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(72) Inventors:

- **Santanocito, Adriana Maria**
95127 Catania (IT)
- **Mazzetti, Stefano**
95127 Catania (IT)
- **Merighi, Roberto**
45025 Rovigo (IT)

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(74) Representative: **Serravalle, Marco**

Marco Serravalle S.L.
Calle Galicia, 18 - Apt. E5
38660 Adeje (ES)

(71) Applicant: **Ohoskin S.r.l.**
95127 Catania (CT) (IT)

(54) **SYNTHETIC LEATHER BASED ON POLYVINYL CHLORIDE REINFORCED WITH CITRUS AND/OR PRICKLY PEAR VEGETABLE FIBRES FOR USE IN THE ROAD, RAIL, MARITIME TRANSPORT AND FURNITURE SECTORS**

(57) The invention relates to a synthetic leather based on polyvinyl chloride (PVC) reinforced with vegetable fibres derived from citrus and/or prickly pear, suitable for use in the road, rail, maritime transport and furniture sectors. The leather comprises at least three layers, wherein a first skin layer contains, per 100 parts of PVC, from 3 to 15 parts by weight of at least one vegetable fibre and from 30 to 50 parts by weight of at least one (cyclo)alkyl-based plasticiser; and second and third

layers, identical or different, contain, per 100 parts by weight of PVC, from 10 to 40 parts by weight of at least one vegetable fibre and from 80 to 120 parts by weight of at least one (cyclo)alkyl-based plasticiser, and a blowing agent. The vegetable fibres are obtained from citrus and/or prickly pear and impart improved mechanical, thermal and light resistance to the material, enabling the manufacture of eco-sustainable synthetic leathers with reduced environmental impact and high durability.

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Description**Field of the invention**

5 **[0001]** . The present invention relates to a synthetic leather based on polyvinyl chloride reinforced with citrus and/or prickly pear vegetable fibres and comprising an (cyclo)alkyl-based plasticiser. The synthetic leather according to the invention exhibits highly ecological characteristics while maintaining extremely high performance properties.

Background of the invention

10 **[0002]** . A major trend in industry in general, and in particular in the automotive, railway, maritime and furniture industries, is the use of materials, and thus finished products, made from raw materials that result in a reduced carbon footprint and minimise the consumption of natural resources.

15 **[0003]** . Polyvinyl chloride (PVC) is an extremely versatile plastic material, also used as a main component in the formulation of synthetic leathers. From a chemical standpoint, PVC is an amorphous thermoplastic polymer and therefore exhibits a characteristic structural rigidity, which is lost only above its glass transition temperature (approximately 80 °C), when it begins to soften and gradually becomes more fluid as the temperature increases. In order to render PVC liquid at lower temperatures, plasticising agents are used which interpose themselves between the polymer chains (solvation), reducing the strength of the intermolecular bonds and thereby making the PVC flexible at temperatures at which it would otherwise be too rigid (ambient temperature). Typically, up to 30% by weight of plasticiser is added relative to the PVC.

20 **[0004]** . In the automotive, railway, maritime and furniture sectors, synthetic leathers are normally based on PVC. This is because PVC ensures high durability of the leather, a feature that is essential for use in components typical of the aforementioned industries, which are often expected to last for more than ten years. In fact, the only alternative to PVC is polyurethane, which, however, degrades much more rapidly.

25 **[0005]** . It is therefore necessary to develop PVC-based synthetic leathers having a lower environmental impact. Document US 2022/025576 discloses a foamed PVC-based synthetic leather comprising, in addition to PVC, a lignocellulosic-based material and expandable microspheres.

Summary of the invention

30 **[0006]** The present invention relates to a PVC-based synthetic leather comprising at least one alkyl- or cycloalkyl-based plasticiser and at least one vegetable fibre.

Detailed description of the invention

35 **[0007]** The at least one vegetable fibre is preferably a citrus fibre and/or a prickly pear fibre, used alone or in combination, having the following characteristics. A moisture content lower than 12% by weight, preferably lower than 8% by weight. For citrus fibres, an ADL lignin (van Soest) content lower than 10% by weight, preferably lower than 7% by weight; an ADF fibre content higher than 14% by weight, preferably higher than 16% by weight; an NDF fibre content higher than 24% by weight, preferably higher than 28% by weight. For citrus sources, it is preferable to use fibres dried by the addition of calcium oxide (so-called "calcined" fibres). For citrus fibres, preferably not more than 2% by weight of the powder shall have a particle size above 400 µm.

40 **[0008]** The prickly pear fibres are preferably obtained from pads older than three years and not older than seven years, more preferably not older than five years, which are sufficiently fibrous without being excessively lignified. The prickly pear fibre has an ADL lignin content (van Soest) lower than 6% by weight, preferably lower than 5% by weight; an ADF fibre content (van Soest) higher than 20% by weight, preferably higher than 24% by weight; an NDF fibre content (van Soest) higher than 20% by weight, preferably higher than 24% by weight. For prickly pear powders (which are more fibrous and tend to form "needles" of nonuniform length), not more than 2% by weight of the powder shall have a particle size above 600 µm, preferably with an average diameter D_{50} of about 100-200 µm, more preferably about 150 µm.

50 **[0009]** As regards the plasticisers used within the scope of the invention, they must be nonvolatile, chemically inert towards PVC, stable to heat and light, and resistant to oxidation. Preferably, they should also be non-flammable, odourless and colourless. Various commercial types exist, mostly containing phthalates, which perform well but are suspected of being carcinogenic, and have been extensively investigated under a risk-assessment process conducted by the European Community and subsequently regulated under REACH, being therefore subject to strict limitations. To avoid such issues, also perceived negatively from a commercial standpoint, it is preferable to use non-phthalate plasticisers such as:

- Cyclohexane esters (DINCH): the most commonly used is the diisononyl ester of 1,2-cyclohexane dicarboxylic acid. It has an excellent toxicological profile, low viscosity, low density, excellent low-temperature flexibility, low volatility,

good migration and extraction resistance. It is soluble in most common organic solvents and is miscible and compatible with all monomeric plasticisers (for example, epoxidised fatty acid esters). It is colourless, exhibits excellent low-temperature performance, low initial viscosity and an excellent toxicological profile.

- Epoxidised plasticisers: these are esters containing an epoxide group, such as epoxidised soybean oil (ESBO), which improve heat stability in PVC articles produced by coating processes and can also act as co-stabilisers. Among the epoxidised esters, ESBO is even approved for use under food-contact regulations.

[0010] The thermally conducted reaction (150-210 °C) occurring in synthetic leather manufacturing plants (coating process) enables the formation of intramolecular bonds between the PVC and the plasticiser, which is selected to be fully miscible with the polymer, stably and homogeneously incorporated into its mass, and not prone to migrate towards the surface over time (exudation).

[0011] Stabilising substances may also be added to the liquid PVC/plasticiser mixture. Since the processing of PVC always involves a thermal step, which could otherwise degrade the polymer chains, a series of chemical compounds are preferably used to prevent such degradation and impart the desired properties to the final products; in particular, the stabilisers serve to improve:

- the resistance of PVC to atmospheric agents and ageing, especially for applications intended for outdoor use or for interiors exposed to light (e.g. cars, trains, ships, and furniture);
- the physical properties of PVC articles, particularly their mechanical resistance (to rubbing, abrasion, bending, or cutting); and
- in general, the mechanical recyclability of PVC articles at the end of their first life cycle.

[0012] The final result of the specific combination of PVC/plasticiser/stabiliser allows the finished product to exhibit:

- a density in the range of 0.50-1.35 g/cm³;
- high chemical resistance to acids, bases, and organic solvents (with the exception of ketones and aromatic compounds);
- good self-extinguishing properties;
- thermal resistance up to about 90-110 °C;
- good weldability by ultrasound, adhesion or casting; and
- no water absorption.

[0013] The finished product may also include a mixture of additives such as pigments (mainly used to impart the desired colour to the manufactured article, which may be organic- or inorganic-based, to ensure properties such as colour retention and light stability) and fillers (mostly chemically inert mineral particles incorporated into the PVC to improve flowability, miscibility, impact strength, surface gloss, flame-retardant behaviour, etc.).

[0014] It has been surprisingly found that combining PVC with at least one (cyclo)alkyl-based plasticiser and at least one vegetable fibre as defined above yields a synthetic leather having very high resistance properties (mechanical, thermal, and light resistance), particularly suitable for the automotive, nautical, railway and furniture sectors.

[0015] The PVC-based synthetic leather is a multi-layer coated material, the layers being fused together during the production process. This is achieved by coating specific PVC mixtures onto a preformed release paper designed to impart a specific surface pattern to the leather (for example, the typical grain of natural leather). In industrial production, the paper is provided in rolls several hundred metres long and is passed through multiple ovens, where the PVC thermally reacts with the plasticisers and other additives to obtain a flexible coated material which, in the final stage, is laminated onto a fabric acting as a support layer. In the final step, the paper is peeled off from the coated surface, and the synthetic leather is rolled up for subsequent processing steps (finishing and/or embossing). The coating is therefore produced in reverse order, starting from a very thin first layer (referred to as the "skin layer") in direct contact with the paper, followed by a second layer referred to as the "foam layer", which provides the structural characteristics of the coated material, and a third layer in direct contact with the fabric before oven treatment, serving as an adhesive layer. In the synthetic leather according to the present invention, the formulations of the various layers are based on the above-described vegetable plasticiser/stabiliser mixture.

[0016] The synthetic leather according to the invention is therefore composed of three layers having the following compositions: a first layer, referred to as the *skin layer*, comprising, per 100 parts of PVC, from 3 to 15 parts by weight, preferably from 5 to 10 parts by weight, of at least one vegetable fibre and from 30 to 50 parts by weight of at least one (cyclo)alkyl-based plasticiser; the second and third layers may have identical or different compositions and comprise, per 100 parts by weight of PVC, from 10 to 40 parts by weight, preferably from 15 to 35 parts by weight, of at least one vegetable fibre and from 80 to 120 parts by weight of at least one (cyclo)alkyl-based plasticiser. The second and third layers further comprise a blowing agent.

[0017] The blowing agents are reactive additives that release gas during the processing of thermoplastic materials. They therefore enable the production of foamed materials for a wide variety of applications. A thermal reaction of the additives contained in a blowing agent releases gases, allowing the formation of a stable expanded plastic material. There are two main groups of blowing agents, which differ substantially in their reaction mechanisms: endothermic agents (heat absorption), for example sodium bicarbonate and citric acid; and exothermic agents (heat release), for example azodicarbonamide, encapsulated isopentane, 5-phenyltetrazole, and benzenesulfonyl hydrazide.

[0018] In a preferred embodiment, in the first layer (skin layer), the plasticiser consists of about 50-100% by weight of a dialkyl ester of 1,2-cyclohexane dicarboxylic acid and about 0-50% by weight of an epoxidised fatty acid. Preferably, the alkyl group of the dialkyl ester is a linear or branched C₆-C₁₂ group, more preferably an isononyl group.

[0019] Preferably, the plasticiser used in the second and third layers consists of about 80-100% by weight of a dialkyl ester of 1,2-cyclohexane dicarboxylic acid and about 0-20% by weight of an epoxidised fatty acid. Preferably, the alkyl group of the dialkyl ester is a linear or branched C₆-C₁₂ group, more preferably an isononyl group.

[0020] Even more preferably, in all three layers the plasticiser consists of diisononyl ester of 1,2-cyclohexane dicarboxylic acid, optionally in admixture with epoxidised methyl esters of carboxylic acids.

Examples

Example 1 - Preparation of citrus fibre

[0021] 500 kg of calcined orange fibres, purchased in Sicily and having the following composition, were micronised using an industrial microniser with a 100 µm mesh size:

- Moisture: 5.7%
- NDF fibre: 30.4% (as is)
- ADF fibre: 16.8% (as is)
- ADL fibre: 6.8% (as is)

[0022] Particle size analysis of the resulting powder showed a modal value of 107 µm; more than 50% of the mass was between 20 and 100 µm; 98.4% of the mass was below 400 µm; and no particles above 1,000 µm were detected.

Example 2 - Preparation of prickly pear fibre

[0023] 200 kg of "mature" prickly pear pads, aged between approximately 5 and 7 years and purchased in Sicily, were ground using an agricultural shredder and then micronised as in Example 1. The composition of the resulting fibres was as follows:

- Moisture: 10.3%
- NDF fibre: 26.1% (as is)
- ADF fibre: 25.1% (as is)
- ADL fibre: 4.2% (as is)

[0024] Particle size analysis of the powder showed the following profile: modal value 136 µm; more than 50% of the mass between 20 and 160 µm; 98.6% of the mass below 600 µm; and no detectable particles above 1,000 µm.

[0025] In order to be suitable for use, the micronised fibres are preferably formulated by mixing them with the plasticiser to form a ready-to-use vegetable plasticiser/stabiliser mixture. The citrus and prickly pear fibres must be blended to obtain a mixture having a moisture content below 8%, a lignin content below 6%, and an NDF fibre content above 25%.

[0026] The optimal formulation provides a fibre content between 5 and 30%, up to a maximum of 50%, in a mixture with a phthalate-free plasticiser and/or a bioplasticiser.

Example 3 - Preparation of the fibre-plasticiser mixture

[0027] 25 grams of micronised citrus powder obtained as described in Example 1 were mixed with 10 grams of micronised prickly pear powder obtained as described in Example 2, and 65 grams of commercial diisononyl cyclohexane were added to the mixture. The mixture was vigorously homogenised using a bench homogeniser to obtain a rather dark cream. One gram of titanium dioxide was then added to the mixture to make it white.

Example 4 - Preparation of the fibre-plasticiser mixture

[0028] 25 grams of micronised citrus powder obtained as described in Example 1 were mixed with 10 grams of micronised prickly pear powder obtained as described in Example 2, and 40 grams of commercial diisononyl cyclohexane and 25 grams of epoxidised methyl esters of fatty acids were added to the mixture. The mixture was vigorously homogenised using a bench homogeniser to obtain a rather dark cream. One gram of titanium dioxide was then added to the mixture to make it white.

Example 5 - Preparation of the synthetic leather

[0029] First layer (skin): 30 grams of the mixture obtained as described in Example 3 were combined with 100 grams of commercial bio-attributed PVC (bio-attributed meaning derived from an industrial batch produced from plant-based sources, although for purely plant design reasons it cannot be certified as 100% plant-derived). To this mixture, 40 grams of commercial diisononyl cyclohexane were added, and the mixture was homogenised using a laboratory mixer. Subsequently, 7 grams of flame retardant and 6 grams of black pigment were added, yielding a fluid black mixture (skin). A thin layer (0.1 mm) of the above "skin" mixture was applied onto a preformed release paper, and the whole was placed in a laboratory oven at 150 °C for one minute.

[0030] Second layer: 120 grams of the mixture obtained as described in Example 3 were combined with 100 grams of commercial bio-attributed PVC, followed by the addition of 20 grams of commercial diisononyl cyclohexane. The mixture was homogenised using a laboratory mixer. Then, 7 grams of flame retardant, 40 grams of aluminium hydroxide, 3 grams of blowing agent, and 7 grams of black pigment were added, yielding a fluid black mixture (foam). A layer (0.25 mm) of this mixture was applied over the "skin" layer, and the whole was placed in a laboratory oven at 180 °C for one minute.

[0031] Finally, another layer of the "foam" mixture was applied, onto which a layer of recycled polyamide fabric was placed. The entire assembly was placed in a laboratory oven at 170 °C for one minute. The resulting sample had a thickness of 1.1 mm and a density of 860 g/m².

Example 6

[0032] A second sample was produced as in Example 5, except that a recycled polyethersulfone fabric was used, obtaining a product having the composition indicated in the accompanying diagram. The sample had a thickness of 1.1 mm and a density of 885 g/m².

Example 7

[0033] 120 grams of the mixture obtained as described in Example 4 were combined with 100 grams of commercial bio-attributed PVC, and 20 grams of commercial diisononyl cyclohexane were added. The mixture was homogenised using a laboratory mixer. Subsequently, 7 grams of flame retardant, 40 grams of aluminium hydroxide, 3 grams of blowing agent, and 7 grams of black pigment were added, yielding a fluid black mixture (foam II).

[0034] A third sample was thus prepared as described in Example 5, using the foam formulation defined above (foam II). The sample had a thickness of 1.1 mm and a density of 860 g/m².

Example 8

[0035] A final sample was produced as described in Example 7, except that a recycled polyethersulfone fabric was used. The sample had a thickness of 1.1 mm and a density of 885 g/m².

[0036] The samples of Example 6 were tested in accordance with the most stringent commercial specifications for the automotive, railway, furniture and nautical sectors. The analytical results confirmed that the products comply with such specifications and that citrus and/or prickly pear fibres represent a valid alternative to synthetic PVC stabilisers, enabling the production of more eco-sustainable products with a reduced environmental impact.

Table 1 - Wear and Abrasion Resistance

Test	Method / Standard	Conditions	Result
Wear resistance	Cesconi 50455	3,000 cycles	OK - No detachment or surface wear
Martindale abrasion resistance	ISO 12947-1	20,000 cycles	OK - No detachment or surface wear
Taber abrasion resistance	SAE J948, 500 g load	800 cycles	OK - No discolouration and no significant coating wear

Table 2 - Surface Resistance Properties

	Test	Method / Standard	Conditions / Medium	Result
5	Coating blocking resistance	LP.7M008	2 h at 75 ± 2 °C	OK - No blocking or colour transfer
	Colour fastness to rubbing - water and soap	50444	-	OK - Grade 5 on grey scale
10	Colour fastness to rubbing - artificial sweat	50444	-	OK - Grade 5 on grey scale
	Colour fastness to rubbing - alcohol	50444	-	OK - Grade 5 on grey scale
15	Light exposure resistance (Xenon test)	SAE J2412, Q/B filter	225 / 601 / 1240 kJ·m ⁻² exposure	OK - No colour change above grade 4 on grey scale
	Resistance to fluids	LP-463PB-31-01	Sun lotion; air fresheners; pine oil; water & soap	OK - No softening, swelling, peeling, blistering, delamination or other negative effects
20	Cleanability	LP-463KC-04-01	Grease; hot coffee; chocolate; ketchup; orange juice	OK - No softening, swelling, peeling, blistering, delamination or other negative effects

Table 3 - Thermal and Ageing Resistance

	Test	Method / Standard	Conditions	Result
25	Thermal cycling	LP-463LB-12-01, Method A	-	OK - No colour change above grade 4 on grey scale; no stickiness or embossing loss
30	Oven ageing	-	48 h at 90 ± 2 °C	OK - No visible change
	Dimensional stability	-	6 h at 85 °C	2.5 %
	Humidity resistance	-	240 h at 40 °C, 95 % RH	OK - No negative effects; no colour change above grade 4
35	Thermal ageing control panel	LP-463LB-13-01	-	OK - No negative effects; no colour change above grade 4

Claims

- 40 1. A synthetic leather based on polyvinyl chloride reinforced with citrus and/or prickly pear vegetable fibres for use in the road, rail, maritime transport and furniture sectors, said leather comprising at least three layers, wherein:
 - 45 a. a first layer, referred to as the *skin layer*, comprises, per 100 parts of PVC, from 3 to 15 parts by weight, preferably from 5 to 10 parts by weight, of at least one vegetable fibre and from 30 to 50 parts by weight of at least one (cyclo)alkyl-based plasticiser;
 - b. a second and a third layer, having identical or different compositions, comprise, per 100 parts by weight of PVC, from 10 to 40 parts by weight, preferably from 15 to 35 parts by weight, of at least one vegetable fibre and from 80 to 120 parts by weight of at least one (cyclo)alkyl-based plasticiser, and a blowing agent; and
- 50 wherein the at least one vegetable fibre is selected from a fibre derived from citrus, a fibre derived from prickly pear pads, or a mixture thereof.
2. The synthetic leather according to claim 1, wherein the vegetable fibre has a moisture content equal to or lower than 12% by weight, preferably equal to or lower than 8% by weight.
- 55 3. The synthetic leather according to any one of claims 1-2, wherein the leather contains a citrus-derived fibre having an ADL lignin content (van Soest) of less than 10% by weight, preferably less than 7% by weight; an ADF fibre content (van Soest) greater than 14% by weight, preferably greater than 16% by weight; and an NDF fibre content (van Soest)

greater than 24% by weight, preferably greater than 28% by weight.

4. The synthetic leather according to any one of claims 1-2, wherein the leather comprises a prickly pear-derived fibre obtained from pads older than three years and not older than seven years, preferably not older than five years.
5. The synthetic leather according to claim 4, wherein the prickly pear-derived fibre has an ADL lignin content according to van Soest of less than 6% by weight, preferably less than 5% by weight; an ADF fibre content according to van Soest greater than 20% by weight, preferably greater than 24% by weight; and an NDF fibre content according to van Soest greater than 20% by weight, preferably greater than 24% by weight.
6. The synthetic leather according to any one of claims 1-5, wherein the plasticiser of the first layer consists of 50-100% by weight of a dialkyl ester of 1,2-cyclohexane dicarboxylic acid and 0-50% by weight of an epoxidised fatty acid.
7. The synthetic leather according to any one of claims 1-6, wherein the plasticiser used in the second and third layers consists of 80-100% by weight of a dialkyl ester of 1,2-cyclohexane dicarboxylic acid and 0-20% by weight of an epoxidised fatty acid.
8. The synthetic leather according to any one of claims 1-7, wherein the citrus fibres are calcined fibres.
9. The synthetic leather according to any one of claims 6-7, wherein the plasticiser is a mixture of diisononyl ester of 1,2-cyclohexane dicarboxylic acid and epoxidised soybean oil.
10. The synthetic leather according to any one of claims 1-9, wherein the citrus fibres contain less than 2% by weight of particles having a diameter greater than 400 μm .



EUROPEAN SEARCH REPORT

Application Number

EP 25 21 3798

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			TECHNICAL FIELDS SEARCHED (IPC)
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The present search report has been drawn up for all claims			
Place of search		Date of completion of the search	Examiner
The Hague		12 March 2026	Rella, Giulia
CATEGORY OF CITED DOCUMENTS			
X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document			
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