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(54) **GRAPHENE-COATED PAPER OR PAPERBOARD**

(57) The present disclosure provides a coated paper product comprising:
- a paper or paperboard substrate;
- a barrier coating, wherein the barrier coating comprises graphene and a polyolefin copolymer bearing carboxylic

acid moieties, wherein the polyolefin copolymer bearing carboxylic acid moieties preferably is ethylene-acrylic acid copolymer (EAA) or ethylene-methacrylic-acid-copolymer (EMAA) or maleic acid ethylene copolymer (MAE).

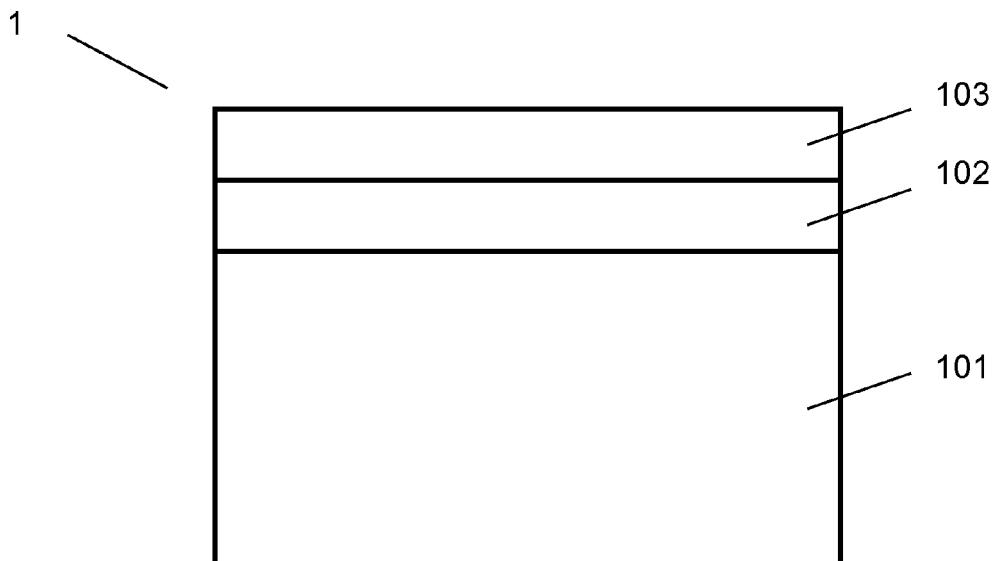


Fig. 1

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Description**TECHNICAL FIELD**

5 **[0001]** The present disclosure relates to the field of coated paper and paperboard and in particular to barrier-coated paper and paperboard.

BACKGROUND

10 **[0002]** The packaging manufacturing industry faces a major challenge to develop concepts that enable the production of packages based on sustainable resources. This concerns in particular those segments of industry that produce packaging for sensitive products such as food, beverages and pharmaceuticals.

15 **[0003]** Currently, these packaging products contain several layers of fossil-based polymers such as polyethylene, in combination with aluminum, which shield the sensitive content from e.g. moisture. It is interesting to note that even though aluminum constitutes the smallest barrier component in the packaging product, the aluminum accounts for most of the energy that is consumed in the packaging manufacturing process. Efforts to replace these materials are ongoing, but not free from challenges.

SUMMARY

20 **[0004]** The present disclosure aims to provide a paper-based material for use in packaging where water-vapour barrier properties are important. There is, thus, provided the following listing of itemized embodiments:

25 1. A coated paper product comprising:

- a paper or paperboard substrate;
- a barrier coating, wherein the barrier coating comprises graphene and a polyolefin copolymer bearing carboxylic acid moieties, wherein the polyolefin copolymer bearing carboxylic acid moieties preferably is ethylene-acrylic acid copolymer (EAA) or ethylene-methacrylic-acid-copolymer (EMAA) or maleic acid ethylene copolymer (MAE).

35 2. The coated paper product of item 1, wherein the coat weight of the barrier coating is at least 4 g/m², such as 4-15 g/m², preferably 5-10 g/m².

40 3. The coated paper product of item 1 or 2, wherein the coated paper product comprises a pre-coating between the substrate and the barrier coating.

45 4. The coated paper product of item 3, wherein the pre-coating comprises a polyolefin copolymer bearing carboxylic acid moieties or vinyl acetate acrylate copolymer (VAcA) or styrene-acrylate copolymer (SA) or styrene-butadiene copolymer (SB), wherein the polyolefin copolymer bearing carboxylic acid moieties preferably is ethylene-acrylic acid copolymer (EAA) or ethylene-methacrylic-acid-copolymer (EMAA) or maleic acid ethylene copolymer (MAE).

50 5. The coated paper product of item 3 or 4, wherein the coat weight of the pre-coating is at least 4 g/m², such as 4-15 g/m², preferably 5-10 g/m².

55 6. The coated paper product of any of the items 3-5, wherein the total coat weight of the pre-coating and the barrier coating is 5-30 g/m², such as 8-25 g/m², preferably 8-20 g/m².

 7. The coated paper product of any of the preceding items, wherein the graphene content in the barrier coating is 0.3-3 wt%, such as 0.5-2 wt%, based on the dry weight of the barrier coating.

 8. The coated paper product of any of the preceding items, wherein the content of polyolefin copolymer bearing carboxylic acid moieties in the barrier coating is at least 20 wt%, such as at least 40 wt%, such as at least 60 wt%, such as at least 80 wt%, such as at least 90 wt%, such as at least 95 wt% based on the dry weight of the barrier coating.

 9. The coated paper product of any one of the preceding items, wherein the water vapor transmission rate (WVTR)

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measured according to ISO 15106-1 at 23°C and 50% relative humidity (RH) of the coated paper product is below 10 g/(m² day).

10. The coated paper product of any one of the preceding items, wherein the water vapor transmission rate (WVTR) measured according to ISO 15106-1 at 30°C and 80% relative humidity (RH) of the coated paper product is below 30 g/(m² day).

11. The coated paper product of any one of the preceding items, wherein the water vapor transmission rate (WVTR) measured according to ISO 15106-1 at 38°C and 90% relative humidity (RH) of the coated paper product is below 65 g/(m² day).

12. The coated paper product of any one of the preceding items, wherein the density measured according to ISO 534:2011 of the paper substrate is 600-900 kg/m³.

13. The coated paper product of any one of the preceding items, wherein the grammage measured according to ISO 536:2020 of the paper substrate is 70-350 g/m².

14. The coated paper product of any one of items 1-11, wherein the coated paper is for use in a flow wrapping process and the substrate is a machine-glazed (MG) kraft paper.

15. The coated paper product of item 14, wherein the first side of the paper substrate is the non-glazed side of the MG paper and wherein the glazed side is optionally printed.

16. The coated paper product of item 14 or 15, wherein the thickness measured according to ISO 534:2011 of the paper substrate is 50-64 µm, such as 52-61 µm.

17. The coated paper product of any one of items 14-16, wherein the thickness measured according to ISO 534:2011 of the coated paper product is 55-70 µm, such as 57-67 µm.

18. The coated paper product of any one of items 14-17, wherein the density measured according to ISO 534:2011 of the paper substrate is 800-900 kg/m³.

19. The coated paper product of any one of items 14-18, wherein the grammage measured according to ISO 536:2020 of the paper substrate is 40-60 g/m², such as 42-55 g/m².

20. The coated paper product of any one of items 14-19, wherein the density measured according to ISO 534:2011 of the coated paper product is 950-1100 kg/m³.

21. The coated paper product of any one of items 14-20, wherein the paper substrate is bleached, e.g. has an ISO Brightness according to ISO 2470 of at least 77.

22. The coated paper product of any one of the preceding items wherein the paper product is heat-sealable.

23. The coated paper product of any one of the preceding items, wherein a sealant layer, such as cold-seal layer, is arranged at least partially on the barrier coating layer.

24. A packaging comprising the coated paper product according to any one of the preceding items.

25. A method of producing a coated paper product comprising the steps of:

- providing a paper or paperboard substrate; and
- coating the substrate with a barrier coating, wherein the barrier coating comprises graphene and a polyolefin copolymer bearing carboxylic acid moieties, wherein the polyolefin copolymer bearing carboxylic acid moieties preferably is ethylene-acrylic acid copolymer (EAA) or ethylene-methacrylic-acid-copolymer (EMAA) or maleic acid ethylene copolymer (MAE).

26. The method of item 25, wherein the barrier coating is applied in-line.

27. The method of item 25 or 26, further comprising the step of:

- coating the substrate with a pre-coating prior to coating with the barrier coating so that the pre-coating is arranged between the substrate and the barrier coating in the coated paper product.

28. The method of item 27 comprising drying between the pre-coating step and the barrier-coating step.

29. The method of item 27 or 28, wherein the pre-coating is applied in-line.

30. Use of a coating comprising graphene and a polyolefin copolymer bearing carboxylic acid moieties as a water vapor barrier on a paper or paperboard substrate, wherein the polyolefin copolymer bearing carboxylic acid moieties preferably is ethylene-acrylic acid copolymer (EAA) or ethylene-methacrylic-acid-copolymer (EMAA) or maleic acid ethylene copolymer (MAE).

BRIEF DESCRIPTION OF THE DRAWINGS

[0005]

Fig 1 is a schematic illustration of an embodiment of the coated paper product 1 of the present disclosure. The paper product consists of a paper substrate 101, a pre-coating layer 102 and a barrier coating layer 103.

Fig 2 is a graph of the water vapor transmission rate (WVTR) at 38°C and 90% relative humidity (RH) of a paper coated with neat latex and a paper coated with latex where graphene or graphene oxide has been added.

DETAILED DESCRIPTION

[0006] As a first aspect of the present disclosure, there is provided a coated paper product comprising:

- a paper or paperboard substrate;
- a barrier coating, wherein the barrier coating comprises graphene and a polyolefin copolymer bearing carboxylic acid moieties, wherein the polyolefin copolymer bearing carboxylic acid moieties preferably is ethylene-acrylic acid copolymer (EAA) or ethylene-methacrylic-acid-copolymer (EMAA) or maleic acid ethylene copolymer (MAE).

[0007] In the context of the present disclosure, the term graphene includes reduced graphene oxide having an oxygen content below 4 % measured with XPS.

[0008] The polyolefin copolymer bearing carboxylic acid moieties is typically a random copolymer, alternating copolymer or statistical copolymer and not a block copolymer. Preferably the polyolefin copolymer bearing carboxylic acid moieties is ethylene-acrylic acid copolymer (EAA) or ethylene-methacrylic-acid-copolymer (EMAA) or maleic acid ethylene copolymer (MAE). Most preferred polyolefin copolymer bearing carboxylic acid moieties is ethylene-acrylic acid copolymer (EAA). The carboxylic acid moieties refer to the carboxylic acid moiety present in the monomeric unit that has been copolymerized with the olefin. Examples of carboxylic acid moieties are the carboxylic acid moieties present in acrylic acid in EAA, carboxylic acid moieties present methacrylic acid in EMA and carboxylic acid moieties present in maleic acid in MAE. MAE may be provided in the form of maleic anhydride ethylene copolymer, which is hydrolysed into maleic acid ethylene copolymer.

[0009] Typically, the graphene content in the barrier coating is 0.3-3 wt%, such as 0.5-2 wt%, based on the dry weight of the barrier coating. Typically, the content of polyolefin copolymer bearing carboxylic acid moieties in the barrier coating is at least 80 wt%, such as at least 90 wt%, such as at least 95 wt% based on the dry weight of the barrier coating. As an example, the content of polyolefin copolymer bearing carboxylic acid moieties in the barrier coating is 20-99 wt%, such as 40-99 wt%, such as 60-99 wt%, such as 80-99 wt%, based on the dry weight of the barrier coating.

[0010] The coated paper product may comprise a pre-coating between the substrate and the barrier coating. If the surface of the substrate is rough, it is beneficial to apply a pre-coating to level out the surface. On the other hand, if the surface is sufficiently smooth, such as in a machine-glazed paper or hard-calendered paper, a pre-coating is typically not needed to level out the surface. The pre-coating comprises a polyolefin copolymer bearing carboxylic acid moieties or vinyl acetate acrylate copolymer (VAcA) or styrene-acrylate copolymer (SA) or styrene-butadiene copolymer (SB), wherein the polyolefin copolymer bearing carboxylic acid moieties preferably is ethylene-acrylic acid copolymer (EAA) or ethylene-methacrylic-acid-copolymer (EMAA) or maleic acid ethylene copolymer (MAE). The polymer in the pre-coating is applied as a latex to the substrate. Such polymers provide a sufficiently smooth surface.

[0011] The coat weight of the barrier coating and the optional pre-coating, if provided, is typically at least 4 g/m², such as 4-15 g/m², preferably 5-10 g/m². From an economical and environmental perspective, it is advantageous to coat with as low grammage as possible and in a full-scale coating machine the coating weight can be lower than in a laboratory equipment.

[0012] The coated paper product is typically heat-sealable. Polyolefin copolymer bearing carboxylic acid moieties such as EAA and EMA and MAE are inherently heat-sealable and this heat-sealability is typically maintained upon graphene addition. A sealant layer, such as cold-seal layer, may also be arranged at least partially on the barrier coating layer. In such case, it is not necessary to apply heat to seal the coated paper product. It is advantageous for the coated paper product to be sealable in order to allow the formation of a packages by sealing the paper to itself.

[0013] The coated paper product is typically a water vapor barrier. The water vapor transmission rate (WVTR) measured according to ISO 15106-1 at 23°C and 50% relative humidity (RH) of the coated paper product is typically below 10 g/(m² day). The WVTR measured according to ISO 15106-1 at 30°C and 80% relative humidity (RH) of the coated paper product is typically below 30 g/(m² day). The WVTR measured according to ISO 15106-1 at 38°C and 90% relative humidity (RH) of the coated paper product is typically below 65 g/(m² day).

[0014] As a second aspect of the present disclosure, there is provided a packaging comprising the coated paper product according to the first aspect. Such product includes, but is not limited to, food or liquid packaging where water barrier properties are important, such as flow-wrap packaging or packaging for cold and hot food or liquids, or pharmaceutical packaging or industrial packaging, such as cement sacks.

[0015] The examples and embodiments discussed above in connection to the first aspect apply to the second aspect *mutatis mutandis*.

[0016] As a third aspect of the present disclosure there is provided a method of producing a coated paper product comprising the steps of:

- providing a paper or paperboard substrate; and
- coating the substrate with a barrier coating, wherein the barrier coating comprises graphene and a polyolefin copolymer bearing carboxylic acid moieties, wherein the polyolefin copolymer bearing carboxylic acid moieties preferably is ethylene-acrylic acid copolymer (EAA) or ethylene-methacrylic-acid-copolymer (EMAA) or maleic acid ethylene copolymer (MAE).

[0017] The method may comprise the step of coating the substrate with a pre-coating prior to coating with the barrier coating so that the pre-coating is arranged between the substrate and the barrier coating in the coated paper product.

[0018] In one embodiment, the method comprises drying between the application of the pre-coating and the application of the barrier coating. Drying is typically performed with non-contact drying, such as IR and/or hot air, or contact drying, such as a drying cylinder, or a combination of non-contact and contact drying.

[0019] The barrier coating and the optional pre-coating, if applied, may be applied in-line (also referred to as on-line). In such case, the productivity is increased by eliminating the handling operations linked to off-line treatment and by eliminating, or at least reducing, the amount of waste. When an in-line process is conducted and a pre-coating is applied, the coating weight is typically below 10 g/m² in both the barrier coating and the pre-coating to allow for sufficient drying between coating steps as well as prior to reeling.

[0020] The examples and embodiments discussed above in connection to the first and second aspects apply to the third aspect *mutatis mutandis*.

[0021] As a fourth aspect there is provided a use of a coating comprising graphene and a polyolefin copolymer bearing carboxylic acid moieties as a water vapor barrier on a paper or paperboard substrate, wherein the polyolefin copolymer bearing carboxylic acid moieties preferably is ethylene-acrylic acid copolymer (EAA) or ethylene-methacrylic-acid-copolymer (EMAA) or maleic acid ethylene copolymer (MAE).

[0022] The examples and embodiments discussed above in connection to the first, second and third aspects apply to the fourth aspect *mutatis mutandis*.

EXAMPLES

Example - coating of graphene coating on paper substrate

[0023] A paper substrate (80 g/m²) was pre-coated with SA latex (Trinseo, XZ95085.01) using a laboratory rod coater followed by drying with IR or in an oven at 110 °C.

[0024] Top-coatings were prepared from following latexes:

- SB latex with wax (BASF, Epotal 106D)

- EAA latex-1 (Michelman, Michem Prime 4983)
- EAA latex-2 (Michelman, Michem Flex HS 1130)
- SA latex (Trinseo, XZ95085.01)
- SB latex (Trinseo, XZ94378.00)
- VAcA latex (CH Polymers, CHP 118)

[0025] Barrier coatings were prepared by dispersing graphene, namely reduced graphene oxide having an oxygen content of 2.5% measured with XPS (av-70-1-2,5 from Avanzare), in respective latex. The graphene was added in a content of 1 wt% based on the dry weight of the barrier coating. The dispersion was performed by ultrasonication of the graphene in respective latex during 30 minutes.

[0026] Reference barrier coatings without any graphene were used as is. Moreover, as a reference also graphene oxide (GO) was added to an EAA latex (Michelman, Michem Prime 4983) through vigorous stirring to provide a reference barrier coating.

[0027] The barrier coatings were applied to the pre-coated paper substrates using a laboratory rod coater followed by drying in an oven at about 110 °C for about 4 minutes.

Water vapor transmission rate (WVTR)

[0028] The water vapor transmission rate (WVTR) was measured according to ISO 15106-1 at 23°C and 50% relative humidity (RH), at 30 °C and 80%RH and at 38°C and 90 % RH.

[0029] The coated paper products as well as the WVTR of those products are presented in Table 1 below.

Table 1. Coated paper products and WVTR.

Type	Graphene	GO	Pre-coating (g/m ²)	Barrier coating (g/m ²)	WVTR (g/m ² /d) at 23°C/ 50%RH	WVTR (g/m ² /d) at 30°C/ 80%RH	WVTR (g/m ² /d) at 38°C/ 90%RH
SB with wax	1.0%	0.0%	15	20			71
SB with wax	0.0%	0.0%	15	18			18
EAA-1	1.0%	0.0%	10	10	8.0	26.9	61.7
EAA-1	0.0%	0.0%	10	10	11.3	36.2	81.4
EAA-2	1.0%	0.0%	10	10	6.3	21.1	50.4
EAA-2	0.0%	0.0%	9	12	7.3	24.8	59.2
SA	1.0%	0.0%	15	11	74.9	251.5	512.2
SA	0.0%	0.0%	14	11	79.4	303.1	613.4
SB	1.0%	0.0%	15	11	29.0	96.9	218.8
SB	0.0%	0.0%	15	12	24.9	107.4	277.9
VAcA	1.0%	0.0%	15	12	91.6	341.3	701.9
VAcA	0.0%	0.0%	14	12	99.6	365.0	743.4

(continued)

Type	Graphene	GO	Pre-coating (g/m ²)	Barrier coating (g/m ²)	WVTR (g/m ² /d) at 23°C/ 50%RH	WVTR (g/m ² /d) at 30°C/ 80%RH	WVTR (g/m ² /d) at 38°C/ 90%RH
EAA-1*	0.0%	1.0%	10	11	11.8	40.9	93.3
EAA-1	0.0%	0.0%	10	10	10.7	36.4	83.6

[0030] The effect of graphene/GO-addition on WVTR measured at 38°C and 90% RH is presented also in Fig 2. The samples denoted "neat latex" are coatings based on latex without any graphene/GO-addition, whereas the samples denoted "graphene/GO-addition" are coatings based on latex with graphene or GO addition.

[0031] The best, i.e. lowest, WVTR for graphene-GO coatings were obtained for EAA-1 and EAA-2. The already inherently promising WVTR of neat EAA-1 and EAA-2 could, even though rather low from the beginning, be improved with 24% and 15% for EAA-1 and EAA-2, respectively. An improvement made even though the total coat weights of EAA-1 and EAA-2 was around 20 g/m².

[0032] For SA and SB latex a similar improvement was obtained. However, the total coat weight was much higher, in total 25-27 g/m², and the absolute WVTR values were, compared with the EAA-latexes, around ten and four times higher for SA and SB latexes. Hence, an unsatisfying barrier was yet obtained and despite the percentage improvement, the result is not considered promising.

[0033] VAcA had the highest WVTR-values both as neat latex and after the graphene had been introduced, despite that it from the beginning had the highest WVTR-values and a total coating grammage of 26-27 g/m². Moreover, the improvement of graphene was only 6 %.

[0034] As a further comparison, GO was added to EAA-1, denoted EAA-1*, and therein an increase in WVTR, of 12%, was instead obtained.

[0035] For SB with wax without any addition of graphene the lowest absolute WVTR value was obtained. However, in this case the total coat weight was 33 g/m², i.e. about 60% more coating than on the EAA-coated papers.

[0036] This shows that EAA, being a polyolefin copolymer bearing carboxylic acid moieties, is the polymer latex best suited for realizing the barrier potential of graphene in dispersion coating of paper or board.

Claims

1. A coated paper product comprising:

- a paper or paperboard substrate;
- a barrier coating, wherein the barrier coating comprises graphene and a polyolefin copolymer bearing carboxylic acid moieties, wherein the polyolefin copolymer bearing carboxylic acid moieties preferably is ethylene-acrylic acid copolymer (EAA) or ethylene-methacrylic-acid-copolymer (EMAA) or maleic acid ethylene copolymer (MAE).

2. The coated paper product of claim 1, wherein the coat weight of the barrier coating is at least 4 g/m², such as 4-15 g/m², preferably 5-10 g/m².

3. The coated paper product of claim 1 or 2, wherein the coated paper product comprises a pre-coating between the substrate and the barrier coating.

4. The coated paper product of claim 3, wherein the pre-coating comprises a polyolefin copolymer bearing carboxylic acid moieties or vinyl acetate acrylate copolymer (VAcA) or styrene-acrylate copolymer (SA) or styrene-butadiene copolymer (SB), wherein the polyolefin copolymer bearing carboxylic acid moieties preferably is ethylene-acrylic acid copolymer (EAA) or ethylene-methacrylic-acid-copolymer (EMAA) or maleic acid ethylene copolymer (MAE).

5. The coated paper product of claim 3 or 4, wherein the coat weight of the pre-coating is at least 4 g/m², such as 4-15 g/m², preferably 5-10 g/m².

6. The coated paper product of any of the preceding claims, wherein the graphene content in the barrier coating is 0.3-3 wt%, such as 0.5-2 wt%, based on the dry weight of the barrier coating.

7. The coated paper product of any one of the preceding claims, wherein the water vapor transmission rate (WVTR) measured according to ISO 15106-1 at 38°C and 90% relative humidity (RH) of the coated paper product is below 65 g/(m² day).

8. The coated paper product of any one of the preceding claims wherein the paper product is heat-sealable.

9. A packaging comprising the coated paper product according to any one of the preceding claims.

10. A method of producing a coated paper product comprising the steps of:

- providing a paper or paperboard substrate; and
- coating the substrate with a barrier coating, wherein the barrier coating comprises graphene and a polyolefin copolymer bearing carboxylic acid moieties, wherein the polyolefin copolymer bearing carboxylic acid moieties preferably is ethylene-acrylic acid copolymer (EAA) or ethylene-methacrylic-acid-copolymer (EMAA) or maleic acid ethylene copolymer (MAE).

11. The method of claim 10, wherein the barrier coating is applied in-line.

12. The method of claim 10 or 11, further comprising the step of:

- coating the substrate with a pre-coating prior to coating with the barrier coating so that the pre-coating is arranged between the substrate and the barrier coating in the coated paper product.

13. The method of claim 12 comprising drying between the pre-coating step and the barrier-coating step.

14. The method of claim 12 or 13, wherein the pre-coating is applied in-line.

15. Use of a coating comprising graphene and a polyolefin copolymer bearing carboxylic acid moieties as a water vapor barrier on a paper or paperboard substrate, wherein the polyolefin copolymer bearing carboxylic acid moieties preferably is ethylene-acrylic acid copolymer (EAA) or ethylene-methacrylic-acid-copolymer (EMAA) or maleic acid ethylene copolymer (MAE).

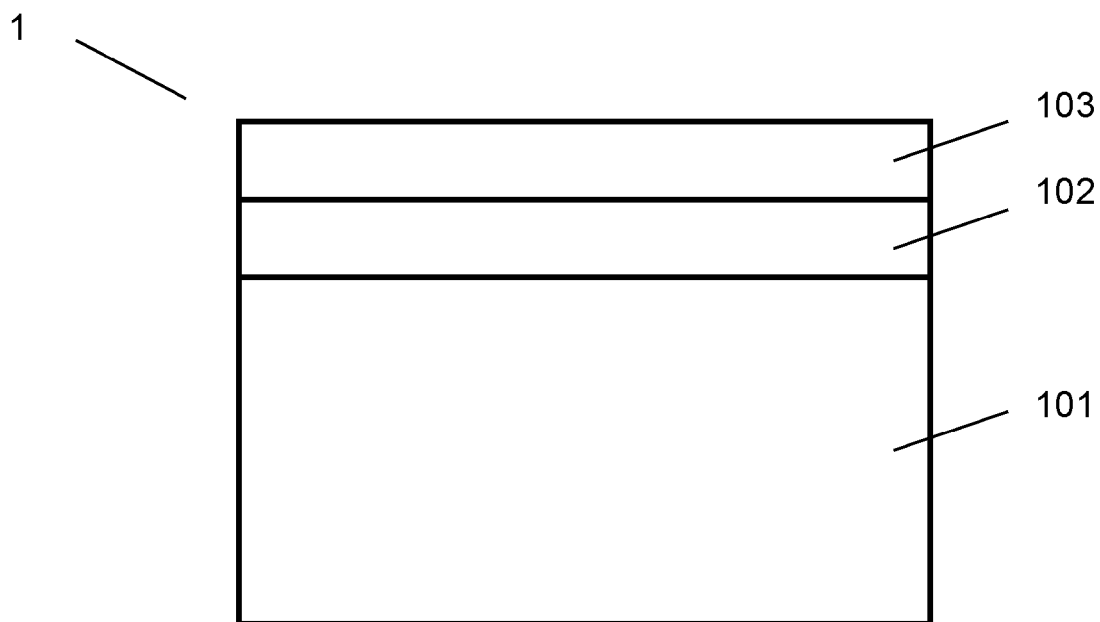


Fig. 1

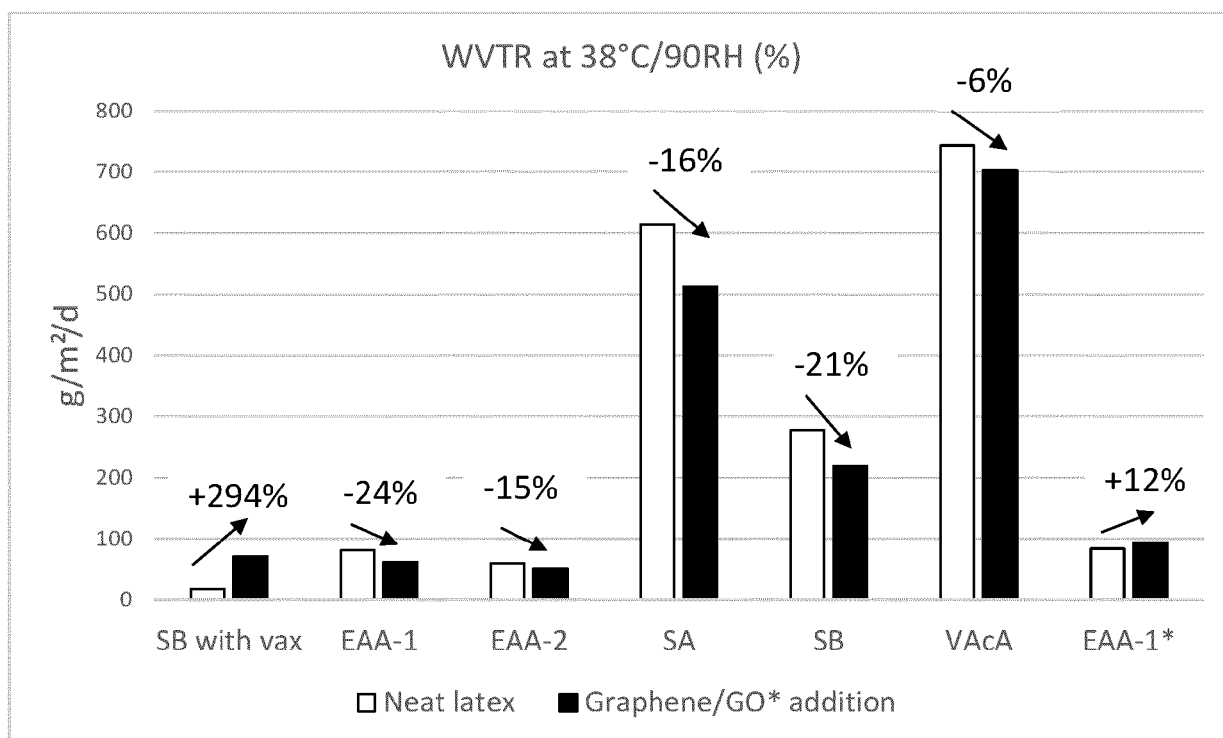


Fig. 2



EUROPEAN SEARCH REPORT

Application Number

EP 21 21 2901

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (IPC)
X	US 9 540 498 B1 (SCHEFFER DAN [US] ET AL) 10 January 2017 (2017-01-10) * claims 1,16,18 *	1-15	INV. B65D65/42 D21H17/00 D21H19/58 D21H19/64 D21H27/10
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			TECHNICAL FIELDS SEARCHED (IPC)
			B65D D21H
The present search report has been drawn up for all claims			
Place of search		Date of completion of the search	Examiner
Munich		9 May 2022	Ponsaud, Philippe
CATEGORY OF CITED DOCUMENTS		T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons	
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EPO FORM 1503 03.82 (P04C01)

**ANNEX TO THE EUROPEAN SEARCH REPORT
ON EUROPEAN PATENT APPLICATION NO.**

EP 21 21 2901

5 This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report.
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09-05-2022

10	Patent document cited in search report	Publication date	Patent family member(s)	Publication date
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