

UNITED STATES PATENT OFFICE.

THOMAS CLIFFORD WOODMAN, OF TEDDINGTON, AND WILLIAM ALEXANDER DICKIE,
OF SPONDON, NEAR DERBY, ENGLAND, ASSIGNORS TO CELANESE CORPORATION
OF AMERICA, A CORPORATION OF DELAWARE.

FABRIC OR MATERIAL AND THE MANUFACTURE THEREOF.

No. Drawing. Application filed December 8, 1925, Serial No. 74,183, and in Great Britain January 10, 1925.

This invention relates to the manufacture of new fabrics or materials possessing waterproof to gas-proof properties or capable of other useful applications.

5 According to the invention, woven, knitted, or other fabrics made of yarns or threads composed of filaments or fibres consisting or having a basis of cellulose acetate, ethyl-, methyl- or benzyl-cellulose, nitro cellulose or other esters or ethers of cellulose (or mixtures of such cellulose derivatives) alone or associated with silk, cotton, wool, artificial filaments or fibres of the cellulose type or other fibres or filaments, or fabrics 10 made of such yarns or threads woven, knitted or associated with yarns or threads of such other filaments or fibres, are subjected to heat and pressure (without or with employment or application of plasticizing or softening agents or solvents of the cellulose derivative or derivatives), thereby producing a greater or less melting effect on the filaments or fibres of the cellulose derivative or derivatives by the action of the heat 25 and pressure, so that the pores or interstices of the fabric are reduced to extremely minute dimensions, or closed completely.

The extent of the melting effect and the degree of closing of the pores or interstices of the fabric, that is to say the degree of permeability of the fabrics produced, can vary with the degrees of heat and pressure employed and with whether plasticizers or softeners or solvents are employed, or other 35 circumstances.

Thus for example the heat and pressure (with or without employment or assistance of plasticizing or softening agents or solvents) may be such as to close or reduce to minute dimensions the pores or interstices of the fabric and produce a glazed water resisting or even a gas resisting face or faces thereon without causing the filaments or fibres of cellulose derivative or derivatives to coalesce or disappear. Or the heat 45 and pressure (with or without employment or assistance of plasticizing or softening agents or solvents) may be such as to cause the filaments or fibres of cellulose derivative or derivatives to melt and coalesce partly or entirely on the face or faces of the fabric or throughout its substance.

It is to be understood that the conditions of heat, pressure and duration of pressure

are inter-dependent and that any or all of these conditions may be varied according to circumstances or requirements. For example, the less the heat greater or the longer the pressure required to produce a given effect and vice versa, or again, the same conditions of heat and pressure may be applied for more or less time to produce the effect in a more or less pronounced degree. 55

The degree of melting effect may be increased or accentuated by the employment of plasticizing or softening agents or solvents as referred to, and it is to be understood that such agents or solvents may be applied to the fabric before the application of the heat and pressure or may be incorporated in the filaments or fibres of the cellulose derivatives themselves in the production thereof, for example by employing such agents in the spinning solutions. 60

Any known plasticizing or softening agents or high boiling or relatively high boiling solvents of the cellulose derivative or derivatives may be employed. As some instances there may be mentioned for example triacetin, paratoluene sulphonamide or its derivatives, diethylphthalate, and paratoluene sulphonanilide, high boiling alkylated xylene-sulphonamide derivatives or preparations (for instance monomethyl xylene sulphonamide), and so forth. 65

As the melting effect is increased or accentuated by the plasticizing or softening agents or solvents, one can employ less heat or/and pressure or a shorter application of heat and pressure for the production of a given effect when such agents or solvents are employed. 70

The fabrics employed may consist wholly of yarns or threads of the cellulose derivative or derivatives (cellulose esters or ethers) or may consist partly of such yarns or threads and partly of yarns or threads of other textile filaments or fibres, for instance silk, cotton, wool or artificial silks of the cellulose type (e. g. viscose silk, cuprammonium cellulose silk or denitrated cellulose silk). 75

Thus for example we may employ woven fabrics composed of yarns of cellulose acetate or other cellulose ester or ether in the warp and with a back or weft of cotton, wool, silk, cellulose, or any other well known texture; or yarns of cellulose acetate or other 80

cellulose ester or ether may be mixed in both warp and weft with yarns of such other fibre or fibres; or any other suitable arrangement or combination of yarns of cellulose acetate or other cellulose ester or ether with yarns of such other fibre or fibres may be employed in the fabrics.

Or again yarns or threads may be employed consisting of filaments or fibres of the cellulose derivative or derivatives doubled or associated with such other filaments or fibres, and the fabrics may consist wholly of such compound yarns or threads or partly of same and partly of yarns or threads of the other textile fibres or filaments.

The invention is particularly applicable to the treatment of fabrics made of or containing cellulose acetate, and will hereinafter be described in this connection, it being understood however that other cellulose esters or ethers may be employed as before indicated.

The heat and pressure may be applied in any appropriate manner to the fabrics, for example by passage between heated pressure rollers, or between a heated roller and a heated or cold plate or surface, or by pressure between heated plates or between a heated plate and a cold plate or surface, or by passing the fabric under tension over a single heated roller, e. g. a calender roller, or in any other appropriate way, the conditions of temperature, pressure and duration of pressure being variable with the extent of the melting effect to be produced on the filaments or fibres of cellulose acetate or other cellulose derivatives, and with whether plasticizers, softeners or solvents are employed, or other circumstances. In cases where the fabric is passed under tension over a single heated roller, the roller may with advantage be rotated in an opposite direction in the travel of the fabric.

The manner in which the invention may be carried into effect can be illustrated by the following more detailed description, it being understood that the forms or examples described are given only by way of illustration and can be varied widely without departing from the spirit of the invention.

Fabrics entirely of cellulose acetate.

A fabric made entirely from cellulose acetate yarn, such for instance as a jap or "tabby" weave fabric, or a warp knitted fabric, may be subjected to heat and pressure alone, to give a fabric possessing desired degrees of resistance to penetration by water or gases, according to the temperature and pressure and duration of the pressure, the conditions of heat, pressure and time being interdependent. The less heat the greater or the longer pressure required to produce a given effect, and vice versa, or again, the same conditions of heat and pres-

sure may be applied for more or less time to produce the effect in a more or less pronounced degree.

Thus for example a cellulose acetate fabric such as referred to may be pressed between smooth plates at a temperature of about 100° C. under a pressure of about 500 lb. per square inch for about 5 minutes, to give a fabric substantially waterproof, very supple and rubber-like in its texture and capable of being sewn and manipulated similarly to an ordinary fabric; such a fabric retains the structure of the woven fabric, but the surfaces become highly glazed, the translucency much increased, and the interstices in the fabric become so greatly reduced in dimensions that surface tension effects produce waterproof qualities. By increasing or reducing any or all of the factors; heat, pressure and duration of pressure, the degree of melting effect on the cellulose acetate yarns can be increased or reduced according to requirement to give fabrics of any desired degree of resistance to penetration by water or gases, and if required, they may be adapted to cause partial or complete melting together or coalescing of the yarns constituting the fabric.

Similar results may be obtained by passing the cellulose acetate fabric between heated pressure rollers, for instance calender rollers. This method has the advantage of being continuous. The conditions of heat, pressure and time are interdependent as before mentioned. By way of example the fabric may be passed slowly through heated calender rollers at temperatures between about 100° and 180° C. under pressures of from about 300 to 600 lb. or more per square inch, according to the degree of melting effect and impermeability desired in the fabric. The fabric may be passed repeatedly between the heated rollers if desired, according to the degree of effect required.

Or the fabric may be passed under tension over a single heated calender roller, which may with advantage be rotated in an opposite direction to the travel of the fabric.

Or again, the fabric may be passed once or repeatedly between a heated roller and a cold roller or platen or pressed once or repeatedly between a heated plate and a cold plate so that in either case the effect is produced chiefly on one face of the fabric.

As before mentioned the fabrics employed for the purpose of the invention may be treated with plasticizing or softening agents or solvents of the cellulose derivative or derivatives before being subjected to the heat and pressure. This is especially of advantage where a high degree of impermeability to water is desired or for obtaining gas proof properties. By way of example a cellulose acetate fabric may be first treated with small quantities of water-insoluble,

non-volatile plasticizers, softeners or solvents of cellulose acetate before being subjected to the heat and pressure. These quantities may vary for instance from about 5 1% to about 30% of the total quantity of cellulose acetate in the fabric, but more or less may be employed. The non-volatile plasticizers, softeners or solvents may be applied in any appropriate way, for example 10 dissolved in a volatile solvent thereof which is a non-solvent of the cellulose acetate. A suitable plasticizer or softener for cellulose acetate is, for instance, monomethylxylene sulphonamide and a suitable volatile vehicle 15 is for instance coal tar benzole. For the treatment of a cellulose acetate fabric suitable proportions could be for example for each 100 grams of cellulose acetate fabric about 20 grams of monomethylxylene sulphonamide dissolved in 100 grams of benzol, 20 the proportions of the non-volatile plasticizer and the volatile solvent thereof being such that together they make a non-solvent mixture for the cellulose acetate. The mixture 25 is distributed over the fabric by dipping, spraying or in other well known ways. When the volatile solvent of the plasticizer or softener evaporates, the plasticizer remains distributed uniformly on the fabric, 30 with partial absorption by the cellulose acetate. Heat and pressure are then applied to cause more or less solution or melting of the cellulose acetate of the fabric, thus closing the interstices and producing a fabric resistant to penetration by water or it may be to 35 gases, according to the degree of dissolving or melting effect etc. produced on the cellulose acetate by the conditions of heat and pressure employed.

40 The heat and pressure may be applied by any suitable means such as before referred to, and as the melting effect on the cellulose acetate fibres or filaments is increased or accentuated by the plasticizers, softeners or 45 solvents, less heat or/and pressure, or a shorter application of pressure, will suffice for the production of a given effect than in the case of fabrics not treated with or containing the same.

50 *Composite fabrics.*

As before mentioned we do not confine ourselves to fabrics composed entirely of 55 yarns or threads of cellulose acetate or other cellulose esters or ethers. We have found considerable advantage to be gained by employing fabrics, which, after treatment of the cellulose acetate (or other cellulose ester or ether) component, will have the backing 60 or support of another material. Thus for instance a fabric can be employed composed of cellulose acetate yarn in the warp and with a back or weft of silk, wool, cotton, cellulose or any other well known texture 65 not substantially affected by the conditions

of heat or pressure or the plasticizers, softeners, solvents, etc., used. Or better still, cellulose acetate can be mixed in both warp and weft with such other fibre or fibres, so that after the treatment a backing or support 70 remains which has been unaffected or substantially unaffected by the treatment.

As an example, good effects can be obtained on a warp-faced satin material, in which the warp is composed of cellulose 75 acetate yarn, with the weft or backing of silk or cotton.

Or the warp may contain some silk, wool, cotton, cellulose or other ends or yarns, for example one in every two, four, six, eight, 80 ten, twenty, etc. The weft may be similarly disposed by "checking". Or any other suitable composite fabric may be employed composed of cellulose acetate yarns in any suitable alternation or interposition 85 with yarns of silk, wool, cotton, cellulose or other fibres, whether in warp or weft or both warp and weft or otherwise. It will be understood that many forms or designs of woven or knitted composite fabrics could 90 be employed without departing from the invention.

The composite fabrics may be subjected to the heat and pressure between heated 95 plates or rollers or between a hot roller and a cold roller or platen or by passage under tension over a heated calender roller, or in other suitable way, with or without employment of plasticizing or softening agents or solvents as referred to, and the heat and 100 pressure or heat, pressure and duration of pressure may be adapted to cause any desired degree of melting of the cellulose acetate (or other cellulose ester or ether) component and of closing of the pores or 105 interstices of the fabric, whether while retaining the structure of the cellulose acetate yarns in the fabric, or if required while causing more or less complete melting and disappearance or merging of the cellulose 110 acetate or other cellulose ester or ether in the fabric so as to leave a film of cellulose acetate (or other cellulose ester or ether) united to a support or backing of the non-acetate 115 yarns.

It is also to be understood that mixtures of cellulose esters and/or ethers may be employed in the yarns or threads employed in the fabrics, or that filaments or fibres spun from mixed solutions of the cellulose esters 120 or ethers may be employed for the fabrics.

The invention is very suitable for the production of waterproof fabrics, or fabrics presenting glazed or ciré or moiré or like ornamental or other effects. It may be employed 125 with great advantage for making gas proof fabrics suitable for airships or for other gas containers. For making gas proof fabrics it is preferable to employ or treat the fabrics with plasticizing or softening agents or sol- 130

vents prior to the application of the heat and pressure.

In the case of making gas proof fabrics we may with advantage use composite fabrics composed of yarns of cellulose acetate or other cellulose esters or ether, woven or knitted with yarns or threads of silk or other textile fibers such as referred to, the yarns of said other fibers being preferably employed in close alternation with those of the cellulose acetate or other cellulose esters or ethers; for instance woven fabrics may with advantage be employed in which a yarn of the other fibre succeeds each yarn of cellulose acetate in the warp or weft or in both warp and weft. We do not however limit ourselves in this respect and any suitable composite fabrics may be employed.

While nitro cellulose filaments or fibres may be employed for the yarns or threads used in the fabrics, this is less advantageous on account of the inflammability of nitro cellulose. The term softening agents in the claims is used to include both plasticizers and solvents for the cellulose derivatives employed.

What we claim and desire to secure by Letters Patent is:

1. Process for the manufacture of sheet material which comprises subjecting a fabric containing filaments having a basis of a thermoplastic derivative of cellulose to heat and pressure, thereby softening said derivative and increasing the impermeability of the material.

2. Process for the manufacture of sheet material, which comprises subjecting a fabric containing filaments having a basis of a cellulose ester to heat and pressure, thereby softening said ester and increasing the impermeability of the material.

3. Process for the manufacture of sheet material, which comprises subjecting a fabric containing filaments having a basis of cellulose acetate to heat and pressure, thereby softening said acetate and increasing the impermeability of the material.

4. Process for the manufacture of sheet material having waterproof to gasproof properties, which comprises subjecting a fabric containing filaments having a basis of a thermoplastic derivative of cellulose to pressure between heated rolls, thereby softening said derivative and increasing the impermeability of the material.

5. Process for the manufacture of sheet material having waterproof to gasproof properties which comprises subjecting a fabric containing filaments having a basis of a cellulose ester to pressure between heated rolls, thereby softening the cellulose ester and increasing the impermeability of the material.

6. Process for the manufacture of sheet material having waterproof to gasproof

properties which comprises subjecting a fabric containing filaments having a basis of cellulose acetate to pressure between heated rolls, thereby softening the cellulose acetate and increasing the impermeability of the material.

7. Process of treating fabric containing filaments of a thermoplastic derivative of cellulose, which comprises subjecting the fabric to heat and pressure in the presence of a softening agent for the cellulose derivative, thereby softening the cellulose derivative thereby increasing the impermeability of the material.

8. Process of treating fabric containing filaments of cellulose acetate, which comprises subjecting the fabric to heat and pressure in the presence of a softening agent for the cellulose acetate, thereby softening the cellulose acetate and increasing the impermeability of the material.

9. Process of treating fabric containing filaments having a basis of a thermoplastic derivative of cellulose, which comprises treating the fabric with a softening agent for the cellulose derivative, and subjecting the fabric to the action of heat and pressure, thereby increasing the impermeability of the material.

10. Process of treating fabric containing filaments having a basis of cellulose acetate to produce material of waterproof to gasproof properties, which comprises treating the fabric with a softening agent for the acetate, and subjecting the fabric to the action of heat and pressure.

11. Process of treating fabric containing filaments having a basis of a thermoplastic derivative of cellulose, which comprises treating the fabric with a solution of a softening agent for the cellulose derivative in volatile solvents which are non-solvents of the cellulose derivative, and subjecting the fabric to the action of heat and pressure, thereby increasing the impermeability of the material.

12. Process of treating fabric containing filaments having a basis of cellulose acetate to produce material of waterproof to gasproof properties, which comprises treating the fabric with a solution of a softening agent for the acetate in volatile solvents which are non-solvents of the acetate, and subjecting the fabric to the action of heat and pressure.

13. Process of treating fabric containing filaments having a basis of a thermoplastic derivative of cellulose to produce material of waterproof to gasproof properties, which consists in treating the fabric with a softening agent for the cellulose derivative, and subjecting the fabric to pressure between heated rolls.

14. A sheet material consisting of a fabric containing filaments having a basis of a

thermoplastic derivative of cellulose, said filaments where exposed on at least one of the faces of the fabric being flattened and constituting a glazed surface of increased impermeability.

15. A sheet material consisting of a fabric containing filaments having a basis of a cellulose ester, said filaments where exposed on at least one of the faces of the fabric being flattened and constituting a glazed surface of increased impermeability.

16. A sheet material consisting of a fabric containing filaments having a basis of a cellulose acetate, said filaments where exposed on at least one of the faces of the fabric being flattened and constituting a glazed surface of increased impermeability.

17. A sheet material consisting of a

"mixed" fabric comprising filaments having a basis of a thermoplastic derivative of cellulose and also comprising fibres of non-thermoplastic material, said filaments where exposed on at least one of the faces of the fabric being flattened and constituting a glazed surface of increased impermeability.

18. A sheet material having a weft consisting of a yarn of non-thermoplastic material and a warp consisting of a yarn of cellulose acetate filaments, having increased impermeability, and glazed on at least one of its faces.

In testimony whereof we have hereunto subscribed our names.

THOMAS CLIFFORD WOODMAN.
WILLIAM ALEXANDER DICKIE.