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## WEB AND BITUMINIZED ARTICLE MADE THEREFROM AND PROCESS OF MAKING SAME

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This invention relates to webs and bituminized articles made therefrom and to the process of making the same. It also relates to fibrous webs or felts which are impregnated with bituminous saturants or other impregnating materials and which are designed to afford roofing, flooring and other building materials and the like.

It is an object of this invention to afford web material which has high flexibility and tear-resistance and at the same time possesses high tensile strength and toughness.

It is a further object of this invention to afford a bituminized web to be used for roofing, flooring and the like which has the combined properties of high tear-resistance, high flexibility, toughness, high tensile strength and good weathering and wearing properties.

There are on the market at the present time certain types of wood fiber which will be referred to herein as "purified fiber." In physical properties the purified fiber is notable for its flexibility, softness, high absorbency and resistance to weathering or deterioration under atmospheric influences. In chemical properties purified fiber is distinguished by a relatively high $\alpha$ cellulose content, a relatively low content of $\beta$ and $\gamma$ celluloses, a low lignin content and a pentosan content approximating that found in unbleached coniferous sulphite pulps. One type of purified fiber which may be used for purposes of illustration herein has an $\alpha$ cellulose content of about 93% or more, about 0.1% to 4% each of the $\beta$ and $\gamma$ celluloses, a lignin content usually less than about 1%, a pentosan content of about 1% to 4% and a resin content of usually less than about 0.5%.

We have found that when purified fiber is mixed with suitable unpurified fiber, and a web is then made up from the mixed fiber as described herein, the resultant web has properties which are different from and are distinctly superior to, for many purposes, the properties of webs which are composed substantially of purified fiber on the one hand or unpurified fiber on the other.

"Unpurified fiber" as that term is used herein refers to the ordinary types of kraft and sulphite pulps, kraft paper scrap, felt-maker's rags, cotton mill waste, cotton linters, wool carpet clippings and the like.

Improvements in webs, bituminized or otherwise can be best recognized by the degree to which they respond to certain tests for determining such characteristics as tear-resistance, bursting strength, resistance to failure under flexing, tensile strength, elongation, toughness and resistance to indentation. Such tests are well known in the art and complete descriptions of apparatus used and methods of making tests on felts, and bituminized roofings and floorings will be found in "Asphalts and Allied Substances" by Abraham, 1929 edition, and in "Paper Testing Methods" published by the Technical Association of the Pulp and Paper Industry, 1929 edition. Brief reference to certain tests will, however, be made herein as follows:

Tear-resistance as given herein is determined by using the standard Elmendorf tear tester, the results being given in grams, for a single thickness of the sheet. The test is to be assumed to be taken at 77° F. unless otherwise stated.

Bursting strength is determined by the standard Mullen tester, in which a sheet of the material is subjected at about 77° F. to tension uniformly increased on a one square inch area by pressure applied to a rubber diaphragm under a uniform rate of displacement of an incompressible fluid, until the sample ruptures.

Resistance to failure under flexing may be determined in several ways, as hand flexing or bending around mandrels of various diameters, particularly at low temperatures. The Reeve and Yeager flexibility tester is a suitable instrument for such tests.

Tensile strength is determined by the use of standard Amsler or Scott tensile testing machines. Samples 2 inches wide by 5 inches long clamped between the jaws are stressed in tension. The maximum load sustained by the material expressed in pounds per inch of width of the sample or per square inch of original cross-section is taken as the tensile strength.

In testing the material on the Amsler or Scott testing machine as above described the elongation up to maximum tensile load, in per cent, is the "elongation" value. The material in most cases continues to elongate after maximum tensile load is reached, until the breaking point is reached. Elongation up to the breaking point in per cent is taken as the "total elongation."

The work done in bringing the material up to maximum load is taken as the toughness value.

It may be calculated from the area between the stress-strain curve and elongation axis up to the maximum load point and is expressed as work absorbed per cubic inch of material.

Resistance to indentation is determined by placing a sample of the bituminized web on a piece of sponge rubber sheet ½ inch thick and then subjecting the sample for one hour to a ⅜ inch diameter steel ball-bearing supporting a vertically imposed load of 5 kilograms. The depth of indentation of the ball into the sample measured in millimeters is taken as the "indentation" value.

A customary test for unsaturated webs is the kerosene absorption test. A weighed oven-dried sample is immersed in kerosene for 5 minutes, the excess kerosene on the surface is carefully removed with the edge of a blotter and the sample weighed immediately. The difference in weight of the saturated and unsaturated sample divided by the weight of the unsaturated sample, multiplied by 100 is the percent kerosene absorption.

The purpose of the kerosene absorption test is to determine the absorbency or porosity of the web, with particular reference to its "saturating capacity" for asphalt or other thermoplastic impregnating materials.

This invention will now be illustrated in connection with certain specific examples of the combination in webs of purified and unpurified fibers according to this invention.

This invention will first be exemplified in connection with a water-laid felted web containing about 60% of purified fiber and about 40% of kraft fiber. In the manufacture of the web the purified fiber is preferably beaten separately until the lumps are substantially removed and the fibers are thoroughly distributed. Excessive beating beyond what is necessary to distribute the fiber and remove lumps is preferably avoided. Since in preferred practice the beating is only enough to distribute the fiber and remove lumps, such fiber may be referred to herein as "substantially unreduced" to distinguish such fiber from fiber which has been subjected to more prolonged beating that results in substantial reduction in size of the fiber. The kraft fiber is preferably beaten until the fiber has been reduced to a furnish which would be satisfactory in making kraft paper. The purified fiber and kraft fiber are then blended in desired proportions. The mixed fiber furnish, at suitable dilution, is then taken to a paper-making vat wherein an interfelted web is formed on a paper-making screen cylinder as is well known in the art. A Fourdrinier paper machine may also be used for formation of the web. The web is then subjected to rolling and drying mechanisms which need not be described as numerous types of such mechanisms are well known in the art.

By adjusting the dilution of the fiber in the cylinder vat, the cylinder water level differential and/or the pressure on the couch roll and press rolls the density of the felt or index of compactness can be controlled. The index of compactness is computed by the following formula:

$$\frac{\text{caliper of the sheet in mils}}{\text{felt number}} = \text{index of compactness}$$

wherein the felt number is the weight in pounds of 480 square feet of felt. The greater the numerical value of the compactness index the less compact and more absorbent is the felt.

In one example a felted web is made up having an index of compactness of about 1.04 (i. e. having a felt number of 68.0 and a caliper of .071 inch). The sheet is formed under a pressure of about 30 pounds per square inch. The dry felt (60% purified fiber and 40% kraft fiber) is found to have the following characteristics as compared with a felt made 100% from purified wood fiber under similar conditions during initial formation of the wet web, but subjected thereafter to a pressure of only about 10 lbs. per square inch.

| | 100% purified fiber | 60% purified—40% kraft fiber |
|---|---|---|
| Tear-resistance, grams | 140 | 926 |
| Mullen strength, lbs. | 26 | 121 |
| Compactness index | 1.34 | 1.04 |
| Kerosene absorption | 235 | 121 |
| Indentation (millimeters) | 2.8 | 1.7 |
| Tensile strength, lbs. per inch width | 24 | 108 |

The web made up and formed as above described is thus seen to have very high tear-resistance and Mullen strength. When compared with felt made up using 100% purified fiber under similar conditions, the new web is found to have over six times the tear-resistance and over four times the Mullen strength of web composed of 100% purified fiber. Moreover, the new web shows about 40% less kerosene absorption and also about 40% less indentation. The new web also has a high degree of flexibility and resistance to cracking when flexed.

The combination of purified cellulose wood fiber which is low in lignin and pentosan and which is soft and absorbent, with a wood fiber containing higher lignin and/or pentosans and which is hard and stiff, as described herein, appears to impart to the felted web the unusual combination of characteristics above described. Thus in the above illustration the kraft fiber mentioned has a lignin content of about 4% to about 8% and a pentosan content of about 6% to about 9%. In the case of wood fiber, fiber having more than about 6% pentosan and/or about 4% lignin is preferable as hard type fiber for addition to purified fiber.

While relatively high lignin and/or pentosans are believed to impart stiffness and hardness to unpurified wood fibers, particularly those liberated by alkaline treatment, fiber of hard type may be used which is stiff and hard by virtue of other characteristics and illustrations of such additional hard type fibers will be given below. Thus, while specific mention of kraft fiber is made in the above example, other hard type fiber may also be used instead of kraft fiber as will be disclosed more in detail below.

While a specific proportion of purified and kraft fiber and a specific index of compactness are mentioned above, it is to be understood that this has been done for purposes of illustration only. Thus by reducing the water level differential in the cylinder vat or by lowering the pressure on the couch rolls the index of compactness can be increased. Thus a pressure of 10 pounds per square inch gives a web which is less compact and more porous. Variation in proportions of purified cellulose wood fiber and unpurified fiber may be made within the scope of this invention. Usually, however, a web containing about 50% or more of purified fiber and about 15% to about 50% of hard type fiber such as kraft fiber gives best results.

In making interfelted water-laid web material

any grade of purified or unpurified fiber suitable for making felts and papers may be employed.

Web material, particularly water-laid felts, containing mixtures of substantial proportions of purified fiber and of unpurified fiber and in an unimpregnated condition, are to be regarded as a feature of this invention. Moreover, such webs, when impregnated with a saturating compound or compounds such as bituminous cements, hydraulic cements, rubber latex, resins, varnishes or other binders such as sodium silicate and the like, also exhibit remarkably improved properties and are to be regarded as being within the scope of this invention. The employment of the purified fiber in major proportions and the blending of the purified fiber with hard type fiber such as kraft are further features of this invention.

When web material embodying this invention is bituminized, as in the manufacture of roofing or flooring materials, the bituminized web has new and improved properties, but not in the direct ratio found in the unsaturated webs and such bituminized webs are to be regarded as a particular feature of this invention. This will be brought out in connection with specific examples of bituminized webs embodying this invention to be given below.

In one embodiment of this invention which is illustrative thereof, a bituminized web is made up which is suitable, for example, as a roofing material and which contains about 60% of purified fiber, about 30% of kraft fiber and 10% wool carpet clippings. In the manufacture of bituminized felts suitable for roofing according to this invention, it is preferable to employ felt having an index of compactness of about 1.20 to about 1.40. This felt can be made up as above described. The felt after having been formed is then impregnated with bituminous saturant. In the concrete example here given a web having an index of compactness of about 1.30 is impregnated with a saturant having a softening point of about 100° F. (by the ball and ring method) and a penetration of about 125 at 77° F. The saturant is preferably applied at a temperature of about 400° F.

The impregnation operation may be carried out by any suitable apparatus of which several types are known. The felt will be found to absorb the bituminous saturant much more readily than does ordinary roofing felt and to take up about 225% to 275% of asphalt on the weight of the dry felt. These figures have been found to be preferable in the manufacture of roofing described herein. While the fibrous web could be made so soft and porous as to absorb still further quantities of saturant, it has been found that further quantities of saturant result in decreased mechanical properties such as tear-resistance and Mullen strength, accompanied by excessive elongation under tensile loads. A relatively high proportion of saturant such as within the range of the proportions mentioned, is, however, desirable in affording roofing having very high resistance to weathering. In the illustration given there is absorbed in the web about 240% of saturant.

The saturated felt is then treated with a bituminous coating on both sides, having, for example, a softening point of about 235% F. and a penetration of about 13 at 77° F. The coating material is maintained at about 375° F. for the coating operation. Flake mica is applied as a surface mineral layer on the finished roofing.

The roofing material made as above described, to a finished weight of about 75 lbs. per 108 sq. ft., is found to have the following characteristics as determined by tests above described, in comparison with rag felt roofing of high quality.

| | High grade rag felt roofing | Purified wood fiber-kraft-wool felt roofing |
|---|---|---|
| Tear-resistance per 001" thickness of base felt, average, grams | 33.5 | 57.5 |
| Bursting strength (Mullen), lbs. | 193 | 353 |
| Tensile xtrength, average, lbs. per inch width | 67.5 | 86.5 |
| Elongation, at maximum tensile load, average | 6.6% | 10.2% |
| Toughness index, inch. lbs. | 35.5 | 46.0 |

The tear-resistance of the bituminized web is relatively high not only at 77° F. but at lower temperatures as well. Thus breaking and cracking of roofing materials when handled at low temperatures is reduced.

The foregoing data show that the roofing thus produced has a very high tear-resistance and a high bursting strength (Mullen). The strength, elongation and toughness values are very much greater than those of bituminized felt roofings heretofore manufactured. Properties such as these afford roofing which is highly resistant to tearing during application and to pulling and tearing at nails. The elongation of the material makes it well suited for roofing purposes. In the first place the material does not elongate excessively and therefore does not tend to stretch out of shape and become permanently deformed. On the other hand, it is not excessively "short", and the roofing has sufficient elongation so that it is capable of adapting itself without rupture to deformations and movements of roof decks to which it is secured.

The foregoing is illustrative of a felt desirable for roofing. For purposes of further illustration a bituminized web especially adapted for flooring will be given.

The web for the flooring may be made up for the purpose of this illustration by using about 80% purified fiber and about 20% kraft fiber. The method of manufacturing the flooring felt is the same as that employed in making the roofing except that the dilution of the mixed fiber furnish, the cylinder vat water level differential and the pressure on the couch and press rolls are adjusted so that the index of compactness will be about 0.95 to about 1.15, values which have been found to be desirable in the manufacture of flooring. In the illustration given the web has an index of compactness of about 1.05, (i. e., a caliper of .060" and a felt number of 57).

The saturant used in the flooring has a softening point of about 160° F. and a penetration of about 25 at 77° F. and is applied at a temperature of about 400° F. The felt preferably is caused to take up on the saturating machine about 125% to 150% of saturant and in the illustration here given there is 137% of absorbed saturant. Excess of saturant may cause "bleeding" and staining of the surface of the flooring. The saturated felt is given a coating of priming paint, followed by a coating of print paint and a surface finish of lacquer and is then baked for four or five days at 140°–150° F., the finished flooring having a caliper of about .071" and weighing about 40 lbs. per 100 sq. ft.

The resulting product when tested is found to

have the following characteristics, in comparison with a high quality rag felt flooring made in the same way.

| | Rag felt flooring | Purified fiber—kraft felt flooring |
|---|---|---|
| Tear-resistance per .001" thickness of base felt, grams | 24.2 | 48.1 |
| Bursting strength (Mullen) lbs. | 166 | 295 |
| Tensile strength, lbs. per inch width, average | 102 | 133 |
| Elongation, per cent | 4.0 | 5.8 |
| Tensile load at 2% elongation, lbs., average | 85 | 108 |
| Indentation, mm | 1.8 | 1.6 |

From the above data it is seen that the flooring is very resistant to tearing and that the Mullen strength and tensile strength are high. Moreover, the material is flexible and capable of being rolled and unrolled without cracking, but is not subject to excessive stretching and deformation. The flooring is resistant to indentation. This latter is a very desirable attribute with respect to flooring. According to this invention a flooring can be produced which is not only flexible and resistant to tearing but is also very resistant to indentation.

The employment of kraft fiber with purified fiber is regarded as preferable in the practice of this invention. In the manufacture of webs impregnated with bitumen, it has been found that high tear-resistance is afforded in combination with high bursting strength when about 10% to about 40% of kraft fiber is used with about 50% to about 80% of purified fiber, with minor proportions of unpurified fiber other than kraft. In these proportions a high degree of tear-resistance is retained. In unimpregnated webs a higher proportion of kraft fiber can be employed without sacrifice of tear-resistance.

While the employment of kraft fiber is regarded as preferable according to this invention other fiber may be used such as cotton and wool No. 1 and No. 2 felt-makers' (roofing) rags, cotton linters, cotton mill waste, wool, wool waste, wool carpet clippings, jute and the like. The character of these fibers is such that they make up harder, denser and less absorbent felt or other web than does the purified fiber. Fiber such as illustrated will be referred to as "hard type" fiber herein. So called "mixed paper" fiber is also to be regarded as hard type fiber.

Mixtures of purified fiber and hard type fiber, with other fibers may also be employed in the practice of this invention. In the practice of this invention it is preferable to employ with the purified fiber a mixture of hard type fiber and other fiber which produces a harder, denser and stronger felt or other web than does the purified fiber by itself.

Thus, for example, felts may be manufactured containing about 60% purified fiber, about 30% kraft and about 10% cotton waste, or containing about 50% purified fiber, about 40% kraft and about 10% wool carpet clippings. Such felts when impregnated with roofing or flooring saturants produce superior roofings and floorings.

In the practice of this invention it is preferable to employ a major proportion of purified fiber and about 10% to 50% of hard type fiber. Particularly satisfactory results are obtained when about 20% to 40% of hard type fiber is employed and the balance of the fiber used is substantially composed of purified fiber. When larger amounts of unpurified fiber are used, e. g., up to as much as 50% in proportion to the purified fiber, it is preferable to employ lesser degrees of pressure in making the web as by increasing the proportions of unpurified fiber the susceptibility of the web to impregnation is decreased.

The following is a table giving the approximate softening points (by the ball and ring method) and penetration of bituminous saturants, desirable for roofing and flooring.

| Bitumen | Softening point °F. | Penetration at 77° F. |
|---|---|---|
| Shingle saturant | 105–200 | 5–70 |
| Roll roofing saturant | 75–160 | 45–200 |
| Flooring felt saturant | 115–200 | 15–80 |
| Shingle coating | 190–275 | 5–50 |
| Roll roofing coating | 190–275 | 10–50 |

The above table is designed merely to define generally the types of asphalts which are useful in the manufacture of bituminized webs for the purposes stated and is intended as a guide in the selection by one skilled in the art of bituminous saturants and coating materials which may be used in the practice of this invention. The type of saturant used will depend upon the properties desired in the finished product and upon the compactness and strength qualities of the felt web which is to be impregnated.

While this invention has been described in connection with certain specific examples it is to be understood that this has been done merely for the purpose of illustration and that the scope of this invention is not to be limited thereby.

Moreover, while certain data has been given as to the results of tests upon certain examples of materials embodying this invention, it is to be understood that this has been done merely for the purpose of affording a better understanding of this invention without imposing a limitation thereon and that in different embodiments of this invention the specific properties thereof will vary depending upon the types and proportions of material used, the process of manufacture and the like within the scope of this invention as determined by the language of the following claims.

We claim:

1. As an article of manufacture a felted web having an index of compactness of approximately 0.95 to approximately 1.4 and containing a major proportion of substantially unreduced purified cellulose wood fiber interfelted with over about 10% of a harder type wood fiber than said purified fiber, said web being impregnated with a bituminous saturant.

2. As an article of manufacture a felted web containing purified cellulose wood fiber as a major portion interfelted with a minor portion of harder type substantially unpurified fiber including kraft fiber and a fiber selected from a group consisting of cotton linters, jute, wool, cotton mill waste, No. 1 and No. 2 felt maker's rags, wool carpet clippings, and mixed paper, said web being impregnated with a bituminous saturant.

3. As an article of manufacture a felted web having an index of compactness of from about 1.20 to about 1.40 and containing purified cellulose fiber as a major portion of fiber and a harder type wood fiber than said purified fiber as a minor portion of fiber, said web being impregnated with a bituminous saturant to the extent of about 225% to about 275% on the weight of dry web and said saturant having a softening point of from

about 75° to about 200° F. and a penetration of about 200 to about 5 at 77° F.

4. As an article of manufacture a felted web having an index of compactness of from about 0.95 to about 1.15 and containing puried cellulose fiber as a major portion of fiber and as a minor portion of wood fiber a harder type fiber than said purified fiber, said web being impregnated with bituminous saturant to the extent of about 125% to about 150% on the weight of dry web and said saturant having a softening point of about 115° F. to about 200° F. and a penetration of about 80 to about 15.

5. As roofing material, a felted web having an index of compactness of from about 1.20 to about 1.40 and containing from about 50% to about 80% of substantially unreduced purified cellulose wood fiber and about 10% to about 40% of substantially unpurified kraft fiber, said web being impregnated to the extent of about 225% to about 275% on the weight of dry web with bituminous saturant having a softening point of from about 75° F. to about 200° F., said bituminized web having a surface coating of bituminous material.

6. As a flooring material a felted web having an index of compactness of from about 0.95 to about 1.15 and containing about 50% to about 80% of substantially unreduced purified cellulose wood fiber and about 10% to 40% of substantially unpurified kraft fiber, said web being impregnated to the extent of about 125% to about 150% on the weight of dry web with a bituminous saturant having a softening point of about 115° to about 200° F., said bituminized web having a wear-resistant surface coating.

7. A process of making a water-laid felted web containing a mixture of purified cellulose fiber and unpurified fiber which comprises beating the purified fiber separately only until lumps are substantially removed and without substantially reducing said fiber in size, beating the unpurified fiber separately until lumps are substantially removed and until the unpurified fiber has been reduced to a paper making furnish, mixing the separately beaten fibers and blending same and thereafter forming the same into a felted web in a paper making operation including felting the aqueous fiber furnish on a screen and pressing the resulting felt, the dilution of the furnish and the pressure of the pressing resulting in a web having an index of compactness of approximately 0.95 to approximately 1.4.

8. As an article of manufacture, a felted web having an index of compactness of between approximately 0.95 and approximately 1.4 and comprising over about 50% of substantially unreduced purified cellulose fiber interfelted with about 10% to 50% of substantially unpurified kraft fiber.

9. As an article of manufacture, a felted web having an index of compactness of between approximately 0.95 and approximately 1.4 and comprising a major proportion of substantially unreduced wood fiber containing over about 93% of $\alpha$ cellulose, about .1% to about 1.1% of lignin and about 1% to 4% of pentosans, interfelted with a minor proportion of alkali-liberated wood fiber adapted for paper making and containing over about 6% of pentosans and over about 4% of lignin.

10. As an article of manufacture a felted web having an index of compactness of between approximately 0.95 and approximately 1.4 and comprising a major proportion of substantially unreduced purified wood fiber and over about 10% of substantially reduced wood fiber of harder type than said purified fiber.

11. As an article of manufacture a felted web having an index of compactness of between approximately 0.95 and approximately 1.4 and comprising a substantial proportion of substantially unreduced purified cellulose fiber interfelted with a substantial proportion of wood fiber which is harder and less absorbent than said purified fiber.

12. As an article of manufacture a felted web containing over about 50% of substantially unreduced purified cellulose fiber and over about 10% of substantially reduced unpurified wood fiber.

13. As an article of manufacture, a felted web having an index of compactness of from about 0.95 to about 1.4 and containing purified cellulose fiber as a major portion of fiber and as a minor portion of fiber over 10% of a substantially harder type wood fiber than said purified fiber, said web being impregnated throughout with bituminous saturant.

14. As an article of manufacture, a felted web having an index of compactness of from about 0.95 to about 1.4 and containing purified cellulose fiber as a major portion of fiber and as a minor portion of fiber over 10% of a substantially harder type wood fiber than said purified fiber, said web being impregnated throughout with bituminous saturant, to the extent of about 125% to about 275% on the weight of dry web and said saturant having a softening point of from about 75° F. to about 200° F.

15. As an article of manufacture, a felted web having an index of compactness of from about 0.95 to about 1.4 and containing purified cellulose fiber as a major portion of fiber and over about 10% of kraft fiber, said web being impregnated throughout with bituminous saturant, to the extent of about 125% to about 275% on the weight of dry web and said saturant having a softening point of from about 75° F. to about 200° F.

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CERTIFICATE OF CORRECTION.

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It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows: Page 3, first column, line 71, for "235%" read 235°; and second column, line 14, in the table, column 1, for "xtrength" read strength; page 5, first column, line 5, claim 4, for "puried" read purified; and that the said Letters Patent should be read with these corrections therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 16th day of March, A. D. 1937.

Henry Van Arsdale
Acting Commissioner of Patents.

(Seal)