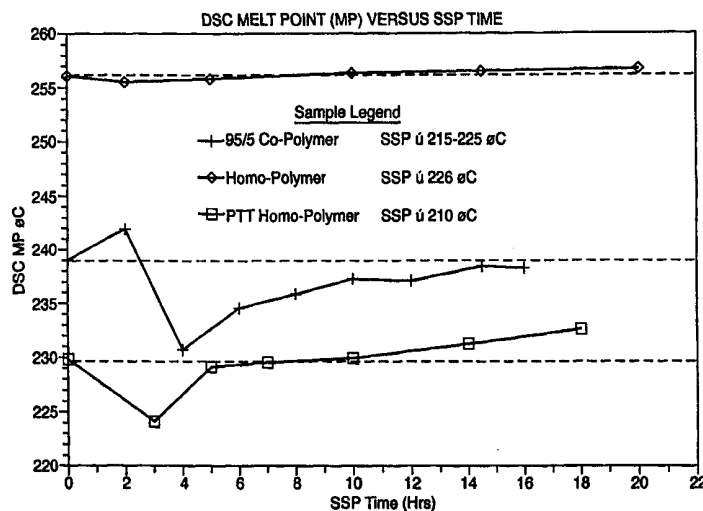




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(54) Title: PROCESS TO PREPARE A POLYESTER RESIN



(57) Abstract

Accordingly, the invention provides a process to prepare a polyester resin having an acetaldehyde concentration of no more than about 1 ppm, which process comprises the steps of: a) reacting ethylene glycol with terephthalic acid or an ester thereof, and isophthalic acid or an ester thereof under constrained reaction conditions effective to prepare a melt phase polymer having an IV in the range of 0.38 dl/g to 0.52 dl/g and a diethylene glycol content of no more than 5 mole% (based on the diol content of the resin); b) recovering the melt phase polymer from step a) as the pre-polymer used in step c); and c) subjecting at least a portion of the pre-polymer recovered in step b) to elevated temperatures over a period of time to elevate the IV to a range of 0.60 dl/g to 0.90 dl/g. The resultant resin can be transformed into a bottle preform, which, when blown, forms a bottle suitable for packaging of mineral water.

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PROCESS TO PREPARE A POLYESTER RESIN

FIELD OF THE INVENTION

This invention relates to process for preparing a polyester resin having a low acetaldehyde (AA) content. The resin is useful in packaging of mineral water or other beverages where AA content must be kept below certain predetermined levels.

BACKGROUND OF THE INVENTION

The background of this invention recognizes that AA content in polyester resins is an important factor when the end-use of the resin is concerned. Generally, resin manufacturers wish to produce a resin with as low an AA content as possible within the economics of a continuous commercial process.

Bottle grade polyester resins are typically prepared by melt phase polymerization, wherein the monomers are linked to form a "prepolymer" or "melt phase polymer", followed by the preparation of pellets of the melt phase polymer, and by solid state polymerization (SSP) of these pellets until a polyester resin of sufficiently high viscosity index (IV) is obtained.

However, traditional melt-phase polymerization entrains undesirable products and unreacted monomer components. AA, a noxious by-product of the melt polymerization process, can spoil the flavor and aroma of contained beverages, especially mineral water. Producers of containers for drinking water and soft-drink containers desire an AA content of no more than 1 $\mu\text{g}/\text{l}$, (i.e., ≤ 1 parts per billion), evolved into the headspace of an empty container. To achieve such a low AA content container, the SSP-ed resin AA content should not exceed 1 part per million (ppm).

Multi-layer containers can also be used to avoid the problem of by-product AA contamination of drinking water and beverages. Each layer is composed of polymers with specific properties. Such containers are more expensive to make and raise concern with recycle of post consumer containers.

In U.S. Patent 4,340,721, a polyester is disclosed having a low AA content. The intrinsic viscosity of the melt condensate is limited to 0.75 to 0.90 of the maximum possible IV, i.e. of about 0.6 dl/g. A further reduction in the AA content, however, remains desirable.

Finally, in U.S. Patent 4,154,920 a pre-polymer is maintained at 0.1 to 0.3 dl/g prior to SSP. When one starts at this level of IV as the feed material to the SSP process, the resin becomes difficult to pelletize.

SUMMARY OF THE INVENTION

This invention provides a process for preparing in commercial quantities polyester resins having unique properties that allow for low melt temperature processing and concomitant slow rate of acetaldehyde reformation. Accordingly, the invention provides a process to prepare a polyester resin having an acetaldehyde concentration of no more than about 1 ppm, which process comprises the steps of:

- a) reacting ethylene glycol with terephthalic acid or an ester thereof, and isophthalic acid or an ester thereof under constrained reaction conditions effective to prepare a melt phase polymer having an IV in the range of 0.38 dl/g to 0.52 dl/g and a diethylene glycol content of no more than 5 mole% (based on the diol content of the resin);
- b) recovering the melt phase polymer from step a) as the pre-polymer used in step c); and
- c) subjecting at least a portion of the pre-polymer recovered in step b) to elevated temperatures over a

period of time to elevate the IV to a range of 0.60 dl/g to 0.90 dl/g.

This results in a resin (and preform and bottle made therefrom) having a desirable advantageous AA content. The resin from the SSP process also has a more favorable melt point (MP) for making bottle preforms and an unexpectedly lower rate of AA reformation rate when measured at 280 °C. This reformation rate is equal to 70 ppm to 190 ppm AA per hour measured at 280 °C based on ASTM D-4509 at 10, 20, and 30 minutes, respectively, and extrapolated to a one hour time interval. Polyester resins according to the present invention indeed achieve a reformation rate that is equal to 100 ppm to 160 ppm AA per hour measured at 280 °C based on the same analytical procedure.

DETAILED DESCRIPTION OF THE INVENTION

The resin is prepared by a combined melt polymerization/solid state polymerization process. This process captures a melt phase polyester resin having a relatively low intrinsic viscosity which is then solid state polymerized (SSP-ed) to a finished IV. This process results in low incipient AA content in the resin bottle preform and its bottle. Such low intrinsic viscosity can be achieved by selecting proper melt phase polymerization conditions in combination with the incorporation of glycols and/or acid co-monomers, with isophthalic acid being the preferred co-monomer. Lower temperature melt extrusion-preform injection molding and reduced shear rate effects combine to reduce AA generation. The resin has properties which combine a low incipient AA content (no more than 1.0 ppm), preferably lower than 0.50 ppm and more preferably lower than 0.24 ppm, and lower melt processing temperature (≤ 252 °C) to produce a bottle having a maximum AA content of 1 $\mu\text{g/l}$.

In a preferred embodiment, the monomers that may be used in the production of the polyester resin consist of terephthalic acid (TPA) or an ester thereof, and isophthalic acid (IPA) or an ester thereof; and ethylene glycol (EG) and/or diethylene glycol (DEG).
5 Copolymerization of these comonomers (optionally with other comonomers) results in the preparation of a poly(ethylene terephthalate/isophthalate) (PETI) polyester.

10 The amount of IPA used in the preparation of the polyester resin is preferably in the range of 1-10 mole% IPA (based on the total acid component of the resin), while 1.5 to 6.6 mole%, better still 3.0 mole% to 5.5 mole% IPA is even more preferred and about 5 mole% is
15 most preferred. The preferred concentrations of EG and DEG are respectively 99-95 mole% and 1-5 mole%, with 96.7-97.5 mole% and 2.5-3.3 mole% being more preferred, based on the total diol component of the resin. This composition lowers the initial DSC melting point (MP) of
20 the polyester resin to a maximum of 245 °C. Other co-monomers, such as naphthalene dicarboxylic acid and/or propylene glycol may also be incorporated into the polyester resin.

Melt extrusion-injection molding of the SSP-ed resin
25 can be accomplished using an antimony triacetate catalyst followed by a phosphoric acid stabilizer and appropriate color toners. Alternatively, the process may employ a phosphoric acid stabilizer followed by cobalt acetate tetra-hydrate color toner and antimony ethylene glycolate
30 catalyst. Instead of cobalt acetate tetra-hydrate, color toning dyes can be added during the melt polymerization or in the melt extrusion-injection molding step.

This process allows melt extrusion-injection molding processing at temperatures up to 252 °C, low enough to
35 reduce AA generation for the production of polyester

preforms, yielding containers with AA concentrations of no more than 1.0 µg/l. This process also allows the resin to maintain properties required for satisfactory container performance.

5 The melt phase polymer so produced and captured herein has properties that make it uniquely useful for preparation of low AA content containers employing melt processing steps. Figure 1 shows how resin produced from a 95/5 PETI formulation described herein yields a lower
10 initial DSC melting point after SSP than other resin compositions.

 The DSC melting point behavior of a normal PET resin shows a drop in the melting point after initial melting. Thereafter, the melting point increases. Eventually, the
15 measured DSC melting point exceeds the melting point at the beginning of SSP. The resin of the present invention shows a drop in the DSC melting point after the initial SSP sample. While the melting point increases thereafter, the melting point of the resin does not rise above the
20 initial melting point on prolonged heating.

 The effect of the lower temperature melt point is not present in homopolymers (comparison is made with PET and PTT). Without a co-monomer present, the melting point rapidly exceeds the initial melting point raising the
25 melt process temperature requirement. Thus, addition of the co-monomer suppresses the melt point increases over the initial melt point, resulting in lower melt process temperature requirements. The resulting resin has a lower AA generation rate and produces lower AA content in the
30 bottle preform and bottle.

 The constrained reaction conditions referred herein before include a temperature of from 260 °C to 290 °C, a pressure of from 480 kPa (initial pressure) to full vacuum (typical final pressure of about 0.1 kPa) and a
35 time of reaction of from 5 to 6 hours.

It is significant to constrain the melt polymerization reaction so that the IV range of the melt phase polymer is held between 0.38 dl/g to 0.52 dl/g, and preferably 0.40 dl/g to 0.50 dl/g, and most preferably at 0.46 dl/g \pm 0.02 dl/g. It is also important that the amount of diethylene glycol content in the copolymer be 5 mole% or less. The diethylene glycol (DEG) may be prepared in-situ or it may added, but it is significant that the content be 5 mole% or less regardless of how the DEG is generated. The melt phase polymer (pre-polymer) will have an AA content of from 15 ppm to 45 ppm.

The molecular weight of the melt phase polymer is not suitable for such uses as the manufacture of beverage bottles. Thus, the melt phase polymer must be SSP-ed to achieve the IV necessary for bottle grade manufacture, such as an IV of 0.60 dl/g to 0.90 dl/g, typically 0.65 to 0.80 dl/g, or preferably about 0.74 dl/g.

The SSP process of this invention can include a crystallization step followed by a solid phase polymerization step to elevate the IV of the resin. The crystallization conditions include a temperature of from 100 °C to 150 °C. The solid phase polymerization reactor (polycondensation) may be operated at temperatures between 200 °C and 232 °C for a period of time sufficient to raise the IV to the desired level. The time may range from about 15 to 21 hours. The preferred polycondensation temperatures include a temperature of from 215 °C to 232 °C. It is significant in this invention that when starting with an IV of 0.38 dl/g to 0.52 dl/g, in the melt phase, the solid phase polymerization reaction can transpire over a longer period of time, thereby reducing resin incipient AA content.

The crystallization and polymerization phase can be performed in a tumbler dryer reaction in a batch-type system or they can comprise one or more vessels in a

continuous solid state process whereby the polymer flows from vessel to vessel after its predetermined treatment in those vessels.

5 The resins produced in this invention by the SSP process may then be converted to a preform of a bottle, which may be subjected to heat over a period of time, and stretch-blown or blown into a bottle for ultimate consumer use. The AA content in the preform will be higher than that in the polyester resin it is made of.
10 Using the polyester resin according to the present invention, preforms are achievable having an AA content of no more than about 2.5 ppm. Preform production is conducted at a temperature of from about 245 °C to about 280 °C. The bottle will possess an AA content of less
15 than 1 µg/l. The expression IV used in this specification refers to the intrinsic viscosity as determined by measuring the viscosity of a dilute solution of the polymer using a 60/40 (w/w) phenol/ tetra-chloroethane or suitable solvent system and using the Billmeyer equation
20 to calculate the IV. This procedure is based on ASTM D-4603.

The following Examples are illustrative of the invention herein claimed and are not presented as a limitation upon the scope of the claims.

25 Examples 1-5

The following Examples demonstrate the fact that different resin formulations result in different manufactured resin. Five resins were prepared using the mole percent of monomers as follows:

Example No.	Composition (mole %)		
	EG + DEG	IPA	TPA
1. Resin A	96.2 + 3.8	0	100
2. Resin B	98.0 + 2.0	2	98
3. Resin C	97.4 + 2.6	2.6	97.4
4. Resin D	95.5 + 4.5	5	95
5. Resin E	95.1 + 4.9	5	95

In Examples 4 and 5, the resins were compared to commercially available resins (Examples 1-3). The following quantities of EG (24.9 kg), PTA (55.8 kg) and IPA (3.0 kg), were loaded in a batch reactor and heated for a period of 5 to 6 hours at temperature conditions of 265 °C to 283 °C. A modified heel process was used in an effort to produce as low a DEG content as possible. Resins A, B, and C, were conventionally prepared using standard melt phase polymerization techniques to obtain an IV of 0.60 ± 0.02 dl/g, resulting in a melt point of 252-253 °C. For Resin D, the melt phase reaction was constrained to an IV of 0.46 ± 0.02 dl/g and the IPA was adjusted to arrive at a melt point no greater than 245 °C. For Resin E, the IV was 0.60 ± 0.02 dl/g. Resin E was incorporated to show the effect of IV on AA content if the IV was not constrained to 0.46 dl/g, but conventionally prepared to 0.60 dl/g. The AA content in these melt phase products were as follows:

Resin No.	AA (ppm)
Resin A	40-60
Resin B	40-60
Resin C	40-50
Resin D	26
Resin E	49

Examples 6-10

In Examples 6-8, the melt phase Resins of Example 1-3 were SSP-ed at temperatures around 230-235 °C for a sufficient period of time to arrive at an IV of 0.72-0.74 dl/g. In Example 9, Resin D, was SSP-ed at 225-228 °C for 21 hours to reach 0.74 dl/g IV. In Example 10, Resin E was SSP-ed at 225 °C to 227 °C for 9 hours to reach a 0.747 dl/g IV. The resins were tested for AA content at 200 °C based on the ASTM D-4509. The Resins were observed to have the following AA content:

Resin No.	AA (ppm)
Resin A	1.10
Resin B	0.48
Resin C	0.75
Resin D	0.24
Resin E	1.15

The data supports the fact that by constraining Resin D IV to 0.46 dl/g versus Resin E IV of 0.60 dl/g yields a substantially lower AA content after SSP.

Examples 11-15

The Resins of Examples 1-5, SSP-ed in Examples 6-10, were converted to bottle preforms and bottles by the following sequence:

1. Dry applicable Resin at 150-170°C for 8-16 hours
2. Extrusion processing of applicable Resin for injection molding of the respective preforms
3. Preform to bottle conversion by stretch-blow molding operation using Infra-Red (IR) re-heating

The preform temperatures and resultant AA content, as determined in Examples 11-15, were as follows:

Preform from Resin No.	Temp (°C)	AA (ppm)
Resin A	266	7.5
Resin B	270	7.5
Resin C	269	8.8
Resin D	250	2.0
Resin E	245	2.3

Examples 16-20

The bottle preforms made in Examples 11-15 were made into 0.6 L (20 oz.) bottles at temperature and blowing conditions as follows:

Preform from Resin No.	Temp (°C)	Bottle AA, µg/l
Resin A	280	3.2
Resin B	250	2.4
Resin C	280	3.3
Resin D	250	0.8
Resin E	245	1.1

5 Even though all attempts were made, including using the lowest preform processing temperature, to suppress AA generation Resin E exceeded the 1 µg/l maximum AA limit. Resin D, prepared from 0.46 dl/g IV melt feed resin, even when processed at 5 °C higher preform process temperature
 10 yielded a lower AA content than the 1 µg/l AA limit.

The results of Examples 1-20 can be summarized in chart form as set out in the Table.

Examples 21-24

15 Resins were prepared in these Examples to show the relationship of DEG content in the TPA/IPA copolymer to acceptable levels of AA in a blown bottle. Four series of resins were prepared using melt polymerization where the melt reactor was constrained to produce an IV of 0.46 dl/g. The Series of Resins were made under the
 20 following formulations:

Resin

- F 100-97.5/2.5 EG-TPA/IPA Copolymer. The DEG level generated is in-situ 2.2 mole% (or as low as possible). IPA content increased as necessary to achieve a maximum MP of 245 °C. Added additives comprised 0.210 parts per thousand (ppt) antimony triacetate, followed by phosphoric acid (0.015 ppt P) and appropriate color toners (0.001 ppt ESTOFIL blue, 0.0005 ppt ESTOFIL red).
- G 100-96/4 EG-TPA/IPA Copolymer modified with the appropriate amount of IPA to achieve a maximum MP of 245 °C MP. A polyester resin was produced having a yellow color index value b* of -2 (determined on a HunterLab Color Spectrophotometer). The process employed a phosphoric acid stabilizer (0.045 ppt P) followed by cobalt acetate tetra-hydrate (0.050 ppt Co) and antimony oxide (0.210 ppt).
- H EG/DEG -TPA/IPA Copolymer modified with one half the amount of IPA determined in Series G and the DEG is varied to achieve a maximum MP of 245 °C and a target b* color of -2 using cobalt. All the other ingredients were the same as used Resin G.
- I EG/DEG - TPA Copolymer modified with the appropriate amount of DEG to achieve a target 245 °C MP. The b* color was adjusted via cobalt.

Examples 25-29

Resins F-I were prepared in the melt phase to a melt point of 245 °C and an IV of 0.46 dl/g as performed with Resin D in the former set of Examples. The SSP of Resins F, G, H and I was terminated when an IV of 0.74 dl/g was

obtained. The AA content was determined to be 0.21-0.24 ppm at 200 °C. The SSP-ed resins were made into bottle preforms and then blown into 0.6 L (20 oz.) bottles. The following table shows the AA content of the 0.6 L bottles.

5

TABLE
Resin Composition and Properties versus Bottle AA content

Experimental Resin ID	Composition					Feed Resin			SSP Resin		Preform	Bottle
	EG	DEG	IPA	TPA	(Mole%)	IV, dl/g	DSC MP, °C	IV, dl/g	AA, ppm	Process Temp. °C		
A	96.2	3.8	0.0	100		0.60	252	0.73	1.10	266	7.5	3.2
B	98.0	2.0	2.0	98.0		0.60	252	0.74	0.48	270	7.5	2.4
C	97.4	2.6	2.0	98.0		0.60	253	0.72	0.75	269	8.8	3.3
D	95.5	4.5	5.0	95.0		0.46	243	0.74	0.24	250	2.0	0.8
E	95.1	4.9	5.0	95.0		0.60	242	0.74	1.15	245	2.3	1.1
F	97.5	2.5	5.5	94.5		0.46	245	0.74	0.21	250	2.3	1.0
G	96.7	3.3	4.8	95.2		0.46	245	0.74	0.24	252	2.3	1.0
H	94.1	5.9	2.3	97.7		0.46	245	0.74	0.22	250	2.5	1.2
I	92.0	8.0	0.2	99.8		0.46	245	0.74	0.21	252	3.9	1.9

Surprisingly, due to the high level of DEG content in Resins H and I, these resins could not be processed into bottles having an AA content of 1.0 µg/l or less. It is postulated that the reduced thermal stability caused by the DEG levels of greater than 5 mole% resulted in the elevated AA content in the blown preform.

C L A I M S

1. A process to prepare a polyester resin having an acetaldehyde concentration of no more than about 1 ppm, which process comprises the steps of:

5 a) reacting ethylene glycol with terephthalic acid or an ester thereof, and isophthalic acid or an ester thereof under constrained reaction conditions effective to prepare a melt phase polymer having an IV in the range of 0.38 dl/g to 0.52 dl/g and a diethylene glycol content of no more than 5 mole% (based on the diol content of the
10 resin);

b) recovering the melt phase polymer from step a) as the pre-polymer used in step c); and

15 c) subjecting at least a portion of the pre-polymer recovered in step b) to elevated temperatures over a period of time to elevate the IV to a range of 0.60 dl/g to 0.90 dl/g.

2. The process of claim 1 wherein the terephthalic acid or the ester thereof, is present in an amount of 99 mole% to 90 mole% and the isophthalic acid or the ester
20 thereof, is present in an amount of 1 mole% to 10 mole%, based on total acid component of the resin.

3. The process of claim 2, where the terephthalic acid or the ester thereof, is present in an amount of about 95 mole% and the isophthalic acid or the ester thereof,
25 is present in an amount of about 5 mole%.

4. The process of claim 1 where the constrained reaction conditions include a temperature of from 260 °C to 290 °C, a pressure of from 480 kPa to full vacuum (0.1 kPa) and a time of reaction of from 5 to 6 hours.

30 5. The process of claim 4, wherein the temperature, pressure and time are maintained to constrain the IV of

- 16 -

the melt phase polymer to a range of 0.40 dl/g to 0.50 dl/g.

6. The process of claim 5, wherein the melt phase polymer contains from 15 ppm AA to 45 ppm AA.

5 7. The process of claim 1 wherein the pre-polymer treatment in step c) comprises a temperature of 200 °C to 232 °C over a period of time of from 15 to 21 hours.

8. The process of claim 7, wherein the conditions include a crystallization temperature of from 100 °C to 150 °C and a subsequent SSP temperature of 215 °C to 232 °C.

9. A process to prepare a beverage container having an AA content of no more than 1 µg/l which comprises:

15 a) reacting ethylene glycol with terephthalic acid or an ester thereof, and isophthalic acid or an ester thereof under constrained reaction conditions effective to prepare a melt phase polymer having an IV in the range of 0.38 dl/g to 0.52 dl/g and a diethylene glycol content of no more than 5 mole% (based on the diol content of the resin);

20 b) recovering the melt phase polymer from step a) as the pre-polymer used in step c);

25 c) subjecting at least a portion of the pre-polymer recovered in step b) to elevated temperatures over a period of time to prepare a polyester resin having an IV in the range of 0.60 dl/g to 0.90 dl/g;

d) forming a beverage container preform from at least a portion of said resin; and

30 e) heating and blowing said preform of step d) at conditions to form said beverage container having an AA of no more than 1 µg/l.

10. A bottle preform and/or beverage container made from said polyester resin made by the process of claim 1.

Fig.1.

