METHOD OF MAKING MICROPOROUS SHEET MATERIAL

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Fig. 1.

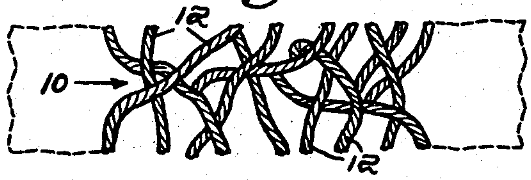

Fig. 2.

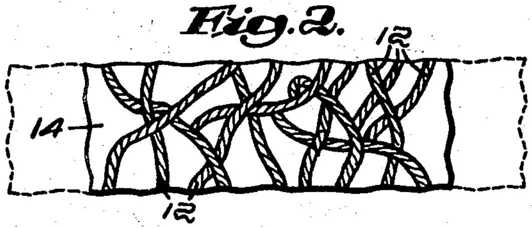

Fig. 3.

Fig. 4.

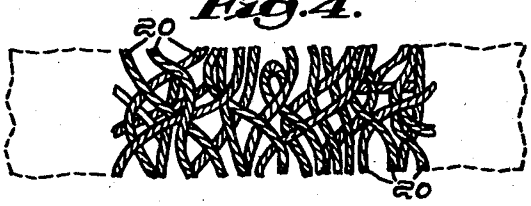

Fig. 5.

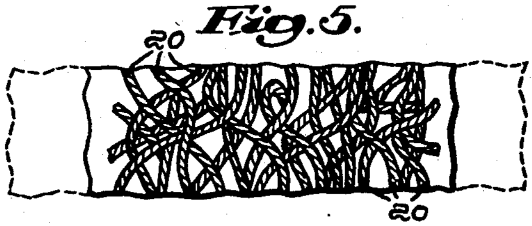

Fig. 6.

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Fig. 7.

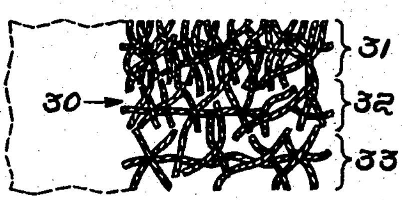


Fig. 8.

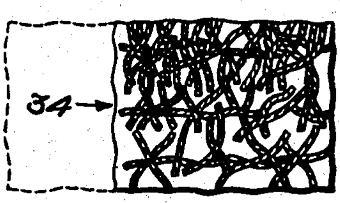


Fig. 9.

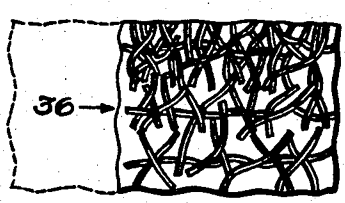


Fig. 10.

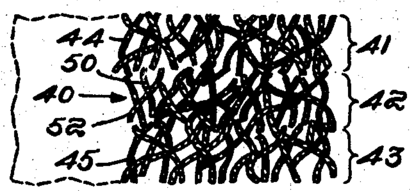


Fig. 11.

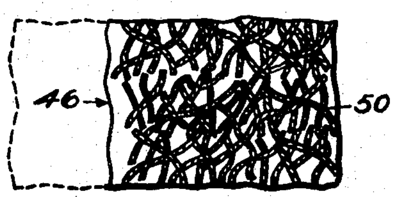
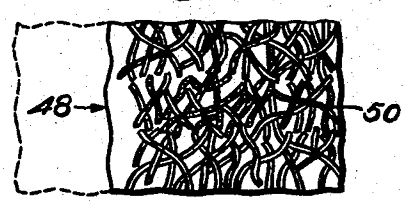


Fig. 12.



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UNITED STATES PATENT OFFICE

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METHOD OF MAKING MICROPOROUS SHEET MATERIAL

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3 Claims. (Cl. 154—33)

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This invention relates to synthetic microporous products and to a novel method of producing the same. The invention specifically concerns microporous sheet materials which may be flexible and resilient like chamois and used for filtering, hospital sheeting, surgical bands and supports, wicks, etc. or which may be of a less flexible nature and serve such uses as battery separators, self lubricating oil seals, etc. In any event the product is not only of a microporous nature but also has permeability and capillary absorption characteristics adapting it particularly to these and other like uses.

The invention especially concerns the manufacture of these novel materials through the employment of natural latex or synthetic latices, resins, plastics or other like compositions capable of self support as a matrix in a two-phase system, in association with a felted structure embodying interlocked wool fibres, the felted structure and its fibres predetermining the character of the final product. Felted structures are fabrics built up by the interlocking of fibres by a suitable combination of mechanical work, chemical action, heat and moisture, and may comprise one or more classes of wool fibres, with or without the admixture of animal, vegetable and synthetic fibres.

In accordance with the invention, a felt of a composition adapted to produce a product having the required characteristics is first manufactured, the character and diameter of the fibres, the fibre pattern or structure, the intersticed fibre relationship and the density of the felt being given careful consideration. The felt is then filled or impregnated to fill the voids between the fibres and form a solid composite sheet, the impregnation being carried out with either a liquid or solid impregnant and preferably by padding, immersion, dipping, extruding, calendering or otherwise, with or without heat and/or pressure. This composite sheet is then treated chemically or thermally to remove such of the wool fibres as is required to produce the desired porosity, permeability and capillary absorption characteristics, the removal of such fibres leaving microscopic tubular pores extending into the sheet and with certain of these pores at the two faces of the sheet interconnected or extended through the sheet to provide the required characteristics.

The porosity and other characteristics are controlled by varying the density of the felt and the diameter, staple length and character of the fibres employed, as hereinafter more specifically described, and our invention furthermore contemplates a controlled differential porosity throughout the thickness of the product sheet.

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Such control may be effected by combining two or more unhardened or semi-hardened batts of different character before final hardening and fulling in which the felting value of each batt, as well as the fibre character, size, distribution and pattern are quite different. Furthermore we can by combining batts of selected fibre composition produce numerous characteristic variations such, for example, as a product porous at its two faces but impervious at its center or intermediate portion.

A further feature of the invention concerns the manufacture of finished articles by our process by molding or prefabricating the fibres or felt before the subsequent treatment to develop characteristics of porosity, permeability and capillary absorption, thereby producing our improved product in the form of finished articles.

These and other objects and features of the invention wil be more readily understood and appreciated from the following description of preferred embodiments thereof selected for purposes of illustration and shown in the accompanying drawings in which—

Fig. 1 is a magnified view of a low density wool felt employed in our invention,

Fig. 2 illustrates the same impregnated or filled with a composition capable of self support as a matrix in a two-phase system,

Fig. 3 illustrates the final product resulting from the destruction and removal of the wool fibres,

Figs. 4, 5 and 6 respectively correspond to Figs. 1, 2 and 3 but show the employment of a high density felt in the production of a sheet material having relatively higher porosity, permeability and capillary absorption,

Figs. 7, 8 and 9 are like views illustrating the manufacture of a product having differential porosity characteristics through the employment of a felt combining two or more felting batts having different characteristics, and

Figs. 10, 11 and 12 are like views illustrating the manufacture of a product which is porous adjacent to its two faces but substantially impervious at its center or intermediate portion.

Our invention contemplates the manufacture of microporous sheet material having predetermined characteristics of porosity, permeability and capillary absorption and these characteristics are predetermined in the final product by careful and purposeful selection of the fibre making up the felt which we employ in the process and by the felting steps and operations employed in the manufacture of the felt. We have discovered that the structural and functional characteristics of the finished product can be quite definitely predetermined and controlled by thus constructing the felt and that numerous and wide variations can be produced in the product. In the accompanying drawings we have illustrated certain differently modified constructions which we will now describe.

In Figs. 1–3 of the drawing we have illustrated the manufacture of a microporous sheet material of low porosity, permeability and capillary absorption. The felt 10 shown in Fig. 1 includes a minor number of wool fibres 12 per unit volume and of a predetermined grade, such as 70's, felted into interlocking relation by known felting processes, including mechanical working, chemical action, heat and moisture and producing a low density felt. The predetermined forming of the felt is the first step in our process. It is well known that animal fibres have the characteristic of interlocking into a felted mass and we preferably employ at least 10% of such fibres in the production of our novel product. The fibre diameter size employed depends upon the character of product desired, such diameter sizes varying from fine fibres, known commercially as 80's, to very coarse fibres, known as 30's.

The second step of the process comprises the filling or impregnating of the felt with a composition 14 capable of self support as a matrix in a two-phase system. This composition can be any elastomer, resin or plastic-like material that will serve the purpose for which the final product is to be used. The impregnant can be either a liquid or solid and the impregnating can be accomplished by any treatment that fully fills the voids between the fibres and forms a solid sheet with the fibres embedded therein. The felt can be liquid filled under pressure or the impregnant can be introduced as a solid under pressure with or without the assistance of heat. For example, we can take silicone putty or uncured elastomers, such as rubber or neoprene, and extrude them into the felt under pressure by means of a combining machine, calender rolls or press plate. Silicone putty is a polymeric derivative of SiO_2 in which the O_2 radical is replaced or combined with selected organics to form a heat resistant, resilient plastic material with silica as its skeleton.

It may be desirable in some cases to impregnate the felt with a two-bath treatment wherein the first bath saturates the felt with the impregnant and the second bath polymerizes or precipitates the product of the first bath. Thiokol impregnations into very dense materials are made in this manner by impregnating with the partially linked Thiokol solution and then treating by means of a second bath to polymerize and complete the packing of the Thiokol molecules. In the case of a Thiokol or organic polysulfide impregnation, the first bath would be a liquid polymer of soluble inorganic polysulfides which would thoroughly penetrate the felt and be reacted in the second bath with an organic dihalide such as ethylene dichloride to form a solid impregnation in the presence of ammonia. Similarly, a liquid polymer of Thiokol applied to the felt in the first bath could be reacted by ammonia in either liquid or gaseous form in the second bath. Thiokol is an organic polysulfide.

Following the steps above described and illustrated in Figs. 1 and 2, the filled sheet is dried and/or cured, in accordance with the requirements of the fill composition used, and is then treated to destroy and remove the wool fibres 12. We have performed this treatment by dissolving and leaching out the wool fibres of the felt in a cold or hot bath of caustic, such as a hot 5% solution or a cold 10% solution of sodium hydroxide, thereafter neutralizing the sheet in water or acid and finally drying the resulting porous sheet. We have also found it possible to attain the same results by thermal-carbonization or ashing of the fibres and, when employing elastomers and resins subject to or requiring curing, we accomplish our thermal disintegration or carbonization of the fibres simultaneously with the curing or processing in either a press or oven. In any event the final product 16 comprises a solid sheet of the composition 14 having microscopic tubular pores 18 extending thereinto from its two opposite faces. Certain of these tubes interconnect within the sheet and others may extend entirely through the sheet.

It will be apparent that the composition 14 employed will depend upon what is wanted in the final product. In the case of flexible, chamois-like products the fill will be of rubber-like materials such as natural latex, Buna S, neoprene, Hycar, vinyl butyrol, synthetic latices, etc. and in the case of more or relatively rigid products such, for example, as may be used for battery separators, the fill will be suitable plastics or resins, as hard natural or synthetic rubber, Bakelite, urea formaldehyde and other suitable materials or compounds. The end product is the felt in reverse, i. e. the fibres become the tubular voids in the filled and otherwise impervious sheet which occupies the interstices that were between the fibres.

Buna S is a co-polymer of butadiene and styrene. Neoprene is polymerized chloroprene. Hycar is a well known butadiene rubber developed by the Goodyear and Goodrich rubber companies. Vinyl-butyrol is the vinyl derivative of butadiene. Bakelite is a well known synthetic resin.

In Figs. 4, 5 and 6 of the drawing we have illustrated the manufacture of a microporous sheet material of high porosity, permeability and capillary absorption. In this case we start our process with a high density felt having a large number of wool fibres 20 per unit volume and of predetermined grade such as 56's. The fibres are felted together into interlocking relationship in the manner above described to produce the felt shown in Fig. 4; this felted sheet then being filled to produce the sheet shown in Fig. 5, that is to say, the interstices or voids between the fibres are completely filled up to substantially the same level; and this felted sheet being then treated to remove the wool fibres and produce the product shown in Fig. 6; all as above described in connection with Figs. 1, 2 and 3.

It will be readily apparent that sheet 22 (Fig. 6) is more highly porous and permeable and has greater capillary absorption than the sheet 16 and that our process makes possible the production of materials having these characteristics in predetermined degree. The porous character and capillary absorption of the sheet is increased with increase of density in the felt employed and is decreased with a decrease in the density of the felt. We desire also to point out that this characteristic can be modified and controlled by varying the fibre composition of the felt, the felting value of the wool fibres employed, the web construction of the carded batt, the degree of initial and final felting imparted respectively by hardening and fulling, and finally, the type of surface finishing employed. It is by these features of the process that we control the fibre diameter, the fibre pattern or structure, the intersticed fibre relationship, and the density of the felt. In the reverse and final phase of the process producing our microporous sheet materials, these factors control respectively the pore size and distribution and the porosity, permeability, capillary absorption and density of the product.

In Figs. 7, 8 and 9 of the drawing we have illustrated a modification of our process to produce a microporous sheet material having controlled differential porosity throughout its thickness. In this case we start the process with a felt **30** made by combining two or more hardened or unhardened batts before final hardening and fulling in which the felting value of each batt, as well as its fibre character, size, distribution and pattern, are quite different from these characteristics in the other batts. We have illustrated the combining of three batts **31**, **32** and **33**, the batt **31** being of relatively high density and fine grade fibre and the batt **33** being of relatively low density and coarse 50's grade fibre. The methods by which the desired characteristics are given to the felt are well known in felting making. In the example, illustrated, we obtain a finished material having a fine fibered and dense face portion **31**, a medium center portion **32**, and a coarse, low density back portion **33**. Numerous other modifications are possible and the effect of these modifications on the character of our microporous sheet material will be apparent.

The interstices or voids of the felt **30** are completely filled to produce the solid sheet **34** (Fig. 8) and this filled sheet is treated to remove the wool fibre and produce the final product **36** (Fig. 9), all as above described in connection with Figs. 1–3. It will be readily apparent that sheet **36** is of different and varied porosity at its two faces and throughout its thickness and that our process makes possible the production of materials having these characteristics in predetermined degree.

In Figs. 10, 11 and 12 of the drawing we have illustrated a further modification of our process to produce a microporous sheet material porous at its two faces but of limited porosity at its center or intermediate portion. In this case we start the process with a felt **40** made by combining two or more batts in the manner above described in connection with Fig. 7. We have illustrated this felt as constructed from three batts **41**, **42** and **43**. The two outer batts **41** and **43** contain predetermined wool fibres **44** and **45** and the inner batt **42** contains cotton and wool, thereby forming a felt with an all-wool face and back and a cotton-wool center. It will be apparent that numerous other fibre and construction modifications can be employed. Furthermore in addition to producing microporous sheet material with differential porosity, we can in this manner and by the selection of a suitable fibre destruction agent, produce a microporous sheet material with cotton, wool, silk, rayon or other fibre remaining in the center or distributed as desired through the mass.

The felt **40** is completely filled to produce the solid sheet **46** (Fig. 11) and this filled sheet is then treated to remove the wool fibres and produce the final product **48** (Fig. 12), all as above described in connection with Figs. 1–3. The caustic dissolves and removes the wool fibres but has no effect on the cotton fibres **50** which are left intact and bonded to the sheet **48** intermediately of its two face portions. As illustrated, the wool fibres **52** at the center portion are dissolved out to produce a limited number of pores, thereby rendering the sheet highly porous at its two face portions but of limited porosity at its center portion. It will be apparent that the sheet can be made impervious at its center portion by employing all vegetable fibres in the center batt **42**.

While, as hereinbefore described, our invention generally comprises microporous sheet material and its manufacture, we desire to point out that our product can also be produced in the form of finished articles by molding or prefabricating the felt into predetermined shape before its subsequent treatment to develop the characteristics of porosity, etc. Preferably the fibres will first be felted into sheet form, shaped and impregnated, and the resulting impregnated shape will be treated to remove selected fibres and produce the required characteristics. Modifications of these specific steps, such as the simultaneous felting and shaping of the fibres and subsequently impregnating the felt, will be apparent. It will also be apparent that this preshaping feature of our invention is of particular value when using a non-elastic resin type filler, such as phenol formaldehyde which is a hard and somewhat brittle form-setting material.

It is particularly emphasized that while the invention incorporates wool fibres in the felt to secure the interlocking felted fibre relation and while we ordinarily remove the wool fibre in whole or in part, as each case requires, we desire it to be understood that some applications of the invention may also require the incorporation of synthetic, vegetable and mineral fibres and also in such cases we may by suitable and known treatment remove certain of these fibres with or without removal of the wool fibres. We furthermore desire to emphasize that our process embodies the full impregnation of the felt and the complete filling of the voids and interstices therein with emulsions, dispersions, solutions, solid compounds, and the like to produce a solid sheet as distinguished from mere coating of the fibres to produce a relatively open product with unfilled interstices, the sheet produced by our invention being substantially impervious except for the fibre-formed pores and being a solid body with opposed finished surfaces and incorporating porosity, permeability and capillary absorption in predetermined degree. The impregnant employed can be any composition capable of self-support as a matrix in a two-phase system of the nature described and where we herein refer to the same as a "plastic" we mean any composition that fulfills this requirement.

Impregnation can be effected from an aqueous dispersion or solvent solution of the impregnant, from molten impregnants, or from solid impregnants introduced under pressure as by calendering. The character and porosity of the finished microporous sheet material is dependent first, on the type of felt used, and second, on the nature of the impregnant. Highly microporous sheet materials are made from felts of high density, and those of low porosity from felts of low density. Differential transverse porosities and other controlled modifications in the finished microporous sheet material are functions of the felt employed and the structural modifications attainable in felt well known to the art.

Having now disclosed our invention what we claim as new and desire to secure by Letters Patent of the United States is:

1. A method of making microporous sheet ma-

terial, which includes the steps of felting selected loose wool fibres into interlocking relation in a felt sheet, filling the felt sheet under pressure with an uncured elastomer and thereby producing a solid self-sustaining matrix with the wool fibres solidly embedded therein, curing the elastomer filling of the sheet, and then dissolving the wool fibre, thereby producing a flexible sheet of chamois-like texture having pores left by the fibres removed from the otherwise solid matrix.

2. The method defined in claim 1 in which the felt sheet is made up of a plurality of wool batts of different density.

3. The method defined in claim 1 in which the wool fibres are combined with non-soluble fibres which remain in the solid matrix of the finished flexible sheet.

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