invention was tested for wash fastness, rubbing fastness, sublimation fastness and light fastness by conventional test methods. The results always rated between the scale of 1 to 5. The fiber or filament gives a rating in the range of 4-5.

The coloured containers or bottles or films were tested for extractible matter or leaching of colour and found to be not leachable.

Coloured thermoplastic polyester of the invention eliminates the need for dyeing of polymer fibers or filament yarns and becomes eco-friendly as it reduces all the emissions related to the step of

dyeing and also eliminates the use of large quantity of water for dyeing. It is also safe to human as it is not degradable in the presence of sunlight and does not leach out the dye. The process imparts inherent colour to the polymer, hence no use of pigments and no extra dyeing step are required. Moreover, since the metal complex is completely chemically linked to the backbone of the polyester, it does not get leached out or stain the equipment. Therefore the process eliminates the cleaning step, more particularly when the plastic to be coloured with different colour than that of the previous. It reduces the time required for maintenance, particularly cleaning. Thereby makes the process cost-effective and efficient and also increases production capacity of plant, further making the product cost-effective. It also eases the spinning process. It is observed that the process achieves uniform distribution of colour with very low loading of master batch. The process and coloured thermoplastic polyester of the invention gives uniformly coloured filament yarns or fibers with excellent resistance properties like wash fastness, rubbing fastness, sublimation fastness or light fastness besides superior mechanical properties and improved spinnability. The process and coloured thermoplastic polyester of the invention gives uniformly coloured molded articles such as containers or bottles or films with no extractible or no leaching of colour.

Although the invention has been described with reference to specific examples, it will be appreciated by those skilled in the art that the invention may be embodied in many other forms.

Example I

Preparation of Master batch (MB) chips (with 1.5 % chromophore by weight)

Copper phthalocyanine tetra carboxylic acid nonanol-ethylene glycol derivative was prepared according to example 5 of our earlier application no 931/MUM/2005 (1.5 % by wt of polyester) which was added to the slurry of monoethylene glycol (MEG) and purified terephthalic acid (PTA) in molar ratio of 2:1 (MEG:PTA) . The Catalyst Sb_2O_3, 400 ppm Sb, was added to the slurry and the esterification reaction was carried out under nitrogen pressure of 1.7 kg/cm^2 and at temperature of 260°C. The coloured oligomer thus obtained was then transferred to a polycondensation reactor. Vacuum was applied slowly to the polycondensation reactor and the final vacuum of around 1mm Hg was obtained in 45 min. The temperature was gradually increased to around 285°C. As the reaction progressed, the viscosity increased due to polymerization, hence torque and power of the agitator increased. After a certain rise in torque, vacuum was broken and the reactor was

pressurized with nitrogen and polymer was drained as strands and quenched in water bath. The strands were then cut into chips in a pelletizer, which were further dried to remove moisture.

Example 2

Preparation of coloured polyethyelene terephthalate (PET) by blending MB chips with PET (chromophoric comonomer concentration :0.125 %).

Polyethylene terephthalate and MB chips were extruded in the ratio of 91.7:8.3 w/w at temperature of 283 to 287° C to obtain blue coloured polyethylene terephthalate (PET) comprising 0.125 % chromophoric co-monomer. The PET was further converted into filament yarn by standard method. The conditions followed to produce filament yarn are given in Table 1. The filament yarn prepared was uniformly coloured. The filament yarn was tested for intrinsic viscosity and mechanical property namely tenacity which are given in table 2. The filament yarn was also tested for wash fastness, rubbing fastness, sublimation fastness and light fastness by standard methods and found to be in the scale of 4-5. (scale rate 5 = excellent and 1 = worst) The yarn was also tested for leaching of colour and extractible matter by standard methods. The leaching of colour or colour extractibles was not found.

Example 3

The blue coloured polyethyelene terephthalate (PET) having concentration of chromophoric comonomer 0.25 % was prepared by following the process as per example 2 except the ratio of PET to MB chips was 83.4 : 16.6 w/w. The PET was further converted into filament yarn by standard method. The conditions followed to produce filament yarn are given in Table 1. The filament yarn prepared was uniformly coloured. The filament yarn was tested for intrinsic viscosity and mechanical property namely tenacity which which are given in table 2. The filament yarn was also tested for wash fastness, rubbing fastness, sublimation fastness and light fastness by standard methods and found to be in the scale of 4-5. (scale rate 5 = excellent and 1 = worst) The yarn was also tested for leaching of colour and coloured extractible matter by standard methods. The leaching of colour of the yarn or colour extractibles was not found.

Example 4

The blue coloured polyethyelene terephthalate (PET) having concentration of chromophoric comonomer 0.375 % was prepared by following the process as per example 2 except the ratio of

PET to MB chips was 75.1 : 24.9 w/w. The PET was further converted into filament yarn by standard method. The conditions followed to produce filament yarn is given in Table 1. The filament yarn prepared was uniformly coloured. The filament yarn was tested for intrinsic viscosity and mechanical property namely tenacity which are given in table 2. The filament yarn was also tested for wash fastness, rubbing fastness, sublimation fastness and light fastness by standard methods and found to be in the scale of 4-5. (scale rate 5 = excellent and 1 = worst) The yarn was also tested for leaching of colour and extractible matter by standard methods. The leaching of colour or colour extractibles was not found.

Example 5

The blue coloured polyethyelene terephthalate (PET) having concentration of chromophoric comonomer 0.5 % was prepared by following the process as per example 2 except the ratio of PET to MB chips was 66.8 : 33.2 w/w. The PET was further converted into filament yarn by standard method. The conditions followed to produce filament yarn is given in Table 1. The filament yarn prepared was uniformly coloured. The filament yarn was tested for intrinsic viscosity and mechanical property namely tenacity which are given in table 2. The filament yarn was also tested for wash fastness, rubbing fastness, sublimation fastness and light fastness by standard methods and found to be in the scale of 4-5. (scale rate 5 = excellent and 1 = worst) The yarn was also tested for leaching of colour and extractible matter by standard methods. The leaching of colour or colour extractibles was not found.

Comparative example 6

The inherently coloured PET was prepared according to Example 2 of our Indian application no 931/MUM/2005 having 0.5 % chromophoric monomer. This was further converted into filament yarn by standard method. The conditions followed to produce filament yarn are given in Table 1. The filament yarn prepared was uniformly coloured. The filament yarn was tested for intrinsic viscosity and mechanical property namely tenacity which are given in table 2. The filament yarn was also tested for wash fastness, rubbing fastness, sublimation fastness and light fastness by standard methods and found to be in the scale of 4-5. (scale rate 5 = excellent and 1 = worst) The yarn was also tested for leaching of colour and extractible matter by standard methods. The leaching of colour or colour extractibles was not found.

Table 1 : process conditions employed to produce filament yarn

Examples	Example 6	Example 2	Example 3	Example 4	Example 5
Denier / Filament	75/36 FDY	75/36 - FDY	75/36 - FDY	75/36 - FDY	75/36 - FDY
Melt temp. (°C)	285.2	284.4	283.9	282.5	282
Pack pressure (Psi)	2320	2040	1950	1890	1810
Quench air temp. (°C)	19	19	19	19	19
Quench air velocity (m/s)	0.42	0.42	0.42	0.42	0.42
Draw ratio	2.9	2.9	2.9	2.9	2.9
Winding velocity (mpm)	3800	3800	3800	3800	3800

Table 2: Mechanical Properties

Examples	Example 6	Example 2	Example 3	Example 4	Example 5
Elongation (%)	36.33	25.0	24.02	29.69	33.1
Tenacity (gpd)	2.6	2.91	3.17	3.08	3.07
IV	0.57	0.561	0.59	0.58	0.552

Table 2 illustrates that mechanical properties of filament yarn prepared according to Examples 2 to 5 of the present invention are better than that of filament yarn prepared according to Example 6.

CLAIMS

1. An efficient and cost-effective process for the production of coloured thermoplastic polyester; the process comprising extrusion of at least one thermoplastic polyester with at least one inherently coloured polyester as a master batch; said inherently coloured polyester comprises at least one chromophoric co-monomer such as carboxyl terminated chromophoric co-monomer, particularly metallo phthalocyanine tetracarboxylic acid, in the polyester backbone, said metal is selected from copper, cobalt, nickel, iron, calcium, barium, zinc or vanadyloxy or any other transition metal.
2. The process as claimed in claim 1, wherein the thermoplastic polyester is selected from polyethylene terephthalate, polybutylene terephthalate, polytrimethylene terephthalate or co-polyesters thereof.
3. The process as claimed in claim 1, wherein the inherently coloured polyester is used as a master batch comprising chromophoric monomer in the range of 1 to 10%.
4. The process as claimed in claim 1, wherein the concentration of chromophoric co-monomer in the coloured thermoplastic polyester is at least 0.01 % by weight of the polyester.
5. The process as claimed in claim 1, wherein the process is either batch process or continuous process.
6. The process as claimed in claim 5, wherein the batch process comprising addition of the thermoplastic polyester and the inherently coloured polyester as a master batch in the form of chips for extrusion; feeding of the master batch at any time during the extrusion but before melt spinning while the continuous process comprising addition of the thermoplastic polyester and the inherently coloured polyester as a master batch to the extruder from a continuous polymerization plant; feeding the master batch by a transfer line injection at any time during the extrusion but before the melt spinning.
7. The process as claimed in claim 1, further comprises melt spinning the extrudate to obtain coloured fiber or filament.
8. The process as claimed in claim 1, further comprises injection molding of the extrudate followed by blow molding to obtain molded article such as container, bottle or film.
9. Uniformly coloured thermoplastic polyester, the polyester comprising at least one thermoplastic polyester and at least one inherently coloured polyester as master batch; said inherently coloured polyester comprises at least one chromophoric co-monomer such as carboxyl terminated chromophoric co-monomer, particularly metallo phthalocyanine

tetracarboxylic acid, in the polyester backbone, said metal is selected from copper, cobalt, nickel, iron, calcium, barium, zinc or vanadyloxy or any other transition metal.

10. Uniformly coloured thermoplastic polyester as claimed in claim 9, wherein the thermoplastic polyester is selected from polyethylene terephthalate, polybutylene terephthalate, polytrimethylene terephthalate or co-polyesters thereof.

11. Uniformly coloured thermoplastic polyester as claimed in claim 9, wherein the master batch of inherently coloured polyester comprising chromophoric co-monomer in the range of 1 to 10%.

12. Uniformly coloured thermoplastic polyester as claimed in claim 9, wherein the concentration of chromophoric co-monomer in the coloured thermoplastic polyester is at least 0.01 % by weight of the polyester.

13. Uniformly coloured thermoplastic polyester as claimed in claim 9, wherein the polyester is further converted into fibers or filament yarns or molded articles such as containers or bottles or films.

14. The fibers or filament yarns comprising the coloured thermoplastic polyester as claimed in claim 9 to 13 and prepared by the process as claimed in claims 1 to 7.

15. The fibers or filament yarns as claimed in claim 14, wherein the fibers or filaments have superior mechanical properties, improved spinnability, wash fastness, rubbing fastness, sublimation fastness, light fastness, tenacity or % elongation, no extractible matter or no leaching of colour or uniform colouring.

16. The molded articles such as containers or bottles or films comprising the coloured thermoplastic polyester as claimed in claim 9 to 13 and prepared by the process as claimed in claims 1 to 6 and 8.

17. The molded articles such as containers or bottles or films as claimed in claim 16, wherein the molded articles have uniform colouring or no extractible matter or no leaching of colour.