

UNITED STATES PATENT OFFICE

2,421,218

NONINFLAMMABLE WATERPROOFED TEXTILES AND METHOD OF MAKING SAME

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No Drawing. Application February 11, 1943,
Serial No. 475,575

6 Claims. (Cl. 117-76)

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The invention relates to the manufacture of waterproof and water-resistant uninflam-
mable organic fibrous materials, such as textiles, fabrics,
artificial silk, and to impregnating substances
adapted to be used in connection therewith. This
application is a continuation in part of my pend-
ing patent application Ser. No. 460,179, filed Sep-
tember 29, 1942.

The textiles and fabrics treated in accordance
with this invention and garments made thereof
cannot be ignited and do not produce a flame
when subjected to combustion. They are also
waterproof because the waterproofing substances
employed for the impregnation of the textiles
and fabrics in accordance with my invention are
so strongly incorporated into the fibre and adhere
thereto so intensely that they cannot be removed
therefrom in spite of energetic treatment with
water.

Garments made from the materials impreg-
nated according to my invention are, therefore,
particularly suited to be worn by military per-
sonnel, by firemen and people employed in instal-
lations where the danger of fire and explosions
prevails.

Many suggestions have been made to impreg-
nate fabrics with fire preventive and with water-
proofing substances; however, these methods have
not resulted in a successful application in spite
of the fact that numerous substances are known
which being individually applied to the fibres
either prevent its combustion or cannot be re-
moved therefrom by washing. Substances, how-
ever, which exert the combinative effect without
rendering the impregnated textiles and fabrics
hard and brittle are not known as yet.

If the requirement with regard to pliability and
softness is not dominant, as for instance in stage
curtains, an impregnation with silica and certain
silicates will serve the purpose. However, gar-
ments impregnated with these substances become
hard and brittle; they do not accommodate them-
selves to the outline of the body and cannot be
folded or otherwise shaped without breaking.
Therefore, silica impregnated textiles or fabrics
are unusable for the manufacture of garments.

Numerous other inorganic substances, such as
aluminumsulfate, ammoniumbromide, ammoni-
umsulfate, ammoniumphosphate, ammonium bo-
rates, boric acid, have been recommended for the
manufacture of uninflammmable goods. These
substances, however, are watersoluble. Water
insoluble organic salts, on the other hand, such
as magnesium borate and titanac acid which are
recommended for the same purpose may only be

incorporated into the fibres by gluing; they are
unusable for any practical work.

It has also been suggested to impregnate tex-
tiles and fabrics with metastannic acid. The thus
treated materials do not burn with a flame; how-
ever, they have the great disadvantage of ex-
tended or after-glowing. The same phenomenon
results from the application of heavy metal salts
which due to this grave failure are exempted from
practical use; the many inorganic impregnating
substances which have been suggested for the
manufacture of uninflammmable textiles and fab-
rics or garments made therefrom are not affected
by water and have never met with practical
success.

Also certain inorganic components have been
recommended for use in the production of un-
inflammmable articles. For instance, a urea form-
aldehyde condensation product has been sug-
gested to render wood uninflammmable.

However, the problem involved in the produc-
tion of uninflammmable wood and uninflammmable
fabrics is of a widely different nature and there-
fore cannot be solved on similar lines. Fabrics
and textiles have a relatively far greater active
surface than wood. The flame expansion, there-
fore, is much more rapid per surface unit of a
fabric, whereas the impregnation capacity is
small. Therefore, it is possible to protect wood
against inflammation by coating the same with
the above mentioned condensation products. If
the wood is subjected to combustion the artificial
resin has sufficient time to be converted into a
voluminous carbonized coating which protects the
wood. Fibres, however, are carbonized instan-
taneously; no time is available for a slagging
action of the artificial resin; the latter, therefore,
cannot be used to render textiles uninflammmable,
or incombustible. Furthermore, a thorough im-
pregnation with a sufficient quantity of these
condensation products leads to very undesired
hardening of the textiles and fabrics.

Hence, it results that the problem of the manu-
facture of simultaneously water resistant unin-
flammmable textiles and fabrics has not been solved
satisfactorily as yet.

In view of the fact that the prospect of finding
an appropriate inorganic impregnating substance
was very remote, it is the object of the inven-
tion to provide organic impregnating compounds
which render fabrics and textiles uninflammmable
and incombustible and prevent the creation of a
long flame.

It is a further object of the invention to provide
impregnating compounds of the aforesaid type

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which being itself practically water-insoluble render the textiles and fabrics to which they are applied inert to the action of water.

It is also an object of the invention to provide compounds of the aforesaid type which strongly adhere to the fibres and cannot be easily rubbed off.

It is also one of the objects of the invention to provide an impregnating substance which in addition to the above referred to properties prevents the impregnated textiles, and fabrics from becoming hard, brittle, and unelastic.

It is a further object of the invention to provide an impregnating substance for textiles and fabrics which prevents extended glowing.

Furthermore, the compounds which answer the requirements of my invention must be colorfree and must not attack the textiles and fabrics to which they are applied.

The last but not the least object of my invention is that the textiles and fabrics impregnated in accordance with the teachings of my invention in order to be utilized on an expanded commercial scale are resistant to treatments with weak soap and sodium carbonate solutions.

Hence, it results that a successful solution of the instant problem presents unexpected and severe difficulties particularly also in view of the fact that the impregnating substances in order to fully answer the instant requirements must be insoluble in ordinary organic solvents.

In the course of extensive experimentation and research work I have found that the insoluble compounds of the amidoderivates of the cyanuric acid most suitably comply with all of the aforesaid objects.

However, in order to complete the invention the difficulty of firmly securing these compounds on the textiles and fabrics had to be overcome. In this direction a satisfactory solution was established by the application of the amido compounds of the cyanuric acid as salts of phosphoric acids and particularly of the pyrophosphoric acid. This acid has the property of swelling the fibres of the textiles and particularly cotton fabrics to a remarkable degree, whereby a satisfactory loosening of the fibres is effected and their absorptive capacity for the amido compounds is greatly enhanced.

Instead of pyrophosphoric acid metaphosphoric acid, sulphosphoric acid and oxalic acid may be advantageously used. As a most suitable substance as far as insolubility and securing capacity is concerned, a melamine pyrophosphate was established which consists of two mols melamine and one mol pyrophosphoric acid. The textiles and fabrics are preferably first impregnated with the acid and afterwards passed through a melamine bath. An insoluble very fine white precipitate is hereby formed which strongly adheres to the fibres and can only be removed by washing or similar water treatment. The herewith impregnated textiles and fabrics carbonize quickly when exposed to combustion, but they do not catch fire and do not cause after-glow.

However, and in spite of their resistance to water, the pyrophosphates of the cyanuric acid amides and particularly the melamine pyrophosphate show a certain sensitivity to the action of alkalis, salts and certain acids. It forms with salts and alkalis water soluble double salts and with certain acids soluble melamine salts. If the impregnated fabrics are contacted with solutions containing substances of the named type, the precipitate is partly dissolved and the combus-

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tibility of the materials treated in accordance with the invention is accordingly increased.

In order to avoid this inefficiency and to improve the adherence of the precipitate in the treated fibre materials the same are coated with a condensation product of melamine or of a mixture of a melamine and thiourea with formaldehyde.

These condensation products and particularly those which contain thiourea added as a hardening and acidifying agent, are practically free of salts, acids and alkalis; they become insoluble and not inflammable after being hardened by heating. By the addition of thiourea an acid medium is generated at an elevated temperature due to the formation of a small quantity of sulphocyanic acid; instead of thiourea other suitable acids to harden the melamine formaldehyde condensation product may be used.

The use of formaldehyde melamine condensation products to improve the adherence of melamine salts on fibrous materials is a decided advantage as the formation and fixation of the melamine salt and the impregnation of the fibre with the viscous resin solution and the coating may be effected in a single bath. A premature precipitation of the insoluble salts is prevented by the colloidal character of the solution and a complete infiltration of the precipitate into the cell structure of the textiles and fabrics is assured.

A further advantage of this method resides in a simplification of the process. When using other coating methods it is generally necessary to apply three consecutive treatment stages, whereas one or two coating steps will be sufficient in the instant case.

The following example may serve to illustrate the invention:

15 parts of 40% formaldehyde solution, slightly alkalinized by sodiumhydrate (pH 8), 12.6 parts of melamine, and 40 parts of water are heated and kept boiling for 20-30 minutes until the formation of the methylol is completed; no precipitate must be formed in the solution when diluted with an excess of water. After cooling one part of thiourea in 20 parts of water is added.

A fabric is impregnated with a 5% solution of pyrophosphoric acid; the excess of the liquid is removed by wringers; the impregnated fabric is passed through a warm chamber; it should not be completely dried to prevent weakening of the fibre. Hereafter, the fabric is passed into the melamine methylol solution. A white precipitate is formed in the fibres. The fabric is again passed through wringers and then heated to 120-140° C., whereby the resin becomes insoluble. The fabric is now ready for use.

The effect of this impregnating method is startling. Not only has the fabric become completely unflammable, but it will retain this property even after a week's exposure or staying in water.

When colored fabrics are used, they will not appreciably change in color; a somewhat cloudy shade may be imparted to them sometimes; this, however, may be easily remedied by the use of a suitably colored solution of the resin.

Colors can also be applied to the treated materials by dyeing; but in this case it is important that the insoluble precipitate is carefully and very uniformly applied to the goods, so as to prevent color spots or stains.

My method can be applied with equally good results to cotton goods, artificial silk, wool and similar materials.

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It is also well applicable to cover a silk or artificial silk textile with a coating to diminish its gloss. In this case, a strongly diluted solution may be used, whereby the fabric itself is not materially changed.

The example given above is not considered to be a limitation of the application or of the methods and products of the invention, because variations in percentages of the material used, in the time and temperature of the different heating steps in the sequence thereof, in the mode of application and in the use of the products of reaction can be made by one skilled in the art to suit different requirements.

I claim:

1. As a new product of manufacture a non-inflammable waterproof organic fibre material impregnated with a water insoluble melamine salt of pyrophosphoric acid and coated with a melamine formaldehyde condensation product.

2. As a new product of manufacture a non-inflammable waterproof organic fibre material impregnated with a water insoluble melamine of pyrophosphoric acid and coated with a mixed melamine thiourea formaldehyde condensation product.

3. As a new product of manufacture a non-inflammable waterproof organic fibre material impregnated with a water insoluble melamine methylol pyrophosphate and coated with a melamine formaldehyde condensation product.

4. As a new product of manufacture a non-inflammable waterproof organic fibre material impregnated with water insoluble melamine methylol pyrophosphate and coated with a mixed

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melamine thiourea formaldehyde condensation product.

5. A method of producing non-inflammable waterproof textiles and fabrics comprising treating the said materials with a solution of pyrophosphoric acid and of melamine methylol, imparting thereby to the fibres a water insoluble precipitate of the melamine and of the pyrophosphoric acid, and drying the thus treated materials at a temperature of about 120 to 140° C.

6. A method of producing non-inflammable waterproof organic textiles and fabrics comprising treating the said materials with a solution of pyrophosphoric acid and of melamine methylol and thiourea, imparting thereby to the fibres a water insoluble precipitate of the melamine and of the pyrophosphoric acid and drying the thus treated materials at a temperature of about 120 to 140° C.

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