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बौद्धिक सम्पदा भवन, मुंबई - ४००३७

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## THE PATENTS ACT, 1970

IT IS HEREBY CERTIFIED THAT, the annex is a true copy of the Patent Application and complete Specification of the applicant, Garware Polyester Ltd., An Indian Company, Garware House, 50-A, Nityananda Marg, Western Express Highway, Vile Parle, Mumbai 400 057, and as granted by this Patent in respect of Patent Application No.316/MUM/2006 and notified under Serial No.220855.

This certificate is issued under the powers vested in me under Section 147 (1) of the Patents Act, 1970. ....

Dated this 25<sup>th</sup> day of August 2008.

(A.T. PATRE)

ASSTT.CONTROLLER OF PATENTS & DESIGNS.

<b>FORM 1</b> <b>THE PATENTS ACT 1970</b> <b>( 39 OF 1970 )</b> <b>&amp;</b> <b>The Patents Rules, 2003</b> <b>APPLICATION FOR GRANT OF PATENT</b> <b>( See section 7,54 &amp; 135 and rule 20 (1) )</b>		<b>( FOR OFFICE USE ONLY )</b>  <b>Application No. :</b> <b>Filing Date :</b> <b>Amount of Fee Paid :</b> <b>CBR No. :</b> <b>Signature :</b>	
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<b>3. TITLE OF THE INVENTION</b> "High Heat Shrinkable Polyester Film"			
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<b>5. PRIORITY PARTICULARS OF THE APPLICATION ( S ) FILED IN CONVENTION COUNTRY</b>			
Country	Application Number	Filing Date	Name of the Applicant
NA			
<b>6. PARTICULARS FOR FILING PATENT COOPERATION TREATY ( PCT ) NATIONAL PHASE</b>			
NA			
<b>9. DECLARATIONS :</b>			
<b>(i) Declaration by the Inventor (s)</b> We, the above named inventor(s) are the true & first inventor(s) for this invention and declare that the applicant(s) herein is our assignee.			
(a) Date			
(b) Signature (s)			
(c) Name(s) SHASHIKANT BHALCHANDRA GARWARE			
(a) Date			
(b) Signature (s)			
(c) Name(s) MOHAN SITARAM ADSUL			
<b>10. Following are the attachments with the application :</b>			
(a) Complete specification.			
(b) Complete specification (in conformation with the international application )/as amended before the International Preliminary Examination Authority (IPEA), as applicable (2 copies ). No. of pages 263 No. of claims 215			
(c) Drawings ( in conformation with the international application ) / as amended before the International Preliminary Examination Authority ( IPEA ), as applicable (2 copies ). No. of sheets 11			
(d) Priority documents			

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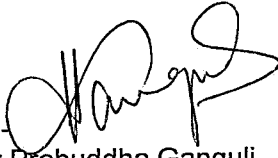
5 FEB 2008

- (e) Translation of priority document / specification/ International Search Report
- (f) Statement and undertaking on Form 3
- (g) Power of Authority
- (h) Declaration of inventorship on Form 5
- (i) Sequence listing in electronic form (floppy disc)
- (j) .....

Fee Rs. 24000 in Cheque bearing no 81891 dated 06/03/06. On Bank.

I/We hereby declare that to the best of my/our knowledge, information and belief the fact and matters stated herein are correct and I/We request that a patent may be granted to me/us for the said invention.

Dated this seventh 1<sup>st</sup> day of February 2006

Signature :   
Name : Dr Prabuddha Ganguli  
Agent on behalf of applicant

To, The Controller of Patent  
The Patent Office, at Mumbai

**FORM 2**

**THE PATENTS ACT, 1970  
(39 of 1970)  
COMPLETE SPECIFICATION  
(See section 10; rule 13)**

**1. Title of the invention. - HIGH HEAT SHRINKABLE POLYESTER FILM**

**2. Applicant**

- (a) Garware Polyester Ltd
- b) Garware House, 50-A Swami Nityananda Marg,  
Western Express Highway, Vile Parle, Mumbai  
400057, India
- (c) An Indian Company.

The following specification particularly describes the nature of this invention and the manner in which it is to be performed

## Field of Invention:

The present invention relates to process for making high heat-shrinkable polyester film without drying the polyester granules prior to extrusion. The polyester film prepared by the process of this invention is capable of being used for covering containers including labeling plastics / glass bottles, containers, batteries / electrolytic condenser.

## Background and Prior Art

Heat shrinkable film used as wrapping or labeling material is required to exhibit high shrinking and sealing properties as well as good printability, transparency, mechanical properties and resistance to chemicals, heat, weather resistance.

Conventional heat-shrinkable films formed of polyvinyl chloride or polystyrene such as polyvinyl chloride is not suitable for recycling due to their poor thermal stability. Further such materials are not environment friendly as they create polluting products on incineration. It is also observed that polystyrene and polyethylene films suffer from poor printability and therefore need special purpose expensive inks.

In contrast heat-shrinkable films formed of polyethylene terephthalate (PET) demonstrate acceptable heat and chemical resistance, weather stability and shrinking properties. High shrinkage ratio of the heat-shrinkable polyester film is generally very high resulting in non-uniform shrinking due to temperature variations in shrinkage tunnel or at the surface of container to be wrapped.

Japanese patent publication numbers 63-139725, 7-53416, 7-53737, 7-216107, 7-216109 and 9-254247 mentioned in US patent 6,231,958 disclosed a method of adjusting the shrinkage rate to achieve uniformity in shrinkage by co-polymerizing dicarbonic component such as, terephthalic acid and an isophthalic acid, and a diol component such as ethylene glycol, 1,4-cyclohexanedimethanol, 2,2-dimethyl (1,3-propanediol) and like. However this method has inherent weaknesses as adjusting the shrinkage by co-polymerizing the polyester with glycol or dicarbonic components enhances the stickiness tendency of the film.

In conventional drying, polyester chips are first crystallised and then dried at 160° C to 170° C for four to six hours. Crystalline polyester chips do not stick to each other and there is no lump formation in the dryer; and process is continuous. The polyester chips used for said type of film are essentially amorphous and do not crystallise. Such chips have very high tendency for lump formation resulting in complex plant operational problems. Moreover it is not possible to dry the chips above 60° C to 70° C and further involve long drying times thereby increasing the cost of production. The conventional drying systems are therefore not suitable for such polyester chips drying. Attempts to treat the polyester with boiling water for crystallisation have not been helpful as it leads to hydrolytic degradation of polyester itself, thereby deteriorating the properties of the polyester and the film produced out of such polyesters. Other methods to crystallise such polyesters involve solvent treatment (like acetone, methanol etc.). These methods are hazardous and are non-eco friendly.

The currently available thermo-shrinkable film do not have adequate resistance to boiling process, retort process, etc generally used to prolong their shelf life

US 6231958 discloses a heat shrinkable polyester film comprising:

a repeating unit of trimethyleneterephthalate of 5-30% by mol;

a repeating unit of 2,2-dimethyl(-1,3-propylene) terephthalate of 5-30 % by mole of the total composition; and

a repeating unit of ethyleneterephthalate for the balance

The content of the repeating unit of trimethyleneterephthalate is in the range of 10-30% by mole and the content of the repeating unit of 2,2-dimethyl (1,3-propylene)terephthalate is in the range of 5-20% by mole. It may be noted that the US patent uses the 1,3 propane diol based polyester or trimethylene terephthalate to improve the edge bowing properties of shrinkable film, it states that atleast 5% by mole of trimethylene terephthalate is to be used (page 9, lines 28-30).

The specification of US 6231958 in column 6 lines 8-13 state that using the compositions disclosed may result in unsatisfactory thermal shrinkage properties of the film when the degree of copolymerization is too high results in a film that is too amorphous. Thus pre-crystallization, which is necessary to prevent melting and agglomeration during a drying process cannot be performed. As a result, the film must be dried at low temperatures for a long period of time. The US Patent 6231958 further discloses in column 6 lines 50 – 55 suggest that the polyester mixture may be melted and extruded for which it prefers a single screw serial random type extruder which shows excellent mixing capability. In examples 1-6, the preparation involves an essential step of pre-drying to moisture levels of 0.005% the polyester mix prior to extrusion. It may also be noted that when the product is prepared *without the addition of trimethyleneterephthalate* it was not possible to measure the properties, as it was not possible to obtain a suitable product (Table 5, comparative example 2).

It would be desirable to provide reliable process without the pre-drying step without the problems associated with the known processes as described in US 6231958 and associated prior art as it would substantially enhance the productivity of any commercial production.

It is a long-standing need of the industry to provide the processes overcoming the problems of stickiness, involving lower drying time and plant operations without interruptions resulting in quality products with high plant productivity.

### **Summary of the Invention:**

The main object of the invention is to provide process for making heat-shrinkable polyester film without pre drying the polyester granules prior to extrusion.

It is another object of the invention is to produce high shrinkable film by extruding amorphous and undried polyester without negatively impacting the mechanical, shrinkage properties of the resulting film.

It is yet another object of the invention to process the polyester without causing hydrolytic degradation during the processing.

It is yet another object of the invention to provide process to prepare high shrinkage films with high transparency.

It is yet another object of the invention to provide process to prepare high shrinkage films with good antistatic property.

It is yet another object of the invention to provide process to prepare high shrinkage films with antiblocking property.

The process of present invention does not require drying at any of the stages of polyester film production, wherein the undried polyester is directly extruded to form the film. The twin-screw extruders melt the polyester at 260° C – 280° C with venting of the moisture.

The film of the present invention is resistant to heat – sterilization by boiling or retort process and provide a good appearance of the film as well as thermo-shrunk product and therefore become suitable for packaging applications in foods, beverage, medicine etc. Further it has higher thermo-shrinking stress and better binding properties compared to PVC.

#### Detailed Description of the Invention

In the conventional method, heat shrinkable polyesters are dried at 65-70°C under dehumidified air for 24 hrs. Increasing the drying temperature beyond 70°C results in sticking of the polyester chips. If polyester is not dried then there is severe degradation of polyester during processing.

The present invention is an improvement over existing processes and comprises making heat-shrinkable polyester film without drying the polyester granules in twin screw extruder and extruding a blend of 5-10% of homopolyester and 90-95% of co-polyester at 250°C - 275°C under vacuum during the process of extrusion. The melt form is cast on a cool drum and quenched. The sheet is introduced to a group of rollers heated to about 80°C -120°C and stretched in the longitudinal direction at draw ratio of 1-2 while passing through the rollers. The uniaxially stretched film is cooled by a group of rollers at about 20-50°C. The uniaxially stretched film is inline coated with antistatic coating. The film is then stretched in the transverse direction at draw ratio of 2.5 to 7 preferably 3 to 5.5 at about 85-90°C followed by winding on jumbo rolls. The film may have regrind of 0-50%.

The PET film has a thickness of about 10 microns to about 100 microns

Surface coating of the uniaxially oriented polyester film may be achieved by roll coating, gravure coating, air knife coating, rod coating and their like, the preferable one being gravure and rod coating. Further it is possible to achieve differential coatings on two sides of the film to have one side antistatic coating and the other side with a coating for print adhesion using a two-side rod coater.

The coating liquid used is 1 to 20 g per m<sup>2</sup>, especially 2 to 12 g per m<sup>2</sup> of the running film to achieve a thickness of 0.005 to 3 micron, preferably 0.01 to 1 micron.

The antistatic coating comprises 5 wt % or more of the water-soluble conductive moieties of molecular weight 200 to 100,000, preferably 500 to 50,000 including those having at least one conductive group selected from sulfonic, sulfonate, quaternary ammonium salt, tertiary ammonium salt, carboxylic group. The water-soluble conductive compound may further contain hydroxy, amino, epoxy, aziridinyl, sulfinic, aldehyde, vinylsulfone, nitrate and phosphate

The antistatic layer preferably contains a binder resin to improve the adhesivity of the antistatic layer to the polyester film. Examples of the binder resin are polyester resin and acrylic resins. The polyester resin is linear polyester containing a dicarboxylic acid component and a glycol component as constituent components. The polyester resin may be copolymerized in addition to the above components with a component having a sulfonic acid salt to impart the resin with hydrophilicity. The acrylic resin is a polymer or copolymer composed mainly of acrylic monomers. The antistatic layer is preferably incorporated with a crosslinking agent composed mainly of a polymer having melamine group. The base film is coated with a water based coating liquid containing antistatic agent (5-100%), binder resin (0-95%), crosslinking agent (1-15%) and/or the surfactant (0-25%). The preferred antistatic agent is Stearamido propyl dimethyl-beta-hydroxy ethylammonium nitrate.

The film may also be in-line coated with anti-blocking agents, UV agents to impart specific properties such as reduced static charge, reduced coefficient of friction and have protection from UV light.

Homo Polyesters and copolyesters may be obtained by transesterification or direct esterification by known methods as is described in D.A.Schiraldi; " New Poly(Ethylene Terephthalate) copolymers" from the book "Modern Polyesters:chemistry and Technology of polyesters and copolyesters" Edited by John Scheirs and Timothy E.Long, pp.245-262. John Wiley & Sons, 2003. A melted slurry of diol or substituted diol and purified dibasic acid or with substituted diacid or with esters of diacids is heated, in the presence of a esterification catalyst, and water / methanol & excess diol are removed under vacuum leaving a residual melt of the polyester which is discharged via strand die into a cooling trough, pelletized, and then further dried to remove residual moisture. The inorganic particles slurry in diol is added prior to polycondensation

Polyesters such as dimethyl terephthalate, polyethylene terephthalate, polybutylene terephthalate, polycyclohexane dimethanol terephthalate, polyethylene alpha beta-bis (2-chloro or 2- methoxyphenoxy) ethane-4,4" dicarboxylate, polyethylene (tere/iso/ortho) phthalate including those produced by using more than one dibasic acid selected from terephthalic acid, isophthalic acid, adipic acid, orthophthalic acid, sebacic acid, naphthalene dicarboxylic acid, azelaic acid and the like and more than one diol selected from ethylene glycol, diethylene glycol, triethylene glycol, butane diol, neopentyl glycol, cyclohexane dimethanol and the like. Dicarboxylic esters are selected from dimethyl terephthalate, dimethyl isophthalate, dimethyl sebacate, dimethyl adipate, naphthalene dicarboxylate and the like.



Inorganic particles such as silicon dioxide, calcium carbonate, talc, kaolin or mixture of thereof are optionally added prior to polycondensation stage.

Inorganic particles are selected from particle sizes of 1 to 5  $\mu$  preferably 2 to 4  $\mu$  and are added in amounts 100 ppm - 1000 ppm, preferably 300 to 500 ppm. The inorganic particles are made into dispersion in a carrier liquid such as mono-ethylene glycol or optionally added into master batches with any of the polyester.

In an embodiment of the invention, homo polyester such as polyethylene terephthalate is prepared using dimethyl terephthalate or purified terephthalic acid and mono ethylene glycol in presence of manganese acetate as transesterification catalyst, and  $Sb_2 O_3$  as polycondensation catalyst, at 140-290°C and up to 1 mm pressure.

A co-polyester such as amorphous polyethylene terephthalate is obtained by transesterification of mixture of 25-35% mono ethylene glycol, 0-15% neo pentyl glycol (NPG), 0-2% di ethylene glycol (DEG), 0-7% isophthalic acid (IPA) and 0-15% 1,4 cyclo hexane dimethanol(CHDM) and 55-65% dimethyl terephthalate(DMT) in the presence of manganese acetate catalyst at 140-230°C. The polycondensation reaction is carried out preferably in presence of  $Sb_2O_2$  catalyst and triphenyl phosphate at 260-290°C up to 1 mm Hg pressure.

The base material for the film is selected from those preferably having intrinsic viscosity of 0.60 to 1.20 dl/g. One may also use a mixture of polyesters so long the polyesters used have intrinsic viscosity within the said range.

#### Measurement of Raw materials and Film Properties:

a) Intrinsic viscosity (I.V.) of the polyesters is measured in phenol and 1,1,2,2 Tetrachloroethane mixture 60:40 w/w and at 25°C using a Ubbelohde viscometer (ASTM- D 4603 for I.V. detection and ASTM-D 446 for Ubbelohde viscometer).

b) Tensile Strength and % elongation are tested with Instron-4411 Instrument. UK instrument (ASTM D-882).

c) Shrinkage is measured by immersing the sample in hot water for 30 seconds.

d) Surface Roughness is an arithmetic mean of absolute value of the profile departure within the measuring length. Stylus type surface roughness tester such as "Surfocorder Analyser AY-41 of Kosaka lab ltd is used to measure the surface roughness. The samples are mounted on the sample port and the surface is scanned by mounting stylus and Ra is measured as the surface roughness.

e) Coefficient of friction is the ratio of the frictional force, to the force, usually gravitational acting perpendicular to the two surfaces in contact. The static or starting coefficient of friction is related to the force measured to begin movement of the surfaces relative to each other. The dynamic or kinetic or sliding coefficient of friction is related to force measured in sustaining this movement. On the coefficient of friction

instrument, The sample is fixed on the test bed. Another sample is attached to the sledge; the sledge is placed on the sample of the test bed and pulled. The maximum force during starting and running is measured.

f) Surface resistivity is the surface resistance reduced to a theoretical square area, the size of the square is immaterial. It is the quotient of the normal voltage gradient and the current per unit width of the current path. Surface resistivity tester such as "Teraohmmeter (motwane) is used to measure the surface roughness. The samples are mounted between electrode and guard ring and the surface resistivity in ohms is measured by applying 500 volts.

Preparation of Polyester for blending:

Example-1:: Preparation of Homopolyester A

Polyethylene terephthalate was obtained by transesterification of Mono ethylene glycol (11.5Kg) and Dimethyl terephthalate (20 Kg). Transesterification was carried out using 0.04% Manganese acetate as catalyst based on DMT. After transesterification reaction at 140-230°C, the product i.e, bishydroxyethyl terephthalate is subjected to polycondensation in presence of 0.04% of  $Sb_2O_2$  catalyst and 0.07% triphenyl phosphate heat stabiliser at 260-290°C by gradually decreasing the pressure from 760 to about 1 mm Hg and holding the system at this stage for 3 hrs. The glycol slurry of synthetic silica 0.005 wt % to about 0.2 wt % based on the weight of the PET film, was optionally added before polycondensation. and the polyester was cast into granules. The I.V of homo polyester was 0.6-0.65.

Example-2: Preparation of Co-polyester B

A stainless steel reactor equipped with stirrer, condenser, fractionating column, nitrogen gas inlet was charged with 60.5 % dimethyl terephthalate (DMT), 28.59 % Monoethylene glycol (MEG) , 9.68 % 2,2-dimethyl (1,3-propanediol) or neopentyl glycol (NPG) and 1.23 % diethylene glycol (DEG). Manganese acetate 0.04 % on weight of DMT was used as the ester exchange catalyst and transesterification was carried out at 158-230°C in an inert atmosphere. The methanol distillate liberated during reaction was collected. The transesterified product was transferred to polymerization reactor. Antimony trioxide 0.04% and 0.07% Triphenyl Phosphate (TPP) both on the weight of DMT were added by making slurry in ethylene glycol and polymerization was carried out for 3 hours at 260-290°C by gradually reducing the pressure from 760 mm Hg to 1.0 mm Hg and then casting in the form of granules.

Example-3

Dimethyl terephthalate (DMT) 61.34 %, MEG 31.41% containing dispersed silica so that final silica content in polyester chips is 0.2%, NPG 6.56 % and DEG 0.69 % was charged in an ester interchange reactor and processed as in example-2. This was Co-polyester C.

#### Comparative Example-1 (Without silica dispersion)

Dimethyl terephthalate (DMT) 61.34%, MEG 31.41%, NPG 6.56 % and DEG 0.69 % was charged in an ester interchange reactor and processed as in example-2.

#### Example-4

DMT 60.1 %, MEG 28.42 % containing dispersed silica so that final silica content in polyester chips is 0.2%, NPG 10.81 %, DEG 0.67% was charged in a ester interchange reactor and processed for transesterification and further polymerization as mentioned in Example-2. The polyester was cast in the form of granules. This is Co-polyester D.

#### Example-5

DMT 60.51%, MEG 29.65 % containing dispersed silica so that final silica content in polyester chips is 0.2%, NPG 8.47 %, DEG 1.37 % was charged in a ester interchange reactor and processed for transesterification and further polymerization as mentioned in Example-2. The polyester is cast in the form of granules. This is Co-polyester E.

#### Example-6

DMT 59.97 %, MEG 26.69 % containing dispersed silica so that final silica content in polyester chips is 0.2%, NPG 12 %, DEG 1.34 % was charged in a ester interchange reactor and processed for transesterification and further polymerization as mentioned in Example-2. The polyester was cast in the form of granules. This is Co-polyester F.

#### Example-7

DMT 57.2 %, MEG 28.6 % containing dispersed silica so that final silica content in polyester chips is 0.2%, NPG 6.38 %, DEG 1.43 % was transesterified till complete removal of the byproduct methanol. Slurry of 6.38 % isophthalic acid (IPA) in MEG is added and esterification reaction was carried out with removal of the liberated water and then polymerized and the polyester was cast in the form of granules. This is Co-polyester G.

#### Example-8

DMT 59.07 %, MEG 27.93 % consisting of dispersed silica so that final silica content in polyester chips is 0.2%, 1,4-cyclohexane dimethanol (CHDM) 13 % are charged into the reactor and processed further as described in example-2. The polyester is cast in the form of granules. This is called Co- polyester H.

The polyesters in examples 2 to 8 exhibit intrinsic viscosity of 0.700-0.720 dl / g.  
Preparation of Blends

Several blends of Polyester A:: Polyester B; Polyester A : Polyester C; Polyester A: Polyester D; Polyester A: Polyester E; Polyester A: Polyester F; Polyester A: Polyester G; Polyester A: Polyester H; were prepared in the ratio of 10:90.

#### Antistatic coating formulations

The antistatic formulations consists of 6% Stearamido propyl dimethyl-beta-hydroxy ethylammonium nitrate agent, 10% Eastman AQ 1350 (binder resin) and 84% water.

#### Film Extrusion

A blend of 10% of homopolyester and 90% of co-polyester was extruded without drying the polyester granules in twin screw extruder at a temperature of 260°C under vacuum during the process of extrusion; The melt form was cast on a cool drum and quenched by electrostatic pinning/quenching then the sheet was introduced to a group of rollers heated to temperature of about 95°C with stretching in a longitudinal direction at a draw ratio of 1.0, cooled by a group of rollers at 38°C. The uniaxially stretched film was inline coated with antistatic coating. The film was introduced into a tenter and clips are used to clamp the edges of the film wherein the film was stretched in a transverse direction at draw ratio 4.1 at 90°C. The film was wound on a winder roll.

In addition to above blends, a blend of Polyester A and the polyester obtained from comparative example 1 was blended in twist screw extruder in the ratio of 10:90. It was found that the extruded film has blocked completely after winding the film on a roll.

The results of the experiments are presented in Tables 1 to 3.

The results in Table-1, 2 & 3 show that, the I.V. drop is higher in un-dried polyesters extruded with conventional extrusion technique resulting in deterioration of mechanical properties of the film. Higher I.V. of the film is always preferred for improved mechanical properties of the film.

Table I demonstrates that the elongation and tensile strength of the film of the present invention is in the range of 410-440% and 1430 to 1500 kg/cm<sup>2</sup> respectively.

Table 2 shows that I.V. drop is reduced and average I.V. drop is 0.06 dl/g. The mechanical properties of the film have improved having elongation from 620 to 680% and having tensile strength from 2050 to 2400 kg/cm<sup>2</sup> (Without drying, elongation is 410-440 % and tensile strength is 1430 to 1600 kg/cm<sup>2</sup>).

Table 3 also shows that the film is antistatic and coefficient of friction (static) is within limit (equal or below 0.6).

The process of the present invention obviates the need to dry the granules prior to extrusion. The film of this invention is comparable with the film manufactured by conventional extrusion by drying the resin prior to extrusion. The drop in I.V. is also less as compared to polyester processed after drying.

The heat shrinkable polyester films according to the present invention satisfy the need for excellent basic properties including transparency, printability, shrinkage properties and mechanical properties. Furthermore, the antistatic coating improves the static charge elimination on the film of this invention.

The film shrinkage property versus temperature of the film of the present invention is shown in figure -1.

The film shrinkage properties of the film of the present invention after exposing the samples at different temperatures in hot air for 30 seconds are shown in figure 2.

The surface roughness of the film made as per the blend of A and comparative example 1 is shown in figure 3.

The surface roughness of the film made as per the blend of A and B is shown in figure 4.

Table-1

## Analysis report of extrusion trials with undried polyester chips

Sr. No	Property	Polyester (A+B)	Polyester (A+C)	Polyester (A+D)	Polyester (A+E)	Polyester (A+F)	Polyester (A+G)	Polyester (A+H)
1	I.V. (dl/g) (Before extrusion)	0.706	0.710	0.708	0.701	0.704	0.712	0.716
2	I.V. (dl/g) (After extrusion)	0.541	0.551	0.539	0.528	0.540	0.539	0.542
3	Tensile strength of film (kg/cm <sup>2</sup> ) MD= TD=	409 1444	425 1530	440 1540	455 1560	435 1600	440 1475	410 1485
4	% elongation of film MD= TD=	150-200 120-130	160-180 130-135	175-205 125-130	230-260 120-135	145-164 130-145	180-195 135-155	180-200 130-145
5	% Shrinkage in boiling water for 30 sec. MD= TD=	2-5 70-74	3-5 52-54	2-4 74-78	3-4 65-68	4-5 78-80	3-4 72-76	2-4 70-74

Each extrusion trial has following compositions, Polyester-A :10%, Polyester B or C or D:90%.

I.V. = Intrinsic Viscosity

MD = Machine or Longitudinal Direction

TD = Transverse direction.

Table-2

## Analysis report of extrusion trials with dried polyester chips

Sr. No	Property	Polyester (A+B)	Polyester (A+C)	Polyester (A+D)	Polyester (A+E)	Polyester (A+F)	Polyester (A+G)	Polyester (A+H)
1	I.V. (dl/g) (Before extrusion)	0.706	0.710	0.708	0.701	0.704	0.712	0.716
2	I.V. (dl/g) (After extrusion)	0.648	0.637	0.646	0.639	0.654	0.648	0.641
3	Tensile strength of film (kg/cm <sup>2</sup> ) MD= TD=	680-710 2050- 2200	600-620 2150- 2350	630-660 2200- 2350	620-640 2180- 2400	640-665 2200- 2375	630-650 2130- 2240	640-655 2000- 2200
4	% elongation of film MD= TD=	650-710 125-140	700-740 135-155	720-750 140-160	680-720 145-155	660-690 140-160	700-710 135-150	705-740 145-155
5	% Shrinkage in boiling water for 30 sec. MD= TD=	2-4 72-75	3-6 53-56	2-4 72-74	3-4 63-66	2-5 76-78	3-5 70-72	2-4 68-71

Each extrusion trial has following compositions, Polyester-A :10%, Polyester B or C or D:90%. Drying is conducted at 70-75°C with 3.5 m<sup>2</sup> /hr/kg N<sub>2</sub> flow for 24 hrs.

Table-3

No	Property	Polyester (A+B)	Polyester (A+C)	Polyester (A+D)	Polyester (A+E)	Polyester (A+F)	Polyester (A+G)	Polyester (A+H)
1	I.V.(dl/g) (Before extrusion)	0.706	0.710	0.708	0.701	0.704	0.712	0.716
2	I.V. (dl/g) (After extrusion)	0.668	0.670	0.672	0.658	0.676	0.662	0.671
3	Tensile strength of film (kg/cm <sup>2</sup> ) MD= TD=	660-680 2200-2300	600-640 2180-2260	580-660 2210-2340	610-660 2150-2210	620-660 2240-2300	600-640 2200-2310	605-630 2250-2310
4	% elongation of film MD= TD=	680-710 125-140	720-740 135-155	690-740 140-160	700-780 145-155	740-810 140-160	720-800 135-150	740-790 145-155
5	% Shrinkage in boiling water for 30 sec. MD= TD=	2-3 72-76	3-4 52-54	2-4 70-72	3-5 60-64	2-4 76-78	4-6 70-74	3-5 67-72
6	Coefficient of friction-static	0.5	0.5	0.45	0.6	0.5	0.6	0.5
7	Surface Resistivity (ohm)	1x10 <sup>11</sup>	2x10 <sup>11</sup>	1x10 <sup>11</sup>	3x10 <sup>11</sup>	1x10 <sup>11</sup>	5x10 <sup>11</sup>	2x10 <sup>11</sup>

Extrusion in twin screw extruder (the polyester was not dried prior to extrusion)

Each extrusion trial has following compositions, Polyester-A : 10%, Polyester B or C or D:90%.



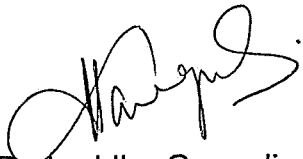
Claims:

We claim

- 1) A process for making high heat-shrinkable polyester film without drying the polyester granules prior to twin screw extrusion comprising:
  - a) extruding a polyester or a blend of 5-10% of homopolyester and 90-95% of copolyester of intrinsic viscosity 0.6-1.2 dl/g and containing 0.1-0.3% silica under vacuum up to 200 mm pressure and 250-275 °C ;
  - b) casting the melt on a cool drum and quenching;
  - c) introducing the sheet to a group of rollers heated to 80°C to 120°C, stretching in a longitudinal direction at a draw ratio of 1-2, while passing through the rollers;
  - d) cooling the film by introducing to a group of rollers at 20-50°C;
  - e) optionally inline coating the film with anti static coating;
  - f) stretching the film in transverse direction at draw ration of 2.5 to 7, preferably 3 to 5.5 at 85-90°C; followed by winding on jumbo rolls and optional adding regrind from 0-50% polyesters.
- 2) A process for making high heat-shrinkable polyester film as claimed in claim 1, wherein the homopolyesters and copolyesters are produced by using more than one dibasic acid selected from terephthalic acid, isophthalic acid, adipic acid, orthophthalic acid, sebacic acid, naphthalene dicarboxylic acid, azalaic acid and more than one diol selected form ethylene glycol, diethylene glycol, butane diol, neopentyl glycol, cyclohexane dimethanol.
- 3) A process for making high heat-shrinkable polyester film as claimed in claims 1-2, wherein the polyesters optionally contain inorganic particles such as silicon dioxide, calcium carbonate, talc, kaolin or mixture of thereof.
- 4) A process for making high heat-shrinkable polyester film as claimed in claim 3, wherein 100 ppm - 1000 ppm, preferably 300 to 500 ppm of inorganic particles of average particle sizes 1 to 5  $\mu$  preferably 2 to 4  $\mu$  are added.
- 5) A process for making high heat-shrinkable polyester film as claimed in claims 1, wherein the antistatic coating contains 5 wt % or more of the water-soluble conductive ionic compounds having atleast one conductive group selected from a sulfonic group, a sulfonate group, a quaternary ammonium salt, a tertiary ammonium salt and a carboxyl group and optionally containing a amino, hydroxy, apoxy, aziridiny, active methyl, sulphinic, aldehyde or vinyl sulfone group.
- 6) A process for making high heat-shrinkable polyester film as claimed in claims 1-5, wherein the antistatic layer preferably is polyester resin and acrylic resins.
- 7) A process for making high heat-shrinkable polyester film as claimed in claim 1-6, wherein the polyester resin is linear polyester containing a dicarboxylic acid component and a glycol component as constituent components.
- 8) A process for making high heat-shrinkable polyester film as claimed in claim 7 wherein, the polyester resin is copolymerized with a component having a sulfonic acid salt group

- 9) A process for making high heat-shrinkable polyester film as claimed in claims 8, wherein the acrylic resin is a polymer or copolymer composed mainly of acrylic monomers.
- 10) A process for making high heat-shrinkable polyester film as claimed in claims 1, 5-9, wherein the antistatic layer of 0.005 to 3 micron, preferably of 0.01 to 1 micron. is formed by coating a base film with a water based coating liquid in amounts of 1 to 20 g, preferably 2 to 12 g per m<sup>2</sup> of the running film containing antistatic agent (5-100%), binder resin (0-95%), crosslinking agent (0-15%) and/or surfactant (0-25%)
- 11) A process for making high heat-shrinkable polyester film as claimed in claims 1, 5-10, wherein the antistatic agent is stearamido propyl dimethyl-beta-hydroxy ethylammonium nitrate.
- 12) A process for making high heat-shrinkable polyester film as claimed in claim 1 – 11, wherein the film orientation is carried out by processes such as roll stretching, longitudinal stretching, tenter stretching..
- 13) A process for making high heat-shrinkable polyester film as claimed in claims 1 – 12, wherein the film is optionally pre-heated before stretching.
- 14) A high heat-shrinkable polyester film with antistatic coating comprising blend of 5-10% homopolymer and 90-95 % copolyester comprising 55-65% of DMT, 25-35% of MEG, 0-15% of NPG, 0-2% of DEG, 0-7% of IPA and 0-15% of CHDM.

March 5<sup>th</sup> 2006

  
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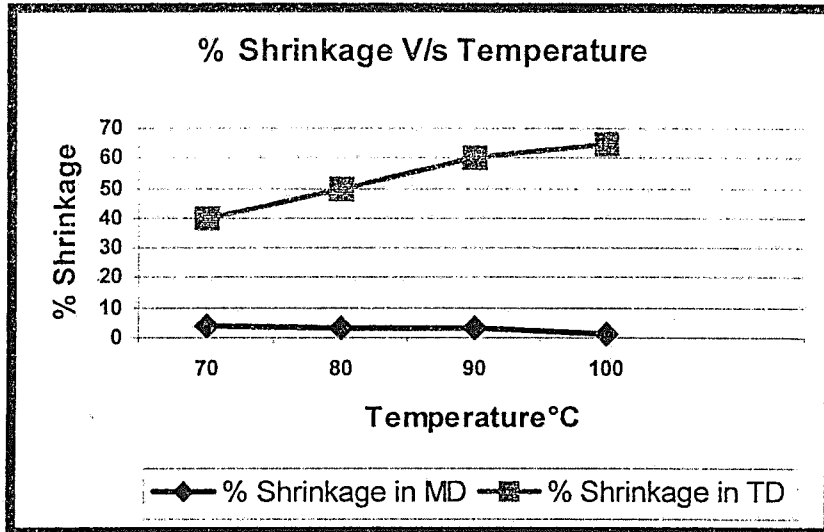


Figure 1

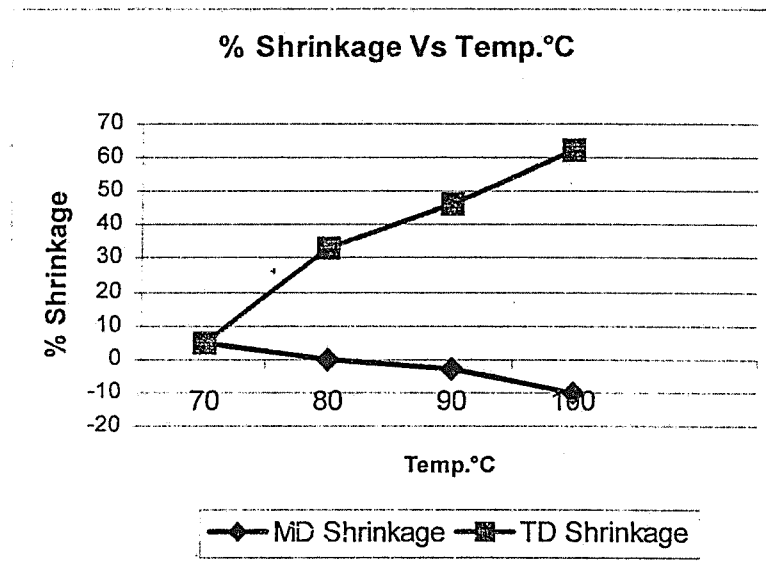
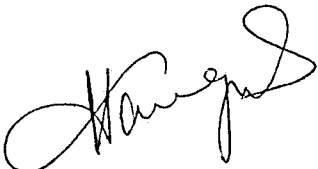
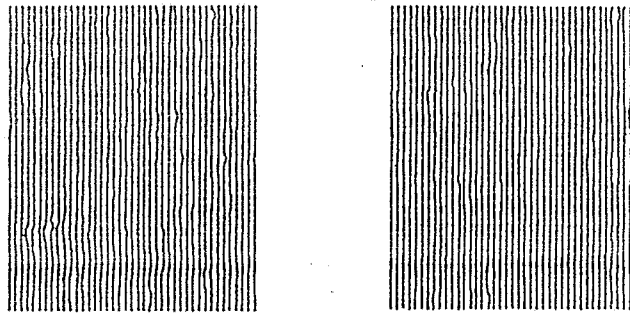


Fig 2

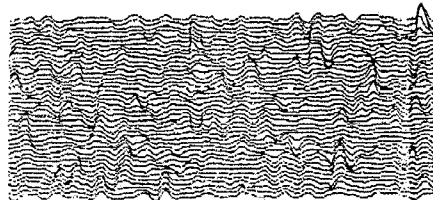
  
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Fig. 3



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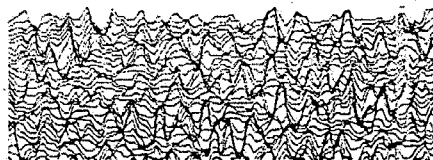
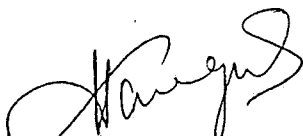


Fig 4

  
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