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(19)



(54) FLEXIBLE, WATER-IMPERMEABLE COVERING SHEET

(71) We, HOLZSTOFF S.A., a Swiss Company, VIAFRANCE, a French Body Corporate, SOCIETE NATIONALE ELF AQUITAINE (PRODUCTION), a French Body Corporate of Malzgasse 15, 4052 Basel, Switzerland; 6, Avenue Percier, F-75008 Paris, France; Tour Aquitaine, F-92 Courbevoie, France, respectively, do hereby declare the invention for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:-

The present invention relates to a process for the manufacture of a new flexible and water-impermeable covering sheet which possesses a fibrous reinforcement and is intended to ensure complete water-impermeability in civil engineering works and building works.

The sheet obtained according to the invention differs from the products used hitherto for the same purpose by the fact that it combines perfect water-impermeability with remarkable mechanical strength properties, especially excellent resistance to tension, flexing, tear and bursting.

It is already known to combine a textile carrier with a conventional aqueous bitumen emulsion. This emulsion however does not ensure complete impermeability because of the breaking of the emulsion and elimination of the water liberated by this breaking of the emulsion. Equally, it is known that the combination of a textile carrier and a conventional bitumen applied hot does not ensure complete water-impermeability except when the proportion of bitumen is above a very high value. In fact, when the bitumen is employed in such proportions, the role of the carrier virtually disappears and one can no longer consider a combination to be involved. At lower proportions of bitumen, that is to say when one is effectively dealing with a combination, on the other hand, neither perfect water-impermeability nor satisfactory behaviour at high temperature is observed; in effect, at these temperatures the viscosity of the bitumen decreases, which causes a flow of the bitumen on the web, above all in applications in a position other than the horizontal, and in parallel therewith causes the disappearance of the homogeneity of the combination. Furthermore, the use of certain special bitumens, and especially oxidised or filled bitumens does not make it possible to produce the combinations directly on the construction site and requires factory manufacture.

Furthermore, using the products currently available on the market requires, at the stage of assembly various widths, the use of special devices and, especially, the use of gluing binders or the supply of a large amount of heat in order to ensure self-adhesion by pressure. Finally, as mentioned above, the products currently on the market do not at one and the same time exhibit the required combination of mechanical properties and complete impermeability.

The present invention provides a process which makes it possible to manufacture a new, homogeneous covering sheet which combines all the abovementioned properties. The process of the invention comprises heating to a temperature of 125 to 160°C a binder consisting of an emulsion of sulphur in bitumen in a ratio of sulphur to bitumen of between 15:85 and 30:70 by weight, the mean size of the sulphur droplets not exceeding 10 microns;

and impregnating a fibrous material uniformly at a temperature below the melting point of the said fibrous material and from 125 to 145°C with this binder ratio is between 1:0.5 and 1:60 by weight, the fibrous material weighing 10 to 1000 g/m<sup>2</sup> and consisting of (A) a non-woven fabric made of continuous or short fibres of a polyester, polyamide, polyvinyl chloride, or acrylic polymer or a copolymer of any of these materials or of an inorganic fibrous material or a mixture of two or more such fibres along or a mixture of one or more such fibres with continuous or short polyolefine fibres, or (B) a woven fabric made of continuous or short fibres of any of the materials listed in A; or (C) a composite of a woven fabric and a non-woven fabric as aforesaid. The weight ratio of the fibrous material to the binder is preferably about 1:10.

Examples of fibres used in the non-woven webs, or in the woven fabrics, or in the composites of one with the other, are given below, with some indications of the melting points or decomposition points:

Polyesters (melting point between 245 and 290°C), especially based on terephthalic acid and ethylene glycol (Dacron<sup>®</sup>, Diolen<sup>®</sup>, Terylene<sup>®</sup>, Trevira<sup>®</sup>; melting point 256°C), those polyesters resulting from the partial replacement of these components, for example by naphthalene-2, 6-dicarboxylic acid, 1,4-bis-(hydroxymethyl)-cyclohexane or 1,4-butanediol, and those based on 4-hydroxybenzoic acid;

Polyamides based especially on ε-caprolactam (Nylon 6, Perlon<sup>®</sup>, Caprolan<sup>®</sup>; melting point 215°C), on laulactam (Nylon 12), or on 1,6-hexanediamine and adipic acid (Nylon 6,6; melting point 260°C) sebacic acid, dodecanoic acid and the like;

polyvinyl chlorides as such (Clevyl R<sup>®</sup>; melting point/decomposition point about 180-200°C) and above all in the form of copolymers, in particular with acrylonitrile (modacrylic fibres; Dynel<sup>®</sup>, Vinyon N<sup>®</sup>; softening point about 150°C) and with vinyl acetate (Vinyon HH<sup>®</sup>);

polyacrylic fibres, that is to say fibres made from acrylonitrile (Orlon<sup>®</sup>, Dralon<sup>®</sup>, Acrilan<sup>®</sup>; melting point 330°C) and from its copolymers with vinyl acetate, allyl alcohols, acrylic and methacrylic esters and vinyl chloride (modacrylic fibres; see above);

inorganic fibres, especially glass fibres and asbestos fibres

finally, in the case of woven fabrics and in the case of non-woven fabrics made of a mixture of fibres of different chemical composition, polyolefines such as polypropylene (melting point 175°C) and polyethylene (melting point 135°C).

The fibrous material which is preferably employed in the process according to the invention consists of (A') a non-woven fabric made of inorganic fibres or of fibres of a polyester or polyamide, or of a mixture of fibres of any of the preceding materials with one another or with fibres of a polyolefine, of (B') a woven fabric based on fibres of a polyolefine or of any of the other materials mentioned under A', or of (C') a composite of a woven fabric and a non-woven fabric, in which the two constituents consist of fibres of identical or different materials mentioned under B'.

Furthermore, it is possible to assemble, by needle-punching, gluing or high frequency welding, non-woven webs of different gauges and especially webs of non-woven and woven fabrics, in order to achieve superior tensometric properties.

The binder is based on a modified or unmodified bitumen but is sufficiently fluid between 125°C and 160°C to permit fine dispersion of the sulphur between these temperatures. Preferably, the average size of the sulphur particles in the emulsion is about 0.5 to 5 microns. The proportion of sulphur in the binder is chosen in accordance with the required properties of the impermeable sheet and the ease of impregnation of the fibrous material. The binder can if desired be modified by adding solvent, fluxes, fillers, inorganic and organic pigments or dyestuffs, herbicidal or fungicidal compounds or other additive suitable for the use envisaged for the sheet.

To manufacture the binder it is highly desirable to proceed as described in French Patent No. 73/18,842 (published under No. 2,230,691), corresponding to British Specification No. 1448297. In this way a fine emulsion of the liquid sulphur in the liquid or pasty bitumen is obtained, in which the size of the sulphur droplets does not exceed 10 microns and is generally between 0.5 and 5 microns.

The impregnation can be carried out either on dry fibrous materials or on the same materials when wet, but treated beforehand with a specific adhesion promoter for the fibres constituting the non-woven or the woven fabric. This adhesion promoter has the effect of repelling the water from the fibres of the material, so as to allow contact with the bitumen; it consists, preferably, either of the acetate of a secondary amine or of a suspension of quaternary amine in coal tar oil, either material being preferably in a 1:10 aqueous suspension and sprayed onto the fibrous material at the rate of about 40 g per m<sup>2</sup>. As an example of an adhesion promoter of the secondary amine acetate type, there may be mentioned the alkylidipropylenetriamines of the formula R-NH-(CH<sub>2</sub>)<sub>3</sub>-NH-(CH<sub>2</sub>)<sub>3</sub>-NH<sub>2</sub>, in which R represents a long-chain aliphatic radical.

The impregnation of the fibrous material can be carried out at a temperature below the melting point of the said fibrous material and from 125° to 145°C, preferably from 130 to 140°C, by spraying, by dipping or by coating. Of course if the binder has previously been heated to a temperature between 145° and 160°C, it is necessary to allow it to cool to 145°C before the impregnation is carried out.

Spraying may be carried out at a temperature of about 140°C and under a pressure of 4 to 6 bars, through special vortex nozzles. These nozzles are mounted on a frame with constant spacing of the nozzles, so chosen as to ensure good transverse distribution. This frame is of the double-wall heated frame type. The material is delivered either by means of a constant pressure of a neutral gas or by means of a metering pump or by a system of a pump and discharge valves. The frame is placed 40 cm above the material to be treated. For impregnation by dipping, the fibrous material may be passed through one or more troughs containing the binder and kept at a temperature of 135 to 145°C. In certain cases, it is preferably to carry out the impregnation by coating, at a temperature of 125 to 145°C. Various conventional coating techniques, such as knife coating, can be used for this purpose.

In order to facilitate the subsequent unwinding of the covering sheet, when it is being applied, the sheet can be provided with a non-adhesive protective film as the last stage of the manufacturing process.

Using the bitumen/sulphur binder described above results in the following advantages:

Firstly, a lowering of the use temperature, due to the fluxing or fluidising power of the sulphur which, at the same time, permits better penetration of the binder into the interior of the fibrous material; and

secondly, progressive crosslinking, due to the partial crystallisation of the sulphur, which reduces the sensitivity of the binder to rises in temperature and consequently avoids migration of the binder both inside the fibrous material and at the surface.

A microscopic study of the sheet manufactured according to the invention, bearing on the question of crystallisation in a bitumen/sulphur medium, in effect shows a genuine entangling between the fibres or filaments and the crystallites [see the attached figure; a) fibres or filaments, b) entangled crystallised materials].

In practice, it has been found that the phenomena of migration of the binder on the fibrous material are greatly reduced from the start of applying the sheet and completely eliminated after a few hours, as a result of the crystallisation of the sulphur between the fibres. The entangling which results therefrom imparts to the covering sheet a homogeneous structure and a high degree of cohesion, which manifest themselves in increased flexibility and increased mechanical strength.

On the other hand, the lowering of the viscosity at a given temperature makes it possible to impregnate the fibrous material at temperatures which are sufficiently low not to damage the material and which can even come down to 125°C. Furthermore, it thus becomes possible to place the binder under high pressure (4 to 6 bars) without causing it to deteriorate, which makes it possible to spray the fibrous material under high pressure and hence to manufacture the covering sheet on site.

The required widths can be assembled either by overlapping or by applying a joint covering strip, that is to say a strip of the same type applied onto the contiguous ends of the widths.

The new covering sheets self-adhere to one another under pressure in the cold, at a temperature of 16°C or above, as a result of the fluxing effect of the sulphur in the binder. This property makes it possible to avoid the use of glue or the application of a large amount of heat for joining up the widths.

Summarising, the covering sheet obtained according to the invention is distinguished, in respect of its manufacture and its application, by the fact that it can be prepared at a relatively low temperature and on site (as a result of the stability of the binder, which withstands high pressure spraying) and that it is self-adhesive to itself in the cold. As regard its effects, the new sheet exhibits complete impermeability to water, coupled with the absence of migration of the binder when the material is applied in positions other than horizontal, and remarkable adaptation to deformation of the carriers to which it is applied, whether this deformation be shearing, punching, flexing, bursting, tearing or tension.

It must be emphasised here that the process according to the invention makes it possible to employ any bitumen, that is to say it is not limited to soft bitumens. In fact, soft bitumens have already been used to impregnate fibrous materials of all kinds at relatively low temperatures, of about 140°C, but the products thus obtained did not exhibit fully satisfactory mechanical strength properties, which consequently limited their use; only applications where these products were not exposed to major mechanical forces were possible. On the other hand, it was also known to impregnate certain fibrous materials with hard bitumens; in this way, impermeable membranes exhibiting excellent mechanical

properties were obtained. However, the need to work at relatively high temperatures, of about 200°C, limited the use of the hard bitumens to fibrous materials having higher melting points or decomposition points; furthermore, it created difficulties, which were sometimes considerable, when the material was applied.

5 Surprisingly, the process according to the invention now makes it possible to achieve 5  
 what has hitherto been proved impossible, namely to manufacture, from soft bitumens, impermeable sheets possessing very high mechanical strength, but also to use hard bitumens at lower temperatures and thus to impregnate fibrous materials of all kinds without danger of damage to the material and without difficulties when the material is 10  
 applied. This result constitutes a doubly remarkable technical advance; it is obviously due 10  
 to the use of the particular binder described above. However, it was not to be expected that the recrystallisation of sulphur within the sheet, after application and cooling, would manifest itself in a decisive improvement of the mechanical properties, and, furthermore, it was not possible to foresee that the addition of sulphur to hard bitumens would exert a 15  
 fluxing effect such that the use temperature was as a result lowered by about 60 to 80°C. 15

The covering sheet described above makes it possible to produce main (water) barriers and barriers to trickling water, for civil engineering work or building work, below or above ground and, furthermore to provide a reinforcement effect or support effect for certain parts of the work. In particular, it makes it possible to produce reinforcements suitable for 20  
 various structures employed in civil engineering and in building, ensuring the impermeability and bonding of the various layers which constitute the structures. 20

The mechanical strength of the structures is improved, and durable carrier layers and bonding layers can be produced.

In the examples which follow, the products usually sold commercially were used as 25  
 fibrous material. Soft bitumens are to be understood as bitumens having a high penetration value (for example 80-100, 100-120 or 180-200), whilst hard bitumens are to be understood as bitumens having a low penetration value (for example 20-30 or 40-50). In each Example, the emulsion of sulphur in bitumen was first heated to a temperature in the range of 125° to 160°C. and then sprayed on to the fibrous material at a temperature of about 140°C. 30  
 30

#### Example 1

The fibrous material is a nonwoven of continuous filaments based on polyamide 6,6; filament gauge: 15 dtex; weight of the web after needle-punching: 350 g/m<sup>2</sup>. For results of the tests, see Table I below. In this table, as well as in the following tables, the abbreviation 35  
 ESB 30:70 denotes an emulsion of sulphur in bitumen in a weight ratio of 30 to 70, whilst the entry 80-100 defines the type of bitumen employed. 35

#### Example 2

The fibrous material is a nonwoven of continuous filaments (double-component fibres) of 40  
 polypropylene and polyamide in a weight ratio of the two components of 70 to 30. The filaments are bonded to one another by heat-bonding, effected in a heat calender; the final web weighs 250 g/m<sup>2</sup>. For the results of the tests, see Table II. 40

#### Example 3

The fibrous material is a nonwoven of short fibres, of gauge 2 dtex, based on polyvinyl 45  
 chloride (Clévy T, registered trademark of Rhône-Poulenc Textiles); weight of the web after needle-punching: 260 g/m<sup>2</sup>. For the results of the tests, see Table III. 45

#### Example 4

The fibrous material is a glass mat weighing 450 g/m<sup>2</sup>. For results of the tests, see Table 50  
 IV. 50

A glass mat is to be understood as a felt of glass fibres, cut to 25 and 50 mm length and agglomerated by means of a binder. It serves to produce laminates possessing average mechanical strength, without preferential orientation of the fibres. 55  
 55

#### Example 5

The fibrous material is a woven fabric consisting of short fibres of polyester; weight of fabric 140 g/m<sup>2</sup>. For results of the tests, see Table V.

60 Example 6 60

The fibrous material is a woven fabric based on yarns composed of short fibres of polyamides 6,6; weight of fabric 130 g/m<sup>2</sup>. For results of the tests, see Table VI.

#### Example 7

65 The fibrous material is a composite of a non-woven of continuous filaments of polyester - 65

filament gauge 8 dtex, weight of the web: 350 g/m<sup>2</sup>, and of a woven fabric based on yarns composed of continuous filaments of polypropylene - weight of the fabric: 115 g/m<sup>2</sup>. The non-woven web and the woven fabric are bonded to one another by needle-punching. For results of the tests, see Table VII.

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*Example 8*

The fibrous material is a needle-punched non-woven, of continuous filaments of polyester - filament gauge 8dtex, weight of the web: 300 g/m<sup>2</sup>. For results of the tests, see Table VIII.

TABLE I

(Needle-punched nonwoven of continuous filaments of polyamide 6.6; 350 g/m<sup>2</sup>)

| Definition of the test                                      | Virgin fibrous material (without binder) | After spraying with ESB 30:70 (80-100) on both faces, 2 kg/m <sup>2</sup> per face |
|---|--|--|
| Impermeability test under water pressure at 20°C            | not relevant                             | impermeable  |
| on a non-deformable porous carrier                          | 2 bars pressure                          | impermeable  |
| on a deformable porous carrier                              | 4 bars pressure                          | impermeable  |
| extrusion and bursting test under water pressure            | 2 bars pressure                          | impermeable  |
|   | 4 bars pressure                          | impermeable  |
|   | on full width                            | bursts at 8 bars   |
|   | on joint                                 | bursts at 5 bars   |
| strength and deformation at break; speed: 1 mm/sec. at 20°C | 16.6                                     | 21.1   |
| breaking load in kg/cm                                      | 110                                      | 120  |
| elongation at break in %                                    | 18.7                                     | 23.5   |
| necking at break in %                                       | 28.0                                     | 30.0   |
| Tear propagation resistance in kg, speed: 1 mm/sec. at 20°C |  |  |

TABLE II

(Heat-bonded nonwoven of continuous filaments of polypropylene/polyamide, 70/30; 250 g/m<sup>2</sup>)

| Definition of the test  | Virgin fibrous material (without binder) | After spraying with ESB 30:70 (80-100) on both faces, 2 kg/m <sup>2</sup> per face |
|---|--|--|
| on a non-deformable porous carrier                                  | 2 bars pressure                          | impermeable  |
| on a deformable porous carrier                                      | 4 bars pressure                          | impermeable  |
| extrusion and bursting test under water pressure at 20°C            | 2 bars pressure                          | impermeable  |
| Impermeability test   | 4 bars pressure                          | leaks at 5 litres/hour   |
|   | on full width                            | bursts at 8 bars   |
|   | on joint                                 | bursts at 5 bars   |
|   | 14.4                                     | 28.6   |
| breaking load in kg/cm  | 90                                       | 110  |
| elongation at break in %  | 51                                       | 68   |
| necking at break in %   | 6.5                                      | 15.0   |
| Tensile strength and deformation at break; speed: 1 mm/sec. at 20°C |  |  |
| Tear propagation resistance in kg, speed: 1 mm/sec. at 20°C         |  |  |

TABLE III

(Needle-punched nonwoven of short fibres of polyvinyl chloride; 260 g/m<sup>2</sup>)

| Definition of the test  | Virgin fibrous material (without binder) | After spraying with ESB 30:70 (80-100) on both faces, 2 kg/m <sup>2</sup> per face |
|---|--|--|
| on a non-deformable porous carrier                                  | 2 bars pressure                          | impermeable  |
| on a deformable porous carrier                                      | 4 bars pressure                          | impermeable  |
| extrusion and bursting test under water pressure                    | 2 bars pressure                          | impermeable  |
|   | 4 bars pressure                          | impermeable  |
| Impermeability test at 20°C   | on full width                            | bursts at 5 bars   |
|   | on joint                                 | bursts at 3 bars   |
| breaking load in kg/cm  | 10.8                                     | 18.7   |
| elongation at break in %  | 114                                      | 130  |
| necking at break in %   | 27                                       | 58   |
| Tensile strength and deformation at break; speed: 1 mm/sec. at 20°C | 8.8                                      | 12.0   |
| Tear propagation resistance in kg, speed: 1 mm/sec. at 20°C         |  |  |

TABLE IV

(Glass mat; 450 g/m<sup>2</sup>)

| Definition of the test  | Virgin fibrous material (without binder)  | After spraying with ESB 30:70 (80-100) on both faces, 2 kg/m <sup>2</sup> per face |
|---|---|--|
| on a non-deformable porous carrier                                  | not relevant                              | impermeable  |
| on a deformable porous carrier                                      | not relevant                              | peripheral leakage rate 23 litres/hour   |
| extrusion and bursting test under water pressure at 20°C            | not relevant                              | impermeable  |
| Impermeability test   | not relevant                              | peripheral leakage rate 27 litres/hour   |
| strength and deformation at break; speed: 1 mm/sec. at 20°C         | not relevant                              | bursts at 12 bars  |
| breaking load in kg/cm  | not relevant                              | bursts at 8 bars   |
| elongation at break in %  | not measurable (lower limit of apparatus) | 9.6  |
| necking at break in %   | 21  | 30   |
| Tensile strength and deformation at break; speed: 1 mm/sec. at 20°C | 0   | 48   |
| Tear propagation resistance in kg, speed: 1 mm/sec. at 20°C         | not measurable (lower limit of apparatus) | 35.0   |

TABLE V  
(Woven fabric of short fibres of polyester, 130 g/m<sup>2</sup>)

| Definition of the test  | Virgin fibrous material (without binder) | After spraying with ESB 30:70 (80-100) on both faces, 2 kg/m <sup>2</sup> per face |
|---|--|--|
| Impermeability test under water pressure at 20°C                    | not relevant                             | impermeable  |
| on a non-deformable porous carrier                                  | 2 bars pressure                          | impermeable  |
| on a deformable porous carrier                                      | 4 bars pressure                          | impermeable  |
| extrusion and bursting test under water pressure                    | 2 bars pressure                          | leaks at 0.7 litre/hour  |
|   | 4 bars pressure                          | burst at 8 bars  |
|   | on full width                            | bursts at 7 bars   |
|   | on joint                                 | 31.6   |
| Tensile strength and deformation at break; speed: 1 mm/sec. at 20°C | 26.8                                     | 55   |
| breaking load in kg/cm  | 49                                       | 14   |
| elongation at break in %  | 24                                       | 14.0   |
| necking at break in %   | 12.0                                     |  |
| Tear propagation resistance in kg, speed: 1 mm/sec. at 20°C         |  |  |

TABLE VI

(Woven fabric of short fibres of polyester 6.6; 130 g/m<sup>2</sup>)

| Definition of the test  | Virgin fibrous material (without binder) | After spraying with ESB 30:70 (80-100) on both faces, 2 kg/m <sup>2</sup> per face |
|---|--|--|
| Impermeability test under water pressure at 20°C                    | not relevant                             | impermeable  |
| on a non-deformable porous carrier                                  | 2 bars pressure                          | impermeable  |
| on a deformable porous carrier                                      | 4 bars pressure                          | impermeable  |
| extrusion and bursting test under water pressure                    | 2 bars pressure                          | impermeable  |
|   | 4 bars pressure                          | leaks at 3 litres/hour   |
|   | on full width                            | bursts at 8 bars   |
|   | on joint                                 | bursts at 6 bars   |
| Tensile strength and deformation at break; speed: 1 mm/sec. at 20°C | 27                                       | 30   |
| breaking load in kg/cm  | 10.5                                     | 15.0   |
| elongation at break in %  | 16                                       | 12   |
| necking at break in %   | 12.0                                     | 16.0   |
| Tear propagation resistance in kg, speed: 1 mm/sec. at 20°C         |  |  |

TABLE VII

(Laminate of a nonwoven of continuous filaments of polyester, 350 g/m<sup>2</sup>, and of a woven fabric of continuous filaments of polypropylene, 115 g/m<sup>2</sup>; needle-punched)

| Definition of the test  | Virgin fibrous material (without binder) | After spraying with ESB 30:70 (80-100) on both faces, 2 kg/m <sup>2</sup> per face |
|---|--|--|
| on a non-deformable porous carrier                                  | 2 bars pressure                          | impermeable  |
| on a deformable porous carrier                                      | 4 bars pressure                          | impermeable  |
| extrusion and bursting test under water pressure                    | 2 bars pressure                          | impermeable  |
|   | 4 bars pressure                          | impermeable  |
| Impermeability test under water pressure at 20°C                    | on full width                            | bursts at 21 bars  |
|   | on joint                                 | bursts at 18 bars  |
| Tensile strength and deformation at break; speed: 1 mm/sec. at 20°C | breaking load in kg/cm                   | 94   |
|   | elongation at break in %                 | 25   |
| Tear propagation resistance in kg. speed: 1 mm/sec. at 20°C         | necking at break in %                    | 17   |
|   |  | 20.0   |

TABLE VIII

(Needle-punched nonwoven of continuous filaments of polyester, 30 g/m<sup>2</sup>)

| Definition of the test  | Virgin fibrous material (without binder) | After spraying with ESB 30:70 (40-50) on both faces, 2 kg/m <sup>2</sup> per face |
|---|--|---|
| Impervability test under water pressure at 20°C                     | 2 bars pressure                          | impermeable   |
| on a non-deformable porous carrier                                  | not relevant                             | impermeable   |
| on a deformable porous carrier                                      | 4 bars pressure                          | impermeable   |
| extrusion and bursting test under water pressure                    | 2 bars pressure                          | impermeable   |
|   | 4 bars pressure                          | impermeable   |
|   | on full width                            | bursts at 11 bars   |
|   | on joint                                 | bursts at 8 bars  |
| Tensile strength and deformation at break; speed: 1 mm/sec. at 20°C | 11                                       | 19  |
| breaking load in kg/cm  | 65                                       | 75  |
| elongation at break in %  | 20                                       | 25  |
| necking at break in %   | 20                                       | 26  |
| Tear propagation resistance in kg, speed: 1mm/sec. at 20°C          |  |   |

## WHAT WE CLAIM IS:-

1. A process for the preparation of a flexible water-impermeable covering sheet with a fibrous reinforcement, which comprises heating to a temperature of 125 to 160°C a binder consisting of an emulsion of sulphur in bitumen in a ratio of sulphur to bitumen of between 5 15:85 and 30:70 by weight, the mean size of the sulphur droplets not exceeding 10 microns; and impregnating a fibrous material uniformly at a temperature below the melting point of the said fibrous material and from 125 to 145°C with this binder to give an impregnated sheet in which the fibrous material to binder ratio is between 1:0.5 and 1:60 by weight, the 10 fibrous material weighing 10 to 1000 g/m<sup>2</sup> and consisting of (A) a non-woven fabric made of continuous or short fibres of a polyester, polyamide, polyvinyl chloride, or acrylic polymer or a copolymer of any of these materials of an inorganic fibrous material or a mixture of two or more such fibres alone or a mixture of one or more such fibres with continuous or short polyolefine fibres, or (B) a woven fabric made of continuous or short fibres of any of the materials listed in A; or (C) a composite of a woven fabric and a non-woven fabric as 15 aforesaid
2. A process according to claim 1 in which the weight ratio of fibrous material to binder is about 1:10 by weight.
3. A process according to claim 1 or 2, in which the fibrous material consists of (A') a 20 non-woven fabric made of inorganic fibres or of fibres of a polyester or polyamide, or of a mixture of fibres of any of the preceding materials with one another or with fibres of a polyolefine, of (B') a woven fabric based on fibres of a polyolefine or of any of the other materials mentioned under A', or of (C') a composite of a woven fabric and a non-woven fabric, in which the two constituents consist of fibres of identical or different materials mentioned under B'.
4. A process according to any of claims 1 to 3, in which wet fibrous material is 25 impregnated, and the impregnation is preceded by a treatment of the fibrous material with an adhesion promoter consisting either of the acetate of a secondary amine or of a suspension of a quaternary amine in coal tar oil.
5. A process according to claim 4 in which the said promoter is sprayed, as a 1:10 30 aqueous suspension, onto the fibrous material, at the rate of about 40 g. per m<sup>2</sup>.
6. A process according to any of claims 1 to 5, in which the impregnation of the fibrous material is carried out by spraying the binder at a temperature of 140°C. and under a pressure of 4 to 6 bars.
7. A process according to any of claims 1 to 5, in which the impregnation of the fibrous 35 material is carried out by dipping in one or more troughs containing the binder at a temperature of 135 to 145°C.
8. A process according to any of claims 1 to 5, in which the impregnation of the fibrous material is carried out by knife-coating at a temperature of 125 to 145°C.
9. A process according to any of claims 1 to 8 in which the said binder contains one or 40 more dyestuffs or pigments, herbicidal or fungicidal compounds, solvents, fluxes and/or fillers.
10. A process according to claim 1 substantially as described in any one of the foregoing Examples.
11. A flexible, water-impermeable covering sheet with a fibrous reinforcement, 45 manufactured by the process according to any one of claims 1 to 10.

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