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- (54) Process for packaging materials sensitive to oxygen and/or to water vapour
- (57) A process for packaging materials sensitive to oxygen and/or to water vapour is disclosed in which the

packaging material is a laminate comprising two layers of thermoplastic polymers, metallised on one face and joined together by a layer of adhesive, one of the thermoplastic polymers being poly(ethylene glycol) terephthalate.

# ERRATUM

### SPECIFICATION NO 2103999A

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#### **SPECIFICATION**

# Process for packaging materials sensitive to oxygen and/or to water vapour

The present invention relates to a process for packaging materials sensitive to oxygen and/or to water vapour.

This process is characterised in that the packaging material is a laminate comprising two layers of thermoplastic polymers, metallised on 10 one face and joined together by a layer of adhesive, one of the two layers of thermoplastic polymers consisting of poly-(ethylene glycol) terephthalate.

Laminates of this type, with various thicknesses

15 for the respective constituent layers, have already been described in the literature.

French Patent 1,494,153 uses them for the purpose of obtaining flexible reflecting surfaces (projection screens, reflecting panels, cat's eyes);

20 French Patent 2,395,832 teaches the use of laminates of this type for reflecting light (parasols, greenhouses, screens on glazing); U.S. Patent 3,562,752 uses laminates of this type as an insulating material in the refrigeration industry or in aeronautics, this use being permitted in this case by the presence of a special adhesive.

Unexpectedly, it has been discovered that, when these laminates are used for packaging, they protect the products contained in a suitably sealed 30 packaging from the action of oxygen (and/or water vapour) outside the packaging.

Published European Application 26,106 describes a packaging containing two outer layers of poly-(ethylene glycol) terephthalate and two
35 inner layers of aluminium, no adhesive being interposed between the two inner layers of aluminium. However, the process for the manufacture of this laminate presents difficulties of practical and industrial production.

40 German Application 1,940,028 discloses laminates obtained by glueing a layer of thermoplastic polymer, metallised on both its faces, to another layer of thermoplastic polymer. However, the packagings obtained from such
 45 laminates have not proved totally satisfactory in preserving their contents from the action of

oxygen and water vapour.

Amongst the products which can be protected from oxygen with the aid of the process according 50 to the invention, in order to keep them stored without substantial deterioration, there may be mentioned numerous food products: solid food products, such as powdered drinks, like powdered milk, soluble coffee and soluble tea, powdered soups, waffles, salted biscuits, and salted dried fruits, like salted almonds, foodstuffs containing liquids, such as fruit as in tins, vegetables and more particularly tomatoes, oils, and drinks, such as fruit juices and wines, pharmaceutical products, in particular those which are hygroscopic, agricultural products, such as seeds for sowing,

The packaging process according to the present

and various industrial products, such as electronic

components and X-ray plates.

65 invention can also be used for the purpose of measuring the release of gaseous products from foodstuffs in the absence of contact with oxygen outside the packaging; it is thus possible to study the development of the ripening of a cheese.

70 The dimensions of the laminate can vary within fairly wide limits; however, the laminate must be sufficiently thin to be used as packaging, but also sufficiently thick to have sufficient mechanical resistance for handling. In general, the thickness of the layers of thermoplastic polymers is from 6 to 300 μm and preferably 12 to 200 μm; the metal layers typically have a thickness of 0.02 to 0.06 μm and preferably 0.03 to 0.04 μm, and the layer of adhesive typically has a thickness of 1 to 10 μm and preferably 2 to 4 μm. It can therefore be seen that the total thickness of the laminate employed according to the invention is generally from 13 to 610 μm.

It is not obligatory for the two layers of 85 thermoplastic polymer to be of the same type and of the same thickness. Likewise, it is not obligatory for the two metal layers to be of the same metal and of the same thickness. In practice, however, a metallised film is generally glued to 90 itself by the metallised layers; in this case, the layers of thermoplastic polymer are usually of identical structure and thickness and the same applies to the metallised layers. The same will apply when a metallised film is glued to itself with 95 the metal face against the face of thermoplastic polymer. In the latter case, to protect the metal layer, it is possible to apply an additional coating of thermoplastic polymer, for example polyethylene, to the outer metal layer, if necessary 100 with the aid of a polyurethane-type adhesive, for example. This additional coating can be used to obtain heat-sealable packagings.

Thermoplastic polymers which can be used include poly-(ethylene glycol) terephthalate,

105 crystalline polypropylene, high-density and low-density polyethylenes, and nylons, in particular nylon 6,6; poly-(ethylene glycol) terephthalate is normally used, preferably after biaxial stretching. It is self-evident that the polymer is chosen

110 according to the material to be packaged, so as to comply with the standards of hygiene and safety in the case of food products and pharmaceutical products.

The metal layers can comprise various metals,

such as aluminium, silver and chromium, by
themselves or in combination; for reasons of
convenience and cost price, it is preferred to use
aluminium. The metal can be deposited on the
plastic sheet by any known means, such as
vaporisation of metal in vacuo or hot transfer; in
general, it is preferred to use the method of
deposition after vaporisation of the metal in vacuo.

The adhesive between the two metal layers is generally a polyurethane: one-component or twocomponent adhesives can be used, with or without solvents.

The stability of the impermeability to oxygen and/or to water vapour of the packagings obtained is high, even after crumpling. The packagings

according to the invention therefore have a noteworthy superiority in this respect, compared with the commercial packagings of thin aluminium foil.

5 The following Examples further illustrate the present invention.

#### **EXAMPLE 1**

One of the faces of a 12 micron polyester film is metallised by vaporisation in vacuo, in accordance with the known techniques, so as to deposit 350 Å (0.035 micron) of aluminium.

After metallisation, an impermeability to oxygen varying between 0.5 and 0.8 cm<sup>3</sup>/m<sup>2</sup>/24 hours is measured on the metallised film.

A layer of commercial 2-component polyurethane adhesive, of the solvent glue type, is deposited on the metal face of this film with the air of an engraved roller of a glueing machine.
 After drying in the drier of the glueing machine,
 the pre-dried layer of adhesive is 4 microns thick.

The above metallised polyester film, coated with adhesive, is then calendered with a second metallised polyester film identical to the film described at the beginning of this example, the metal face of this film being brought into contact with the adhesive.

After calendering, the laminate obtained is rolled up; it is left to crosslink for 5 days. It comprises the following sequence: a layer of polyester, a layer of aluminium, a layer of adhesive, a layer of aluminium and a layer of polyester.

It has the following characteristics:

thickness: 28 microns

35 impermeability to oxygen:

about 0: not measurable with the commercially available measuring

instruments

impermeability 40 to helium:

5 cm<sup>3</sup>/m<sup>2</sup>/24 hours

to helium:

less than 0.1 g/m<sup>2</sup>/24

impermeability to water vapour:

less than 0.1 g/m hours

After crumpling, the laminate essentially retains these characteristics, which is not the case with aluminium foil.

#### **EXAMPLE 2**

The procedure of Example 1 is followed, but a commercial polyurethane glue of the solventless type is used, which avoids the operation for predrying the adhesive. An approximately 1 micron layer of adhesive is deposited.

The characteristics of the laminate obtained are identical to those of Example 1, and the thickness of the compound material is 25 microns.

#### 55 EXAMPLE 3

The laminate obtained in Example 1 or 2 is coated with adhesive on one of its faces, in

accordance with the same technique as that described in these same examples, and then calendered with a 75 micron polyethylene film.

The laminate obtained, which comprises the sequence: a layer of polyester, a layer of aluminium, a layer of adhesive, a layer of aluminium, a layer of polyester, a layer of adhesive and a layer of polyethylene, has a thickness of 101 microns to 107 microns and can thus be sealed on the polyethylene face, which makes it possible to manufacture sachets for packaging dehydrated

powder, such as milk. The impermeability70 characteristics of the laminate are the same as those of Example 1.

#### **EXAMPLE 4**

The metallised polyester film coated with adhesive on its metal face, such as described in 75 Example 1, is calendered with a 75 micron polyethylene film.

The laminate obtained is calendered again with a second metallised polyester film coated with adhesive on its metal face, the polyester face of the compound material being brought into contact with the adhesive.

The laminate obtained, which comprises the sequence: a polyester layer, a layer of aluminium, a layer of adhesive, a layer of polyester, a layer of aluminium, a layer of adhesive and a layer of polyethylene, is sealable on its polyethylene face, which makes it possible to manufacture sachets. The laminate obtained has the same thickness and the same characteristics as that of Example 3.

## 90 EXAMPLE 5

The metallised polyester film coated with adhesive, such as described in Examples 1 and 2, is calendered with a polypropylene film also metallised by vaporisation in vacuo. The adhesive is brought into contact with the metal of the polypropylene film.

The polypropylene film has a thickness of 20 microns, is of the co-extruded type and can be sealed to itself.

The laminate obtained has the following impermeability values:

oxygen:  $0.6 \text{ cm}^3/\text{m}^2/24 \text{ hours}$ 

water vapour:  $0.13 \text{ g/m}^2/24 \text{ hours}.$ 

lt has a thickness of 33 to 36 microns.
It makes it possible to manufacture sachets for packaging seeds for sowing.

## **EXAMPLE 6**

The laminate obtained in Example 1 or 2 is coated with polyurethane adhesive on one of its 110 faces, in accordance with the same technique as that described in Example 1, and then calendered with a 100 micron polyethylene film.

The total thickness of the material is between 126 and 133 microns. The impermeability characteristics are identical to those of Example 1.

This laminate made it possible to manufacture

sachets intended for measuring the breathing coefficient of a cheese during ripening, by means of the addition of a valve sealed to one of the main faces, for sampling the gases.

#### 5 CLAIMS

- 1. Process for packaging a material in which the packaging material is a laminate comprising two layers of thermoplastic polymers, each metallised on one face and joined together by a
- 10 layer of adhesive, one of the two layers of thermoplastic polymers consisting of poly-(ethylene glycol) terephthalate.
- 2. Process according to claim 1 in which the two metallised layers of thermoplastic polymers 15 are joined together at their metallised faces.
  - 3. Process according to claim 1 in which the two metallised layers of thermoplastic polymers are joined together with a metallised face against a thermoplastic face.

- 20 4. Process according to any one of claims 1 to 3 in which the metal is aluminium.
  - 5. Process according to any one of claims 1 to 4 in which the other thermoplastic polymer is poly-(ethylene glycol) terephthalate or
- 25 polypropylene. 6. Process according to any one of claims 1 and 3 to 5 in which the outer metal layer of the laminate has a layer of another thermoplastic
- polymer thereover. 30 7. Process according to any one of the
  - preceding claims in which the adhesive is a polyurethane.
- 8. Process according to any one of claims 1 to 7 for packaging a foodstuff, pharmaceutical 35 product, industrial product or agricultural product.
  - 9. Process according to claim 1 substantially as described in any one of the Examples.
  - 10. A material whenever packaged by a process as claimed in any one of the preceding claims.