(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau

(43) International Publication Date
6 December 2007 (06.12.2007)

PCT

(10) International Publication Number
WO 2007/138096 A1

(51) International Patent Classification:
B32B 37/12 (2006.01) *C08G 18/42* (2006.01)
C08G 18/12 (2006.01) *C09J 175/06* (2006.01)

(21) International Application Number:
PCT/EP2007/055324

(22) International Filing Date: 31 May 2007 (31.05.2007)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
60/809,655 31 May 2006 (31.05.2006) US

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(81) Designated States *(unless otherwise indicated, for every kind of national protection available)*: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BH, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, HR, HU, ID, IL, IN, IS, JP, KE, KG, KM, KN, KP, KR, KZ, LA, LC, LK, LR, LS, LT, LU, LY, MA, MD, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RS, RU, SC, SD, SE, SG, SK, SL, SM, SV, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW.

(84) Designated States *(unless otherwise indicated, for every kind of regional protection available)*: ARIPO (BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IS, IT, LT, LU, LV, MC, MT, NL, PL, PT, RO, SE, SI, SK, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

Declarations under Rule 4.17:
— *as to applicant's entitlement to apply for and be granted a patent (Rule 4.17(ii))*
— *as to the applicant's entitlement to claim the priority of the earlier application (Rule 4.17(iii))*

Published:
— *with international search report*

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

(54) **Title:** PROCESS OF HIGH SPEED LAMINATING

(57) **Abstract:** The invention provides a method of producing laminates by (a) applying a solvent-free adhesive composition comprising (A) 5 to 40 wt% of at least one linear aliphatic polyester based on linear dicarboxylic acids having a carbon number in the range of C3 to C12 and diols, (B) 20 to 50 wt% of at least one linear aliphatic polypropylene glycol having a molecular weight Mn of 400 to 2000, and (C) 20 to 45 wt% of at least one diisocyanate, the wt% are based on the adhesive composition, onto at least one side of the substrates to be joined together at a high speed greater than 100 m/min, and (b) curing the applied composition to avoid misting effects during coating and laminating under high speed.

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PROCESS OF HIGH SPEED LAMINATING

FIELD OF THE INVENTION

The invention is directed to a process for bonding substrates, in particular, for producing foil laminates under high speed, preventing misting effects during laminating.

DESCRIPTION OF RELATED ART

Laminate adhesives are used for large-area bonding of individual foils to form laminates. The properties of the individual foils are combined in such a way that the laminate is suitable for subsequent use as a packaging material for the packaging of goods, such as, for example, foodstuffs, hygiene articles or items which need protection from damage prior to first usage.

Laminate adhesives consist as a rule of isocyanate group-containing polyurethane binders, usually manufactured from hydroxyl-group-containing polyesters, polyether or simultaneously ester-and ether-group-containing hydroxy-functional condensation products and polyfunctional isocyanates, described, for example, in WO 99/24486, U.S. 6,503,981 and U.S. 6,914,102.

Usually, customary coating and laminating machines are used, for example, pressure, laminating and hotmelt machines known by a person skilled in the art, which have a heatable application mechanism, and which are operating, for example, with smooth or patterned rollers or also with suitable wipers. Evaporation zones, IR or hot-air drying or heating ducts can be used. Usually a thin coating application of, for example, 0.5 to 10 g/m2, is sufficient. The coating speed can be in the range of about 50 to 300 m/min or more.

U.S. 4,092,202 describes a method for producing foil laminates by coating under high speed using a solvent-free, free NCO or NCS group

containing reaction product of hydroxyl-functional polyethers or polyesters with polyisocyanates. Preferred polyethers are aliphatic, straight- or branched-chain polyethers; preferred polyesters are linear or branched products made from aliphatic and/or aromatic polycarboxylic acids and polyols. As polyisocyanates aliphatic, aromatic and/or cyclo-aliphatic products may be usable.

Unwanted misting effects during the laminating process may require the machine speed to be limited. Such misting effects can lead to a large amount of pollution inside and outside the laminating machine caused by the laminate adhesive product. Misting can also promote the evaporation of monomeric isocyanates which originate from the adhesive composition and which can be injurious to the health of the operators of the machines.

Therefore, there is a need to minimise or avoid such misting effects during coating and laminating under high speed conditions while maintaining the desired properties, such as, uniform application in extremely thin layers and high mechanical stability without separation of the laminated foil strips.

SUMMARY OF THE INVENTION

The present invention provides a method of producing laminates by applying a solvent-free adhesive composition onto at least one side of the substrates to be joined together at high speed greater than 100 m/min and curing; wherein the solvent-free adhesive composition is comprising :

(A) 5 to 40 wt% of at least one linear aliphatic polyester based on linear dicarboxylic acids having a carbon number in the range of C3 to C12 and diols,

(B) 20 to 50 wt% of at least one linear aliphatic polypropylene glycol having a molecular weight Mn of 400 to 2000, and

(C) 20 to 45 wt% of at least one diisocyanate.

the wt% are based on the adhesive composition.

The method according to the invention avoids the misting effects during coating and laminating under high speed operating conditions. The desired properties, such as, uniform application in thin layers, high mechanical stability without separation of the laminated foil strips can be achieved.

DETAILED DESCRIPTION OF THE INVENTION

The features and advantages of the present invention will be more readily understood, by those of ordinary skill in the art, from reading the following detailed description. It is to be appreciated those certain features of the invention, which are, for clarity, described above and below in the context of separate embodiments, may also be provided in combination in a single embodiment. Conversely, various features of the invention that are, for brevity, described in the context of a single embodiment, may also be provided separately or in any sub-combination. In addition, references in the singular may also include the plural (for example, “a” and “an” may refer to one, or one or more) unless the context specifically states otherwise.

The use of numerical values in the various ranges specified in this application, unless expressly indicated otherwise, is stated as the minimum and maximum values within the stated ranges. In this manner, slight variations above and below the stated ranges can be used to achieve substantially the same results as values within the ranges. Also, the disclosure of these ranges is intended as a continuous range including every value between the minimum and maximum values.

All patents, patent applications and publications referred to herein are incorporated by reference in their entirety.

The method according to the invention provides a method which follows the steps of applying the solvent-free adhesive composition according to the invention onto one or both of the foil sides to be joined together at a speed of more than 100 m/min.

The solvent-free adhesive composition used for the method according to the invention comprises the polyester of component A), the polyethers of component (B) and the diisocyanates of component (C).

The polyesters of component A) are linear aliphatic polyesters based on linear dicarboxylic acids having a carbon number in the range of C3 to C12, preferred in a range of C4 to C8, and diols.

The polyesters are hydroxy functional and exhibit hydroxyl values in the range of 15 to 150 mg of KOH/g and acid values in the range of 0 to 6 mg of KOH/g.

Suitable linear dicarboxylic acids are adipic acid, sebacic acid, acelaic acid, succinic acid, derivatives thereof and mixtures thereof.

Diols which may be used are, for example, ethylene glycol, diethylene glycol, triethylene glycol, hexanediol, butanediol, propylene glycol, methylpropandiol-1.3, neopentylglycol, and mixtures thereof.

The polyesters may be produced in conventional manner by performing an esterification reaction of the acid component with the alcohol component, for example, in a nitrogen atmosphere, for example, at temperatures of between 140 and 260°C, with or without use of conventional esterification catalysts.

The linear aliphatic polypropylene glycols of component B) are hydroxy-functional preferably exhibiting a hydroxyl value of 50 to 400 mg of KOH/g. They may, for example, exhibit a number average molecular weight Mn of 400 to 2,000 g/mol (determined by end group titration of the hydroxyl groups), preferred of 650 to 1100 g/mol. A preferred amount of component B) is 20 to 40 wt%.

The diisocyanates used as component C) may be aliphatic diisocyanates, trimerization products thereof and/or aromatic diisocyanates as known by a person skilled in the art.

Examples of diisocyanates are isophorone diisocyanate (IPDI), or hexamethylene diisocyanate (HDI), dicyclohexylmethane diisocyanate or the trimerization products, for example, aliphatic diisocyanate-based isocyanurates or mixtures thereof. Aromatic diisocyanate compounds may also be present, such as, for example, tolylene diisocyanate (TDI), diphenylalkyl diisocyanates (for example diphenylmethyldiisocyanates or MDI) or mixtures thereof.

Preferably, TDI and MDI are used.

The diisocyanates can be used also in blocked form. Blocking may proceed with conventional agents, e.g., with ethylene glycol monobutyl ether, butanone oxime, phenol, ethyl acetoacetate, malonic ester, dimethylpyrazole or caprolactam.

The solvent-free adhesive composition may be produced with less than 3% chain extenders related to the adhesive composition. The chain extenders may comprise the short-chain diols conventionally used for this purpose, such as, ethylene glycol, 1,2- or 1,3-propanediol, 1,6-hexanediol, di- or tripropyleneglycol. Preferably, no chain-extenders are used.

Components A), B) and C) can be mixed together by techniques known by the art, and can be stored for months. They can also be mixed together directly prior to application onto the substrates to be bonded. Mixing may be performed, for example, in a static mixer or a mixing tube.

The viscosity of the finished adhesive composition lies, for example, in a range from 300 to 1,000 mPas, particularly preferably, between 400 to 800 mPas, measured at 100°C.

The adhesive composition according to the invention may contain the additives conventional in polyurethane chemistry, such as, for example, catalysts, accelerators, light stabilizers, extenders, pigments in the amounts conventional in coating chemistry, e.g., from 0.5 to 5 wt.%, relative to the adhesive composition. Preferably, such additives are used.

Adhesion promoters may be used in quantities under 2 wt% based on the adhesive composition. Preferably, no adhesive promoters are used.

The adhesive composition according to the invention may be used to stick the most varied materials together. These materials may be, for example, web-form materials of plastics, such as, polyethylene, polypropylene, polyamide, PET (polyethylene terephthalate), paper or metal, e.g., metal foils, such as, aluminium foils, or plastics films, such as, metal oxide vapour-deposited or metal vapour-deposited plastics films.

The invention also provides use of the adhesive composition of the invention for bonding substrates, in particular, for producing laminates for packaging.

The application speed employed in the course of the method according to the invention should be in the range of about 100 to 300 m/min, preferably, more than 150 m/min and more preferably, more than 200 m/min. Basically, however one can also use faster speeds if the machines are designed accordingly. A thin application of, for example, about 0.1 to 10 g/m2, preferably about 0.5 to 5 g/m2, is sufficient.

The method according to the invention can be carried out on the most varied types of machinery, for example, pressure, laminating, and hotmelt machines comprising, for example, a heatable application mechanism suitable for thin application at the required machine operating speed. The adhesive application can be accomplished with smooth or patterned rollers, either coaxially or counter-currently, or also with suitable wipers. This is known to a person skilled in the art. To get the best and most uniform application possible, the lamination may be accomplished immediately after the application mechanism. It is possible to connect evaporation zones, IR or hot-air drying or heating ducts or additional devices between the steps, but it is not necessary, even at high machine operating speeds, for example, in excess of 150 m/min.

The processing temperature is selected so that the viscosity of the mass, when the application mechanism is correspondingly adjusted, permits the desired application volume, while avoiding that the foil, which is to be coated with the adhesive composition, is damaged by the temperature applied or is unfavourably influenced in terms of its quality and gauge. The temperature of the adhesive composition therefore should not be above 110°C, preferably between 60°C and 100°C

The application is possible in continuous or discontinuous application manner as known to a person skilled in the art.

With the process according to the invention, it is possible to produce laminates with high processing reliability, and the foils are joined immediately after application of the adhesive composition in such a way that, even at high machine speeds, there will be no bubble or crease formation as a result of the foils being shifted with respect to each other, or there will be no shifting of the foil during windup, and a high resistance of the foil laminate results. Furthermore, it is ensured that no substances injurious to the health of the consumer are developed or released, and no misting effect is noticed. A perfect, uniform coating, even in quantities of above 0.5 g/m^2 is possible.

The present invention is further defined in the following Examples. It should be understood that these Examples are given by way of illustration only. From the above discussion and these Examples, one skilled in the art can ascertain the essential characteristics of this invention, and without departing from the spirit and scope thereof, can make various changes and modifications of the invention to adapt it to various uses and conditions. As a result, the present invention is not limited by the illustrative examples set forth herein below, but rather is defined by the claims contained herein below.

The following Examples illustrate the invention. All parts and percentages are on a weight basis unless otherwise indicated.

EXAMPLES

Example 1a

Production of the Polyester

758 g ethylene glycol, 147 g diethylene glycol and 1095 g adipic acid are heated in a 2l reactor with interior thermometer, stirrer and distillating device. The temperature is increased up to 250°C until an acid value of 10 mg KOH/g is attained.

Then the batch is cooled down to 180°C and fitted to a vacuum pump. The temperature is increased again to 230°C and the batch is condensed to a viscosity of 2500 mPas and a hydroxy value of 90 to 100. After reaching the specification, the batch is cooled below 60°C and discharged.

Acid value:	4mg KOH/g
Viscosity:	2500 to 3000 mPas
Hydroxyl value :	80 to 90 KOH/g
Water content:	below 0.05%

Example 1b

Production of the Adhesive Composition according to the Invention

750 g of 2.4'-Diphenylmethanediisocyanate and 750 g of 4,4' Diphenylmethanediisocyanate are filled into a 4l reactor with interior thermometer and stirrer and homogenized by heating to 50°C. 912 g of the polyester of Example 1 are added and the mixture is heated until the exothermic reaction starts. The batch is kept at 80°C for 1 hour and cooled to 60°C to charge 1508 g of polypropyleneglycol 1100. The batch is heated to 80°C again, stirred for 2 hours at this temperature. 72 g of

dipropyleneglycol is added and one hour later the viscosity and the isocyanate content of the resulted composition is measured.

Result: Isocyanate content: 6.7%

Viscosity (100°C): 564 mPas

Example 2

Use of the Adhesive Compositions and Results

On a Labocombi 400 lamination machine a polyester web (thickness 12μm / 300 mm width) was bonded with aluminium foil (thickness of 9μm / 300 mm width) by use of the adhesive composition of Example 1b to give a laminate, and cured during a time of 3 days. Dry coating weight was approximately 1.5 g/m^2.

A laminate of the same materials was produced under the use of an adhesive of the prior art based on a linear aromatic polyester with Mn 1000 to 1200.

Both adhesives were tested at different temperatures in the application head and the misting effect is measured by visual observation during the application process under different application speeds. The results are shown in Table 1.

Table1

	Misting Effect [80°C]	Misting Effect [90°C]
Adhesive Composition (Invention)	240 m / min	> 250 m / min
Adhesive of the prior art	75 m / min	120 m / min

The adhesive of the prior art shows misting effects already at lower application speeds.

The values for bond strength according to the 90° Peel Test (DIN 53357, method A) of different laminates were measured. The results are shown in Table 2.

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Table 2

	Laminate 1 according to Example 2 based on Aluminium/Polyester	Laminate 2 based on Aluminium/ polyethylene terephthalate
Adhesive Composition (Invention)	7.4	3.3
Adhesive of the prior art	7.8	4.1

The bonding properties of the laminates provided by the invention have similar values as the bonding properties of the laminates provided by the prior art, according to the application standards in this application field.

CLAIMS

1. A method of producing laminates by

(a) applying a solvent-free adhesive composition comprising

(A) 5 to 40 wt% of at least one linear aliphatic polyester based on linear dicarboxylic acids having a carbon number in the range of C3 to C12 and diols,

(B) 20 to 50 wt% of at least one linear aliphatic polypropylene glycol having a molecular weight Mn of 400 to 2000, and

(C) 20 to 45 wt% of at least one diisocyanate,

onto at least one side of the substrates to be joined together, at a high speed greater than 100 m/min, and

(b) curing the applied composition.

2. A method according to claim 1 characterized in that the linear aliphatic polyester of component (A) is based on linear dicarboxylic acids having a carbon number in the range of C4 to C8.

3. A method according to anyone of claims 1 or 2, characterized in that the linear aliphatic polyester of component (A) is hydroxy functional and exhibits hydroxyl values in the range of 15 to 150 mg of KOH/g and acid values in the range of 0 to 6 mg of KOH/g.

4. A method according to anyone of claims 1 to 3, characterized in that the linear aliphatic polypropylene glycols of component B) are hydroxy-functional and exhibit a hydroxyl value of 50 to 400 mg of KOH/g.

5. A method according to anyone of claims 1 to 3, characterized in that the linear aliphatic polypropylene glycols of component B) exhibit a number average molecular weight Mn of 650 to 1100 g/mol.

6. A method according to anyone of claims 1 to 5, characterized in that the solvent-free adhesive composition comprises less than 3 % chain extenders.

7. A method according to anyone of claims 1 to 6, characterized in that the application speed is in the range of about 100 to 300 m/min, preferably more than 150 m/min and more preferably more than 200 m/min.

8. A method according to anyone of claims 1 to 7, characterized in that the solvent-free adhesive composition is applied at about 0.1 to 10 g/m2, preferably at about 0.5 to 5 g/m2.

9. A method according to anyone of claims 1 to 8, characterized in that the solvent-free adhesive composition is applied at a temperature less than 110°C, preferably between 60°C and 100°C.

10. The solvent-free adhesive composition such as defined in anyone of claims 1 to 6.

INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2007/055324

A. CLASSIFICATION OF SUBJECT MATTER
INV. B32B37/12 C08G18/12 C08G18/42 C09J175/06

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
B32B C09J C08L C08G

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)
EPO-Internal

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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[X] Further documents are listed in the continuation of Box C.

[X] See patent family annex.

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Date of the actual completion of the international search	Date of mailing of the international search report
20 August 2007	28/08/2007

Name and mailing address of the ISA/	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL – 2280 HV Rijswijk Tel. (+31–70) 340–2040, Tx. 31 651 epo nl, Fax: (+31–70) 340–3016	Kopp, Christian

INTERNATIONAL SEARCH REPORT

International application No
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