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

(19) World Intellectual Property Organization
International Bureau

(43) International Publication Date
12 April 2012 (12.04.2012)

PCT

(10) International Publication Number
WO 2012/045762 A1

(51) International Patent Classification:
B32B 1/02 (2006.01) ***B60K 15/03*** (2006.01)
B32B 27/08 (2006.01) ***C08K 7/00*** (2006.01)
B32B 27/20 (2006.01)

(21) International Application Number:
PCT/EP2011/067360

(22) International Filing Date:
5 October 2011 (05.10.2011)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
1058169 8 October 2010 (08.10.2010) FR

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(81) Designated States *(unless otherwise indicated, for every kind of national protection available)*: AE, AG, AL, AM, AO, AT, AU, AZ, BA, BB, BG, BH, BR, BW, BY, BZ, CA, CH, CL, 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, ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PE, PG, PH, PL, PT, QA, RO, RS, RU, RW, SC, SD, SE, SG, SK, SL, SM, ST, SV, SY, TH, 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, LR, LS, MW, MZ, NA, RW, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European (AL, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HR, HU, IE, IS, IT, LT, LU, LV, MC, MK, MT, NL, NO, PL, PT, RO, RS, SE, SI, SK, SM, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

Published:
— *with international search report (Art. 21(3))*

WO 2012/045762 A1

(54) Title: PLASTIC FUEL TANK

(57) Abstract: Plastic fuel tank, or accessory for such a tank, comprising a base plastic layer, a hydrocarbon barrier layer and, between these two layers, a layer comprising at least one adhesive plastic, characterized in that the layer comprising the adhesive plastic has a nanoscopic structure.

Plastic fuel tank

The present invention relates to a plastic fuel tank, or to an accessory (and in particular a fill pipe) for such a tank, which is impermeable to liquids and/or gases.

Hollow bodies, in particular the tanks currently used for storing liquids and/or gases, are often constituted mainly of plastic due to the advantages of its weight, mechanical strength, chemical resistance and ease of processing, in particular when they have complex outer shapes.

Plastic tanks are widespread in motor vehicles, where they are used to contain various fluids: brake fluid, windscreen washer fluid and headlight washer fluid, tanks for fuels or for various additives (diesel, petrol, LPG gas, fuel additives, urea, etc.).

It is often imperative, for automotive uses, to ensure the impermeability of onboard tanks, in particular in the case of fuel tanks (FTs).

Various techniques are known for making the plastic articles impermeable. Two different approaches have resulted in solutions being proposed which have been used in the processes for manufacturing these articles.

The first is based on the surface treatment of the article by means of a plasma or a chemical reagent such as sulphuric anhydride or sulphur oxide SO_3 (sulphonation) or fluorine gas that are capable of modifying the molecules of the plastic located at the surface of the article. However, considering the fact that the emissions requirements laid down by current environmental standards (EURO V, LEV II, LEV III and PZEV for example) are increasingly strict, the tanks obtained via this technique do not have sufficiently high performance and/or they risk being no longer suitable in the near future.

Another approach consists in inserting into the thickness of the plastic that constitutes the article (and which is often HDPE (high-density polyethylene) in the case of fuel tanks), a layer comprising a particular material, often a thermoplastic, which has a barrier property with respect to one or more gases or liquids. In the latter case, techniques for processing via coextrusion-blow moulding are widely used. As a barrier material, use is commonly made of EVOH (ethylene/vinyl alcohol copolymer), fluoropolymers, polyamides,

polyacrylonitriles, polyesters (PET, PBT, etc.), liquid crystal polymers (LCPs) and also polyvinylidene halides (PVDF, PVDC).

Generally, the barrier material and the base plastic that constitute most of the article are not compatible so a layer of adhesive must be applied between the two.

Therefore, a structure that is often used and that gives good results in practice is a structure comprising at least 5 layers arranged in the following order (from the inside to the outside of the hollow body):

a) a layer based on virgin polyolefin,

b) a layer comprising at least one adhesive,

c) a hydrocarbon barrier layer,

d) a layer comprising at least one adhesive,

e) an outer layer based on virgin polyolefin,

optionally with a layer comprising recycled plastic between the layers d) and e).

Although this type of structure (in particular based on HDPE as polyolefin, on EVOH as barrier material and on grafted PE (in particular that is grafted with maleic anhydride or MA) as adhesive) makes it possible to satisfy the requirements of the LEV II standard, it might not meet those of the future LEV III standard. Moreover, it has been observed that the performances, in terms of impermeability, of this type of structure in contact with alcohol-containing fuels were degraded in the long term.

The objective of the present invention is to provide (accessories for) FTs based on a multilayer structure including at least one base plastic layer, one layer comprising at least one adhesive and one hydrocarbon barrier layer, the impermeability of which is improved.

For this purpose, the invention relates to a(n) (accessory for a) plastic fuel tank comprising a base plastic layer, a hydrocarbon barrier layer and, between these two layers, a layer comprising at least one adhesive plastic, characterized in that the layer comprising the adhesive plastic has a nanoscopic structure.

The term "FT" is understood to mean, within the context of the invention, petrol, diesel or additive tanks for motor vehicles and also the impermeable accessories intended to equip these tanks. By “accessory” is meant on object performing an active function (like venting, gauging, transporting liquid or gaseous fuel…) in or on the tank and being in contact with the fule (in liquid and/or in gaseous form). As accessories for tanks, mention may be made, non-limitingly, of: filler pipes, fuel transfer lines, canisters intended to contain a

composition that retains the fuel vapours, valves of various types, pipe/tank connections, etc. The invention gives good results for articles obtained by coextrusion-blow moulding or co-injection moulding. Consequently, it applies particularly well to fuel tanks and to their filler pipes.

As base plastic within the context of the invention, use is generally made of standard polymers such as polyolefins or halogenated polymers. Good results have been obtained when the base plastic is mainly constituted of one or more polyolefins chosen from homopolymers and copolymers of ethylene or of propylene. Excellent results have been obtained with high-density polyethylene (HDPE).

The hydrocarbon barrier layer preferably comprises a barrier plastic, i.e. generally a crystalline plastic that is not compatible with the base plastic. Advantageous crystalline polymers are in particular polyamides (PA), fluoropolymers and vinyl alcohol polymers [homopolymers (PVOH) and ethylene/vinyl alcohol copolymers (EVOH)]. Very good results are obtained when the barrier layer is mainly constituted of one or more polymers chosen from polyamides and ethylene/vinyl alcohol copolymers, and very particularly of the latter. However, it should be noted that the polyamides are generally less expensive.

In particular, the present invention gives good results when the base plastic is HDPE and the barrier plastic is EVOH and/or PA.

The polyamides that are suitable may be aliphatic or aromatic. Among the aliphatic polyamides, the following are preferred: homopolymers such as polyamide 6 (PA-6) or polyamide 6,6 (PA-6,6) or polyamide 11 or polyamide 12, or copolymers such as PA-6/12, PA-6/6,6 and copolymers based on polyamide blocks and polyether blocks. Among the aromatic polyamides, those of the MXD6 type give good results. These are polyamides based on m-xylenediamine (MXDA) polycondensed with an adipic acid and therefore comprise an aromatic ring in their main chain. Among the EVOH grades, the following products gave excellent results: those having from 20 to 40 mol% of ethylene and in particular, certain commercial grades having 24 and 32 mol% of ethylene.

According to the invention, a layer comprising an adhesive plastic and having a nanoscopic structure is placed between the base layer and the barrier layer.

The adhesive plastic is chosen in function of the nature of the barrier and

base plastics. Use is frequently made, as adhesive plastic, of a functionalized polyolefin (i.e. comprising functions compatible with the barrier material), and in particular of reactive or functionalized polyethylene (PE), since the tanks are often based on such a material. The functionalization may be obtained by grafting or copolymerization. The aforementioned functions may be of carboxylic acid anhydride type, such as maleic anhydride or glycidyl methacrylate for example. Preferably, the adhesive plastic is mainly constituted of a polyolefin (in particular polyethylene) grafted or copolymerized with maleic anhydride or glycidyl methacrylate. Optionally, a third monomer may be present, such as alkyl (meth)acrylate and, in particular, ethyl acrylate. Such adhesives are namely sold under the name Lotader® by ARKEMA.

Generally, the layer of adhesive is thin relative to the whole of the tank or of the accessory and, in particular, it does not exceed a few % of this thickness. It is generally greater than or equal to 1% by weight of the tank or of the accessory, but does generally not exceed 5%, or even 3% by weight of the tank or of the accessory. It is typically of the order of 2% by weight of the tank or of the accessory.

According to the invention, it has a nanoscopic structure. This is understood to denote a heterogeneous structure comprising at least two components, one of which is present (dispersed in the other) in a form such that it has at least one submicronic dimension, typically of the order of hundreds, preferably tens of nanometres, or even of the order of the nanometre.

Although the layer of adhesive is thin, the Applicant has observed that the fact of giving it a nanoscopic structure made it possible to greatly reduce the emissions of hydrocarbons from the tanks/accessories where it is present. Another advantage of using this structure in an inner layer of the tank is that it influences neither the surface properties (weldability) nor the impact strength of the tank.

According to a first variant of the invention, the dispersed component having a nanometric dimension is an inorganic nanofiller, in particular chosen from the following materials: clay, montmorillonite, eponite, vermiculite, carbon nanotubes, carbon sheets and graphene. These are preferably lamellar exfoliable fillers such as silicates and, in particular, organophilic treated clays.

According to a second variant, the dispersed component having a nanometric dimension is a barrier plastic (or a blend of barrier plastics) so that the adhesive layer is in fact a blend of polymers having a nanoscopic structure,

or a copolymer having such a structure. Very particularly preferably, either this structure is co-continuous (i.e. comprising at least two interpenetrated continuous phases, at least one of which (the one based in a barrier plastic) comprises channels of nanometric diameter), or it is lamellar (comprising lamellae or tubes of barrier plastic, the thickness or the diameter respectively of which is of the order of nanometres). A co-continuous structure is preferred.

In this variant, the layer comprising the adhesive plastic is preferably constituted of a blend of polymers having a two-phase morphology of nanoscopic dimension. The polymer blend comprises at least two phases and is constituted, on the one hand, of a reactive or functionalized polyolefin phase comprising functions that are compatible with the barrier material and, on the other hand, of a hydrocarbon barrier material (it being possible for the barrier material to be chosen from the list above). In particular, the reactive polyolefin phase may be reactive or functionalized polyethylene (PE), since the tanks are often based on such a material. Preferably, the polyolefin phase is mainly constituted of a grafted polyolefin (in particular polyethylene) as described above. The barrier phase of the adhesive plastic may be constituted of crystalline polymers such as, in particular, polyamides, fluoropolymers and vinyl alcohol polymers [homopolymers (PVOH) and ethylene/vinyl alcohol copolymers (EVOH)]. Very good results are obtained when the barrier phase of the adhesive plastic is mainly constituted of one or more polymers chosen from polyamides and ethylene/vinyl alcohol copolymers, and very particularly of the latter.

In order to obtain such a nanoscopic structure, one practical means consists in quenching a thermodynamically unstable structure obtained by blending these two polymers at high temperature and/or under high shear. However, these structures are by definition unstable and therefore do not survive a subsequent processing operation. It is therefore necessary to generate said structure and quench it at the same time as the tank or accessory is manufactured, which is not practical.

Therefore, according to one preferred variant, the co-continuous structure is in fact based on a copolymer of PE and of the barrier polymer having blocks of adequate length to respectively form phases exclusively based on one or the other of the polymers and to self-assemble or self-structure on a nanoscopic scale. This morphology thus structured on a nanoscopic scale is moreover thermodynamically stable.

The expression "based on" is understood to mean that the material is predominantly constituted of such a copolymer, which does not rule out the presence of other polymers (for example, virgin, unfunctionalized HDPE; fillers, etc.). The fact of adding virgin HDPE may help to increase the crystallinity of the material constituting the layer of adhesive and, by doing so, reduce the permeability thereof.

To date, two methods are mainly known for synthesizing such polymers: either by reactive extrusion of the two separate polymers, or by controlled (also known as "living") radical polymerization.

According to the 1st method, the copolymer on which is based the nanostructured multiphase material is obtained by blending (preferably by reactive extrusion in a twin-screw or single-screw extruder) a functionalized PE, preferably one that is functionalized along the entire length of its chains, with a barrier polymer having reactive chain ends. Use can also be made of an internal mixer of the Brabender or Banbury type.

The parameters that a person skilled in the art must optimize in this variant are mainly the molecular weight of the starting polymers, and the distribution of the reactive functional groups over the PE, which distribution should be as uniform as possible.

The copolymers obtained by this method generally also contain monomers that have not reacted and that contribute in fact to the thermodynamic stability of the multiphase material, by giving it an adhesive nature.

According to the 2nd method, the copolymer on which is based the nanostructured multiphase material is obtained by a controlled radical polymerization (ATRP (Atom Transfer Radical Polymerization)), RAFT (Reversible Addition Fragmentation chain Transfer), NMP (Nitroxide Mediated Polymerization), etc.

This variant of the invention gives good results when the barrier polymer is a polyamide (PA) and in particular, with co-continuous structures based on PE and on PA such as those described for example in application WO 2009/019263 in the name of the Applicant (the content of which is, for this purpose, incorporated by reference in the present application) and in the article "Design and properties of co-continuous nanostructured polymers by reactive blending", Nature Materials, September 2002, pages 54-58 (structures based on MA-grafted PE that contains ethyl acrylate, and on PA-6).

It should be noted that the two variants of the invention described above (nanometric filler or nanometric polymer structure) may advantageously be combined. In particular, in the case of the co-continuous structures described above, nanometric fillers may help to reinforce the impermeability of the structure. In this case, preferably, these fillers are distributed in the barrier plastic phase.

The invention applies in particular to at least one of the layers of adhesive of tanks and pipes comprising at least 5 layers arranged in the following order (from the inside to the outside of the hollow body):

a) a layer of base plastic (preferably HDPE) (P),
b) a layer comprising at least one adhesive (A),
c) a hydrocarbon barrier layer (EVOH or PA) (B),
d) a layer comprising at least one adhesive (A),
e) a layer of base plastic (preferably HDPE) (P),

optionally with a layer comprising recycled plastic (R) between the layers d) and e).

This plastic is preferably constituted of waste from the production of similar tanks/pipes. In particular, it may be grindings of such articles (either end-of-life articles or production scrap), optionally subjected to a pretreatment to recover therefrom a significant fraction of the barrier plastic as described in application WO 2005/082615 in the name of the Applicant and the content of which is incorporated by reference in the present application. Such a treatment (process) comprises the following steps:

- the article is fragmented;
- the fragments obtained are subjected to at least one electrostatic separation step, so as to obtain at least one stream (I) of fragments poor in barrier plastic and one stream (II) of fragments richer in barrier plastic.

The stream (I) is that used in the 6-layer structure described above.

Tanks of P/A/B/A/R/P type as described above have been sold for many years both in North America and in Europe, in order to satisfy increasingly demanding evaporative standards. The relative thicknesses of the various layers in these tanks are typically (as % by weight of the structure): 40/2/3/2/40/13.

In this variant, the two layers of adhesives preferably have a nanoscopic structure, but they should not necessarily be of the same composition. Thus, the first layer of adhesive encountered starting from the inside of the tank/pipe (and which is therefore the closest to the fuel) may comprise EVOH and the second

may not (essentially for economic reasons). In particular, the following structure may be considered:

a) a layer based on HDPE,

b) a layer based on a nanostructured PE/PA blend and on EVOH,

c) a layer based on EVOH and/or on PA,

d) a layer based on a nanostructured PE/PA blend,

e) a layer based on HDPE,

optionally with a layer comprising recycled plastic between layers d) and e).

Preferably, the multilayer structures of the invention comprise at least one layer which is electrically conductive, for instance through the addition of a conductive additive (like carbon black) to it. This layer may be the adhesive layer. However, its is preferably a surface layer.

The present invention also relates to the use of a pipe and/or of a tank as described above for storing fuel that contains alcohol, and in particular ethanol.

CLAIMS

1. - Plastic fuel tank, or accessory for such a tank, comprising a base plastic layer, a hydrocarbon barrier layer and, between these two layers, a layer comprising at least one adhesive plastic, characterized in that the layer comprising the adhesive plastic has a nanoscopic structure.

2. - Tank or accessory according to the preceding claim, characterized in that the base plastic is a polyolefin and in that the adhesive plastic contains a polyolefin comprising functions that are compatible with the material of the barrier layer.

3. - Tank or accessory according to any one of the preceding claims, characterized in that the base plastic is HDPE and in that the barrier layer comprises EVOH and/or PA.

4. - Tank or accessory according to any one of the preceding claims, characterized in that the layer comprising the adhesive plastic is greater than or equal to 1% by weight of the tank or of the accessory, but does not exceed 5% by weight of the tank or of the accessory.

5. - Tank or accessory according to any one of the preceding claims, characterized in that the layer comprising the adhesive plastic also comprises an inorganic nanofiller.

6. - Tank or accessory according to the preceding claim, characterized in that the nanofiller is a lamellar exfoliable silicate.

7. - Tank or accessory according to any one of the preceding claims, characterized in that the adhesive plastic is a blend of polymers having a nanoscopic structure, or a copolymer having such a structure.

8. - Tank or accessory according to the preceding claim, characterized in that the nanoscopic structure is co-continuous or lamellar.

9. - Tank or accessory according to the preceding claim, characterized in that the nanoscopic structure is co-continuous, thermodynamically unstable and set by hardening.

10. - Tank or accessory according to any one of Claims 1 to 8, characterized in that the nanoscopic structure is co-continuous and based on a copolymer of PE and of the barrier polymer having blocks of adequate length to respectively form phases exclusively based on one or the other of the polymers and to self-assemble or self-structure on a nanoscopic scale.

11. - Tank or accessory according to the preceding claim, characterized in that the barrier polymer is a polyamide (PA).

12. - Tank or accessory according to any one of the preceding claims, characterized in that it comprises at least 5 layers arranged in the following order (from the inside to the outside):

a) a layer of base plastic (preferably HDPE),

b) a layer comprising at least one adhesive plastic,

c) a hydrocarbon barrier layer (preferably based on EVOH and/or PA),

d) a layer comprising at least one adhesive plastic,

e) a layer of base plastic (preferably HDPE).

13. - Tank or accessory according to the preceding claim, characterized by the presence of a layer comprising recycled plastic between layers d) and e), this plastic being constituted of waste from the production of similar tanks/pipes.

14. – Use of a tank or of an accessory according to any one of the preceding claims, for storing a fuel that comprises an alcohol.

15. – Use according to the preceding claim, according to which the alcohol is ethanol.

INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2011/067360

A. CLASSIFICATION OF SUBJECT MATTER
INV. B32B1/02 B32B27/08 B32B27/20 B60K15/03 C08K7/00
ADD.

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 B60K

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, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X Y	EP 1 607 213 A1 (ARKEMA [FR]) 21 December 2005 (2005-12-21) paragraphs [0002], [0017], [0060], [0077]; claims 1,12	1-3,5,6, 12,14,15 4,7-11, 13
X	US 2002/110658 A1 (LUCKE ROBERT V [US] ET AL) 15 August 2002 (2002-08-15) paragraphs [0029], [0060], [0062], [0069]; claims 1,7; figure 11	1-6,12, 14,15
Y	FR 2 919 534 A1 (INERGY AUTOMOTIVE SYSTEMS RES [BE]) 6 February 2009 (2009-02-06) claims 1,2	7-11

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[X] Further documents are listed in the continuation of Box C.

[X] See patent family annex.

* Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

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"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
2 January 2012	11/01/2012

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Form PCT/ISA/210 (second sheet) (April 2005)

INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2011/067360

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	WO 2005/082615 A1 (INERGY AUTOMOTIVE SYSTEMS RES [BE]; VAN SCHAFTINGEN JULES-JOSEPH [BE];) 9 September 2005 (2005-09-09)	4,13
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INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No
PCT/EP2011/067360

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