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(54) Title: CONTROLLED BRANCHED POLYESTER AND PROCESS FOR MAKING THE SAME

(57) Abstract: A process for making thermoplastic polyester with controlled branching or even with linear structures comprising of branching agents in its backbone, the process comprising a preparation of condensation product of at least one trifunctional carboxylic acid or anhydride thereof with at least one mono ethyl alcohol at temperature 255-265°C and under pressure 1.5- 1.8 kg/cm² and reacting the same with diols such as monoethylene glycol, 1,3 propane diol, 1,6 hexane diol or 1,4 butane diol to form an adduct. The polymer is further polymerized by solid state polymerization in nitrogen atmosphere at 190-210 °C to obtain chips of high molecular weight and are extruded and melt spun into filament or fiber or extruded into films and post process curable articles. The polyester formed resulted in increased rate of Solid state polymerization and reduced rate of polymer crystallizability.

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TITLE OF THE INVENTION**Controlled branched polyester and process for making the same****FIELD OF THE INVENTION**

5 The present invention relates to polyester with controlled branching having composition comprising at least one carboxylic acid or anhydride or ester thereof, at least one diol or polyol and at least one branching agent; said branching agent comprises at least one tri-functional carboxylic acid or anhydride thereof, at least one mono alkyl alcohol and at least one diol which is uniformly distributed in the polymer backbone.

10 The present invention also relates to a process of making the above polyester with controlled and uniformly distributed branching in the polymer backbone.

15 The present invention also relates to use of the above polyester in manufacturing post process curable fiber or filament, molded articles with high impact resistance and transparent films.

BACKGROUND OF THE INVENTION

20 Thermoplastic polyesters are widely used in manufacturing of fibers or filaments or films or container by extrusion, molding (injection or stretch blow). However, the polyester is known for deficiencies in melt rheology. In particular, polyester has typically low melt viscosity, low melt strength and low melt elasticity. Such limitations have left the polyester with narrow processing window and always required specialized equipments for processing. Generally melt viscosity and melt strength of polyester can be improved by introducing branching in the linear chain
25 structure of the polyester and /or increasing molecular weight through chain extension. One approach of introducing branching in polyester is by adding branching agent such as poly-functional carboxylic acids or anhydrides or alcohols directly during the polymerization reaction or melt mixing process. Depending upon the type of branching agent used to modify polyester, the overall effect of the reaction-melt mixing may in fact decrease the molecular weight of the
30 polyester. This will often be the case where the only type of branching agent used is a polyol branching agent. Most widely used branching agents are tri-functional monomer such as trimellitic anhydride, 1,3,5-Benzenetricarboxylic acid, 1,1,1-Tris(hydroxymethyl)ethane, 1,1,1-Tris(hydroxymethyl)propane and tetra-functional monomer such as pentaerythritol for polyester. Regardless of the type of branching agent used to effectively modify the polyester, it is

important that the degree of branching can be controlled during the reaction and that the branching occurs uniformly in the polyester. The most simple way in which the agent can be added to the polyester is by direct addition. However, this mode of addition has been found to lead to gel formation through excessive localized chain coupling and non uniform branching within the modified polyester. This also leads to detrimental discolouration. Furthermore, it is not uncommon to use amount of less than 1000 parts per million (ppm) weight percent of the branching agent, relative to the polyester to be modified. At these low levels, it is difficult to provide a uniform distribution of the branching agent in the polyester by direct addition which leads to non-uniform branching in polyester. The above branching agents, being multifunctional, are active and if used directly in the polymerization reaction, it may be difficult to control the chain length and distribution of branching, which ultimately affects melt viscosity. Thus non-uniform branching makes the polyester product inconsistent in quality including inconsistent polyester rheology which leads to spinning problems as well as abnormalities in down stream processing.

We have not come across any existing reference of the polyester with controlled branching having composition comprising at least one carboxylic acid or anhydride or ester thereof, at least one diol or polyol and at least one branching agent; said branching agent comprises at least one tri-functional carboxylic acid or anhydride thereof, at least one mono alkyl alcohol and at least one diol which is uniformly distributed in the polymer backbone as per the invention.

In our earlier patent application no. 395/MUM/2007 dated 1st March 2007, we have disclosed a process for making novel adducts as branching agents containing at least one trifunctional carboxylic acid or anhydride and one mono alkyl alcohol having carbon chain length C1- C25, The branching agent prepared as per the above mentioned patent application is used between 0.1 –10 percentage by weight preferably between 0.2 to 3 percentages is used in this invention, for making branched condensation polymers. The branching agent is added to a mixture of purified terephthalic acid and monoethylene glycol slurry in different molar ratios at temperature 260-265 °C under nitrogen atmosphere followed by polycondensation at 275-285 °C under reduced pressure,

OBJECTS OF THE INVENTION:

An object of the invention is to provide polyester with controlled branching having composition comprising at least one carboxylic acid or anhydride or ester thereof, at least one diol or polyol

and at least one branching agent; said branching agent comprises at least one tri-functional carboxylic acid or anhydride thereof, at least one mono alkyl alcohol and at least one diol which is uniformly distributed in the polymer backbone.

- 5 Another object of the invention is to provide polyester with controlled branching; said product is cost effective as there is no occurrence of gelling.

Another object of the invention is to provide polyester with controlled branching; said product is consistent in quality as the branching occurred uniformly in the polymer chain and controlled
10 chain length.

Another object of the invention is to provide a process for making the above polyester with controlled branching; said process is cost-effective as there is no occurrence of gelling.

- 15 Another object of the invention is to provide a process for making the above polyester with controlled branching; said process gives product with controlled branching and uniformly distributed in the polymer backbone.

Another object of the invention is to provide the use of the above polyester in manufacturing
20 post process curable fiber or filament, molded articles with high impact resistance, higher melt strength for blown film extrusion and extrusion blow molding and transparent films.

DETAILED DESCRIPTION

According to the invention, there is provided polyester with controlled branching having
25 composition comprising at least one carboxylic acid or anhydride or ester thereof, at least one diol or polyol and at least one branching agent; said branching agent comprises at least one tri-functional carboxylic acid or anhydride thereof, at least one mono alkyl alcohol and at least one diol which is uniformly distributed in the polymer backbone.

- 30 According to the invention, there is provided a process for making the above polyester with controlled branching, the process comprises:

- a esterifying at least one carboxylic acid, anhydride or ester thereof and at least one diol or polyol at temperature in the range of 255 to 265° C under pressure in the range of 1.5 to 1.8 Kg/cm²,
- b adding at least one branching agent which comprises at least one tri-functional carboxylic acid or anhydride thereof, at least one mono alkyl alcohol and at least one diol at the end of the esterification reaction followed by agitating the reaction mixture for at least 10 min; and
- c polycondensing the reaction mixture at temperature in the range of 260 to 290° C under pressure in the range of 0.5 to 1mm Hg and draining the polyester.

The dicarboxylic acid is selected from terephthalic acid, adipic acid, glutaric acid, naphthalene dicarboxylic acid, perylene dicarboxylic acid. The diol or polyol is selected from ethylene glycol, butanediol, 1,3-propane diol, 1,6-hexane diol . The mole ratio of hydroxyl group to carboxyl group is in the range of 1.2 to 2. The reaction temperature also affects the reaction rate of poly-condensation. It is preferred to carry out the poly-condensation reaction at temperature in the range of 260-285°C. The above process is either a batch process or a continuous process. In the batch process the branching agent is added towards the end of esterification reaction followed by agitation for at least 10 minutes while in the continuous process, the molten adduct is added by a transfer line injection followed by mixing for at least 10 minutes.

The tri-functional carboxylic acid or anhydride thereof used in the branching agent is selected from 1,3,5-Benzene-tri-carboxylic acid, 1,2,3-Benzene-tri-carboxylic acid or anhydride thereof, 1,2,4-Benzene-tri-carboxylic acid (Trimellitic acid) or anhydride thereof, 2,3,6-naphthalene tri-carboxylic acid or anhydride thereof, perylene tri-carboxylic acid or anhydride thereof. The mono alkyl alcohol used in the branching agent is a long chain alcohol having carbon chain length of C₁ to C₂₅ or highly substituted branched aliphatic or aromatic alcohol. Preferably, the long chain mono alkyl alcohol is selected from butanol, hexanol, heptanol, octanol, nonanol or dodecanol. Preferably highly substituted branched aliphatic or aromatic alcohol is selected from isobutanol, isooctyl alcohol, isododecyl alcohol, neopentyl alcohol or benzyl alcohol. The diol used in the branching agent is selected from monoethylene glycol, 1,3-propane diol, butane diol or 1, 6 Hexane diol. The ratio of trifunctional carboxylic acid or anhydride thereof, mono alkyl alcohol and diol is in the range of 1:0.5:2 to 1:1.5:7. The branching agent is added in the range of 0.1 to 10 % by wt in the polyester, preferably between 0.4-2 % by weight of the polyester.

In one of the embodiment of the invention, the above polyester is used in the manufacture of low pill fiber or filament.

5 In another embodiment of the invention, the above polyester is used in the manufacture of molded articles with high impact resistance.

In yet another embodiment of the invention, the above polyester is used in the manufacture of transparent films.

10 Thus the present invention allows the change in the composition of matter of polyester in terms of extent and length of branching to suit the polyester for end application requiring specific attributes like post process curable fiber, filament film or molded articles with high impact or transparency.

15 Incorporation of the above branching agent into the polyester backbone increased the free volume due to the disturbance in the dense chain packing of the polyester by the pendent alkyl units, which, in turn, facilitate higher melt strength for blown film extrusion and extrusion blow molding, improved elongation in fibers and filaments. Polyester of the invention has crystallization temperature in the range of 180-219° C and poly-dispersity in the range of 2.35 to
20 3.64. Die swell of the polymer increased with increase in the concentration of branching agent. This suggests the increased chain entanglement with increased branching and decrease in crystallization rate which is seen from the drop in crystallization temperature from DSC data. According to the die swell data, the polyester has uniform branching in the polymer chain and controlling chain length of the alkyl group of the branching agent controls the branching length.
25 There is no occurrence of gelling in the polymerization as well as in the product. Thus the product and process are cost-effective. The process results in products with consistent quality. The base chips can be either melt spun into fibers or melt cast into films, or processed to give molded articles which can be further polymerized in solid state, to give post process curable articles. Moreover, the process described in this invention gives a product with
30 reduced gelling tendency compared to the products produced using conventional branching agents.

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 1 : 1, 4- bis(2-hydroxy ethyl) 2-octyl tribenzoate

Trimellitic anhydride (424 g, 2.2 mol) and 1-octanol (287g, 2.2 mol) was heated at 200°C under
5 nitrogen pressure of 25 psi with stirring. The reaction was carried out for 4 hours. The reaction
mixture was cooled and further depressurized. To this reaction mixture, monoethylene glycol
(MEG) (548 g 8.8 mol) was added. The reaction was further carried out at temperature of 220°C
under the nitrogen pressure of 25 psi. for 4 hours. The reactor was depressurized and product
was drained under hot condition. The product was characterized by NMR and saponification
10 value.

¹H-NMR (CDCl₃, ppm): 8.5 (d, 1H), 8.2 (dd, 1H), 7.7 (d, 1H), 4.7 (m, 2H), 4.4 (m, 2H), 4.3 (m,
2H), 3.9 (m, 4H), 1.8 (m, 2H), 1.3 (m, 10H), 0.95 (t, 3H).

Sap.Value = 410 meq KOH/g

15 Example 2: Polyester without branching agent (Control sample)

PET polymer having IV of 0.6 dl/g prepared by melt-phase polymerization process. Purified
terephthalic acid (PTA) (6 kg) and monoethylene glycol (MEG) (8 Kg) were charged in 1:2
molar ratio. To the above reaction mixture, antimony trioxide catalyst (2.8 gm) were added. The
20 esterification reaction was carried out at 256°C, under a pressure of 1.5-1.85kg/cm² . The
oligomer obtained was then subjected to polycondensation at temperature of 285°C under
reduced pressure 1-5 mm Hg to obtain the polymer. After a certain rise in torque, vacuum was
broken and the reactor was pressurized with nitrogen. The polymer was drained as strands by
quenching in a water bath. The strands are then cut into chips in a pelletizer. The polyester was
25 analysed by IV, GPC, DSC and Rheology and used as control polyester. The prepolymer chips
are amorphous in nature. They were crystallized at 140 °C to obtain the crystalline
prepolymer.

The crystallized prepolymers were polymerized to high molecular weight polymer by solid
30 state polymerization at 200°C. Samples were taken at different time intervals and IV values
were measured. The results are given in Table.2.

Example 3, 4 & 5 : Polyester with 0.21 , 0.42 and 1.0 (wt %) of branching agent prepared according to Example 1;

Monoethylene glycol MEG (6 kg) and purified terephthalic acid (PTA) (8 kg) (1:2 molar ratio) was esterified in the presence of Sb_2O_3 (2.8 g) as a catalyst at 260°C under nitrogen pressure of 1.5 - 1.85 kg / cm^2 . The branching agent prepared according to the example 1 was added at the ratios given in Table 1 at the end of esterification at 260°C and the resultant reaction mixture was further agitated for 30 minutes.

Table 1. Wt % of branching agents used for different examples

Example no.	3	4	5
Wt branching agent from Example 1	12.6 gm (0.21 wt %)	25.2 gm (0.42 wt %)	61.2 gm (1.02 wt %)

The esterified mixture was polycondensed at 285°C under reduced pressure of 1-5 mm Hg. After a certain rise in torque, vacuum was broken and the reactor was pressurized with nitrogen. The polymer was drained as strands by quenching in a water bath. The strands are then cut into chips in a pelletizer. The polymer is characterized by IV, DSC and GPC methods. The presence of branching in copolyester was confirmed by GPC, DSC and Rheology.

The prepolymer chips are amorphous in nature. They were crystallized at 140°C to obtain the crystalline prepolymer.

The crystallized prepolymers were polymerized to high molecular weight polymer by solid state polymerization at 200°C . Samples were taken at different time intervals and IV values were measured. The results are given in Table.2.

Table 2. Build –up of Intrinsic viscosity (IV) of the polymer during SSP at 200 °C

Ex. 2 (Control Sample without Branching Agent)		Ex.3(0.21 wt%)		Ex 4(0.42 wt%)		Ex 5(1.02 wt%)		Ex 9 (0.15% penta erithritol)	
Time	IV*	Time	IV*	Time	IV*	Time	IV*	Time	IV *
Hrs	dl/gm	Hrs	dl/gm	hrs	dl/gm	hrs	dl/gm	hrs	dl/gm
0	0.605	0	0.588	0	0.582	0	0.588	0	0.574
2	0.613	2	0.619	2	0. 612	2	0.653	2	0.677
4	0.681	4	0.653	4	0.655	4	0.721	4	0.708
6	0.693	6	0.675	6	0.69	6	0.851	6	0.763
8	0.74	8	0.695	8	0.736	8	0.81	8	gelled

* IV (intrinsiv viscosity) measured at 30 C in phenol/tetra chloro ethane (60:40 w/w%)

5 according to ASTM D 4603-96 method

Example 6: (Comparative example)

10 The crystallized polymer prepared in Example 4, was solid state polymerized at 160 & 180 °C and IV build-up was measured at different time intervals. The IV build up did not take place and it was remaining at 0.588 even after 8hrs at 160 or at 180 °C indicating solid state polymerization did not take place at the 160 or 180 °C

Example 7 & 8 : Polyester with 2.1 wt %, 3.2 wt % branching agent

15 Monoethylene glycol MEG (6 kg) and purified terephthalic acid (PTA) (8 kg) (1:2 molar ratio) was esterified in the presence of Sb₂O₃ (2. 8 g) as a catalyst at 260°C under nitrogen pressure of 1.5 - 1.85 kg / cm². The branching agent prepared according to the example 1 was added at ratios given in Table 3 at the end of esterification at 260 °C and the resultant reaction mixture was further agitated for 30 minutes.

Table 3. Wt % of branching agents used for different examples

Example no.	7	8
Wt branching agent from Example 1	126 gm (2.1 wt %)	192gm (3.2 wt %)

The esterified mixture was polycondensed at 285°C under reduced pressure of 1-5 mm Hg. After a certain rise in torque, vacuum was broken and the reactor was pressurized with nitrogen.

5 The polymer was drained as strands by quenching in a water bath. The strands are then cut into chips in a pelletizer. The polymer is characterized by IV, DSC and GPC methods. The presence of branching in copolyester was confirmed by GPC, DSC and Rheology.

10 The prepolymer chips are amorphous in nature. They were crystallized at 140 °C to obtain the crystalline prepolymer. The crystallized chips were polymerized further by SSP at 190, 200, and 210 °C. The samples were taken at different time intervals to check molecular weight build-up. Intrinsic viscosity (IV) could not be measured. The polymer got gelled. The gel content measured at different times (for the polymer prepared by Example 4) are given in Table 4

15

Example 9 : Polyester containing 0.15 % (by wt) penta erithritol as branching agent

Monoethylene glycol (MEG) (5.25 kg) and purified terephthalic acid (PTA) (7 kg) (1:2 molar ratio) was esterified in the presence of Sb₂O₃ (2.45 g) as a catalyst at 260°C under nitrogen pressure of 1.5 - 1.85 kg / cm². The branching agent penta erithritol (10.5 gm) was added as a solution in MEG (100ml) at the end of esterification at 260 °C and the resultant reaction mixture was further agitated for 30 minutes.

20

25 The esterified mixture was polycondensed at 285°C under reduced pressure of 1-5 mm Hg. After a certain rise in torque, vacuum was broken and the reactor was pressurized with nitrogen. The polymer was drained as strands by quenching in a water bath. The strands are then cut into chips in a pelletizer. The polymer is characterized by IV, DSC and GPC methods. The presence of branching in copolyester was confirmed by GPC, DSC and Rheology.

30 The prepolymer chips are amorphous in nature. They were crystallized at 140 °C to obtain the crystalline prepolymer. The crystallized chips were polymerized further by SSP at 200°C.

The samples were taken at different time intervals to check molecular weight build-up. Intrinsic Viscosity (IV) data for the polymer is given in Table 2.

Table 4 Gel content of polymer prepared as per Example 7, polymerized in the Solid state at different temperatures

Ex 7(2.1wt%) 210 C		Ex 7(2.1 wt%) 200 C		Ex 7(2.1 wt%) 190 C	
Time hrs	% gel	Time hrs	% gel	Time hrs	% gel
0	-	0	-	0	-
2	23.21	2	22.4	2	21.3
4	35.24	4	33.0	4	32.8
6	44.97	6	42.0	6	40.9
8	46.66	8	44.8	8	43.2

Figure 1 illustrates Die swell ratio (1) obtained data at various shear rates (2) measured at barrel temperature of 275°C using Capillary Rheometer (Die diameter = 1 mm and Die Length = 10 mm). Die swell ratio obtained at various shear rates measured at barrel temperature of 275°C for control sample of Example 2 (i.e. without additive), polyester comprising 2.1 wt % branching agent of Example 7 and polyester comprising 3.2 wt % branching agent of Example 8 is indicated by the numbers 3, 4 and 5 respectively. Die swell of the polyester increased with increase in concentration of branching agent (Figure 1), this suggests the increased chain entanglement with increased branching. The increased branching is seen by the increase in polydispersity as seen in Table 5 and drop in crystallization temperature as seen in Table 6. DSC data also show decrease in rate of crystallization as the branching increases for the samples. Figure 2 illustrates the build up of IV (7) for the polymer chips with time (6) during the SSP at 200°C. 8 indicates the build up of IV (7) for the polymer chips of Example 5 when polymerized in solid state at 200 °C at different time interval. The IV build up data for the corresponding chips without any branching agent (Example 2) is indicated by 10 and with conventional branching agent pentaerithritol (Example 9) is indicated by 9 in the figure 2. The results show that increase of SSP rate for the polymer of Example 5, indicating that the present branching agent has increased the solid state polymerization (SSP) rate, at 200 C,

where as with the conventional branching agent like pentaerithritol the SSP rate is slower and when the chips are heated beyond 6hrs at 200 °C the chips of Example 9 got gelled.

Table 5: Polydispersity data from GPC analysis:

Sample	Polydispersity
Control (Ex 2)	2.35
Ex 3 (0.21 wt%)	2.30
EX 4 (0.42 wt %)	2.40
Ex 5 (1.02 wt %)	2.50
EX 7 (2.1Wt %)	3.08
Ex 8 (3.2wt %)	3.60

5

Table 6: Crystallization temperature data from DSC analysis

Sample	Crystallization temperature (°C)
Control (Ex 2)	216
Ex 3 (0.21 wt%)	221
EX 4 (0.42 wt %)	222
Ex 5 (1.02 wt %)	216
Ex 7 (2.1%)	208
Ex 8 (3.2%)	197

CLAIMS

1. A composition of matter for making thermoplastic polyester with controlled branching or even with linear structures comprising of branching agents containing branching agent in its backbone.
2. The polyester with controlled branching as claimed in Claim 1, where in the branching agent contains at least one trifunctional carboxylic acid or anhydride, one diol selected from monoethylene glycol, 1,3 propane diol, 1,6 hexane diol or 1,4 butane diol and one mono alkyl alcohol having carbon chain length C1- C25 selected from butanol, hexanol, octanol, nonanol or highly substituted branched aliphatic or aromatic alcohol selected from isobutanol, isooctyl alcohol, iso dodecyl alcohol, neopentyl alcohol or benzyl alcohol.
3. Polyester of Claim 1 having the controlled branching agent comprising of at least one trifunctional carboxylic acid or anhydride, one diol selected from monoethylene glycol, 1,3 propane diol, 1,4 butane diol or 1,6 hexane diol and one mono alkyl alcohol having carbon chain length C1- C25 in the range 0.1 wt% to 10 wt%, preferably between 0.2 – 2 wt%
4. The polyester with controlled branching as claimed in claim 1, wherein thermoplastic polyester comprising dicarboxylic acid monomer is selected from terephthalic acid or isophthalic acid; diol monomer is selected from ethylene glycol, 1,3 propane diol or 1,4 butane diol and the branching agent comprises of contains at least one trifunctional carboxylic acid or anhydride, one diol selected from monoethylene glycol, 1,3 propane diol, 1,6 hexane diol or 1,4 butane diol and one mono alkyl alcohol having carbon chain length C1- C25
5. A process for the preparation of polymer, fiber, filament or film of polyester having controlled branching, the process comprising
 - a. Preparing the condensation product of at least one trifunctional carboxylic acid or anhydride thereof with at least one mono alkyl alcohol at temperature upto 260 °C and under pressure upto 2 kg/cm²
 - b. Further reacting with one diol selected from monoethylene glycol, 1,3 propane diol, 1,6 hexane diol or 1,4 butane diol at temperature upto 250°C and at pressure upto 2 kg/cm² to form an adduct
 - c. Isolating the resulting product by evaporation

- d. Preparing a slurry of dicarboxylic acid selected from pure terephthalic acid or isophthalic acid and diol selected from monoethylene glycol, 1,3 propane diol or 1,4 butane diol
 - e. adding the adduct prepared in step "c" above into the slurry of step "d" followed by oligomerisation of the above mixture in presence of catalyst Sb_2O_3 under nitrogen pressure of 1.5-1.8 kg /cm² and at temperature 255-260°C
 - f. polycondensing the oligomer in vacuum at temperature in the range of 280-285 °C to obtain polyester polymer and draining the polymer into strands followed by cutting them into chips
 - g. Crystallising the amorphous chips at temperature in the range of 100-200 °C to obtain crystalline polymer
 - h. Solid state polymerizing the crystalline polymer in nitrogen atmosphere at 190-210 °C to obtain chips of high molecular weight and are extruded and melt spun into filament or fiber or extruded into films .
6. Polyester with controlled branching as claimed in claim 1 with increased rate of Solid state polymerization
 7. Polyester with controlled branching as claimed in Claim 1 with reduced rate of polymer crystallizability.
 8. Polyester with controlled branching as claimed in Claim 1 with reduction in the rate of gelation in solid state
 9. Polyester with controlled branching as claimed in Claim 1 having capability to allow fabrication of extruded films and post process curable articles.

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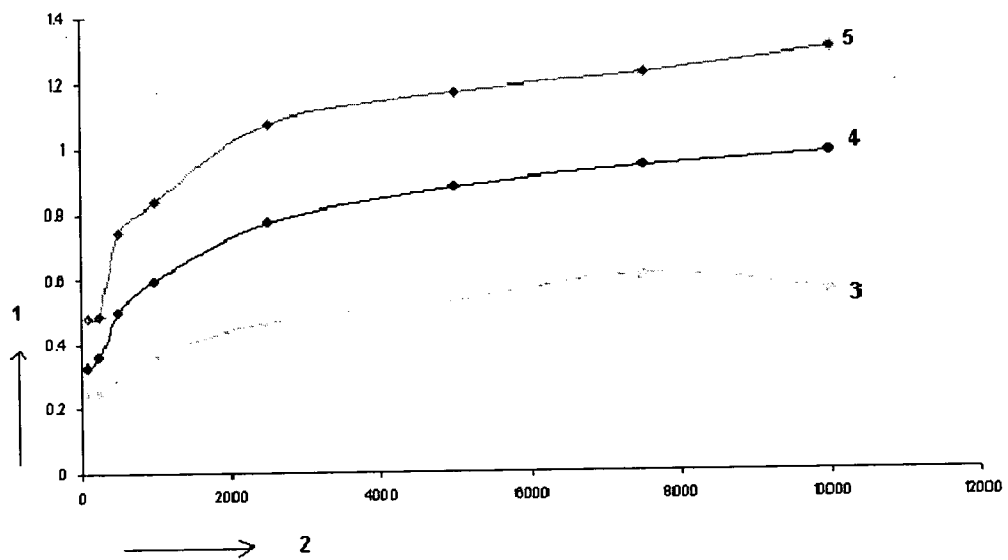


Figure 1

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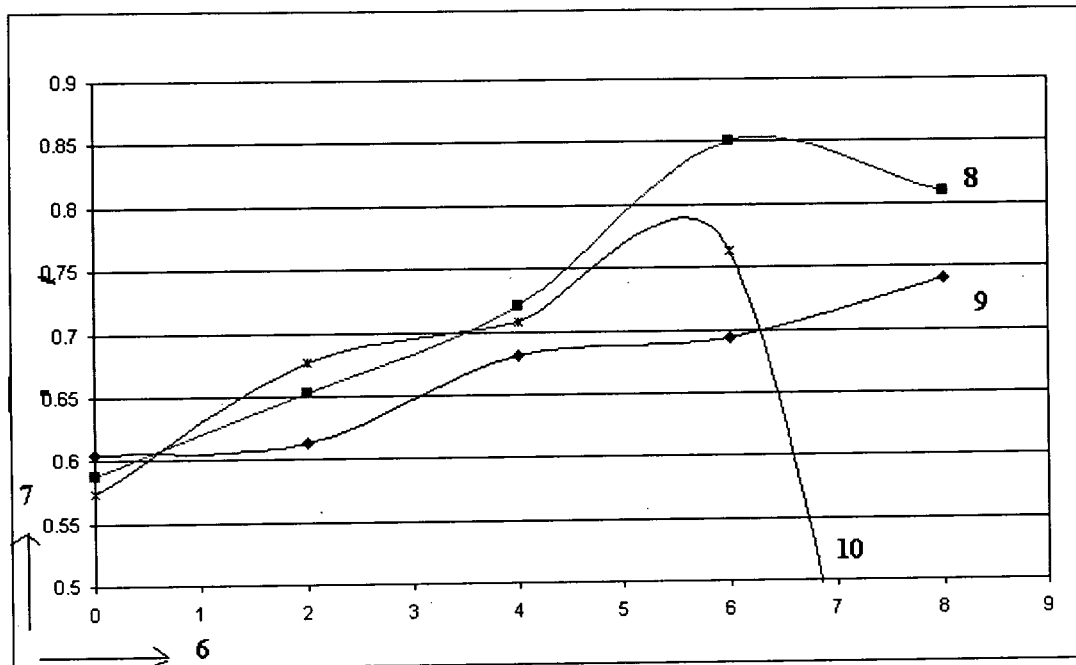


Figure 2