

ALIEN PROPERTY CUSTODIAN

PROCESS FOR THE MANUFACTURE OF PLASTIC MATERIALS AND NEW INDUSTRIAL PRODUCTS RESULTING THEREFROM

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The gelatinisation of secondary cellulose acetate or diacetate takes place easily by malaxation with acetone in open receptacles. On the contrary it is not possible to gelatinise by this means the cellulose acetates of an acetyl content greater than 58%, commonly known as triacetates, and it is necessary in order to succeed in this to replace the acetone by a solvent of the triacetate. Now, the choice of such solvents is limited to toxic chlorinated solvents and to a few non-chlorinated and expensive solvents. It has however likewise been proposed to gelatinise the triacetates at temperatures of 150-190° C in cyclohexanone, benzyl acetate and so on, but this method has the inconvenience of necessitating the employment of large quantities of these solvents of high boiling point.

According to our present invention, we have disclosed that it is possible to prepare with ease plastic materials having a base of cellulose acetates of an acetyl content greater than 58% by treating the latter by a light or medium organic liquid capable of gelatinising them at a temperature above its boiling point under atmospheric pressure, this treatment taking place in a closed receptacle at a temperature above the boiling point of the organic liquid considered and therefore under pressure; then in transforming the gel obtained into a tenacious material according to customary processes.

The acetates of cellulose possessing an acetyl content greater than 58%, or triacetates, to which the invention is applicable are those which can be obtained by starting from natural cellulose, principally in accordance with the processes said to be in a dissolving or homogeneous medium and those said to be in a heterogeneous medium.

The words "light or medium organic liquids" are intended to indicate the organic liquids or mixtures of organic liquids having a boiling point lower than 150°C at atmospheric pressure. These liquids can be classed in three categories in view of their dissolving or gelatinising action in respect of the triacetates:

- a) those which are already solvents or gelatinisers of the triacetates at a temperature below their boiling point;
- b) those which are only solvents or gelatinisers at a temperature above their boiling point;
- c) those which even at a temperature above their boiling point have no solvent or gelatinising effect upon the triacetates.

It is necessary in order to carry out our invention to utilise an organic liquid belonging to

the first or second of these categories. With the liquids of the first category it is possible to gelatinise the triacetates without working under pressure, but recourse to this measure, in accordance with our invention, accelerates the gelatinisation. With the liquids of the second category only the application of the process of our invention permits of the gelatinisation.

As examples of liquids of the first category may be mentioned methylene chloride, mixtures of methylene chloride and lower alcohols, dioxane, mixtures of dioxane and acetone, mixtures of chloroform and lower alcohols.

As examples of liquids of the second category may be mentioned acetone, ethyl acetate, mixtures of acetone and ethyl acetate, of acetone and benzene, of benzene and methyl alcohol and the majority of the solvents of the diacetate.

As some of these examples show, the liquids of the first or the second category can be constituted by mixtures containing liquids belonging to the third category, that is to say having no solvent or gelatinising effect with respect to triacetates, or even by mixtures composed exclusively of liquids of the third category. It is in fact known that a mixture of non-solvent liquids for a given cellulose ester may constitute a solvent for the same ester. In the case of any liquid or mixture of liquids it is easy to determine by experiment to which of the three above categories it belongs and consequently whether it is applicable for carrying out the invention.

In order to carry out the invention it is possible to employ cellulose triacetate of fibrous nature in the form of flocks or in the form of blocks obtained by compression of flocks, which can bring its apparent density to 0.8 for example; these blocks moreover very easily break down in malaxation. According to the invention these flocks or blocks are mixed with the light or medium organic liquid and if desired with other solvents or gelatinisers, diluents, plasticisers, colouring materials, filling materials, other cellulose derivatives, synthetic resins and other ingredients employed in the manufacture of plastic materials. This mixture can advantageously be made in an autoclave with agitation or in a closed malaxator, or in any other appropriate apparatus. During the operation the apparatus is brought to a temperature above the boiling temperature under normal pressure of the most volatile of the liquids employed.

As plasticisers can be employed in particular the phenols substituted by at least one aliphatic or isocyclic chain having more than two atoms of

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carbon, their halogenated derivatives or their derivatives substituted by the group OH. As examples of such plasticisers may be mentioned thymol, monochlorinated or dichlorinated para-tertiary butyl phenol, iso-octyl phenol, the cyclohexyl phenols and their hydroxy-ethyl, dihydroxy-propyl, acetyl and benzoyl derivatives. These products are obtained according to the general known methods by condensation of phenols with alcohols or olefines, followed if desired by halogenation and/or acylation or hydroxy-alkylation.

The gel of tri-acetate obtained according to the process described above is subsequently transformed into a tenacious material according to one of the customary processes for the transformation of gels. It is possible for example to laminate them in the hot or cold in order to obtain sheets which are stacked one on top of the other; the block thus obtained is subjected to pressure in the hot, then to cutting according to the customary practice in the manufacture of celluloid. It is also possible to transform the block into brittle leaves and to grind the latter in order to obtain a moulding powder which is converted into finished products by moulding under pressure. In the course of the laminating or grinding or at any other time in the manufacture it is possible to add to the mass filling materials, colouring materials and other ingredients.

The following examples illustrate the invention, without limiting the same thereto:

Example 1

	Parts
Cellulose tri-acetate, (acetyl content 61,5) --	100
Acetone -----	200
Chlorinated butyl phenol -----	100

are charged into an autoclave and malaxated for some hours at 110° C. The pressure rises to 5 kgs. The mass is converted into a homogeneous gel which, after cooling, can be laminated, then compressed and cut into thin sheets in accordance with practice analogous to that employed in the manufacture of celluloid. Furthermore, this gel can be converted into moulding powders according to customary practice.

When operation is conducted in an open receptacle the temperature is limited to the boiling temperature of the acetone and it is impossible to obtain a gel with the same charge.

Example 2

	Parts
Cellulose tri-acetate, (acetyl content 61,5) --	100
Ethyl acetate -----	200
Chlorinated butyl phenol -----	50
Tri-acetin -----	25

are charged into a malaxator under pressure and heated to 120° C. for some hours. The pressure

rises to 3,500 kgs. A gel is obtained which, after cooling, can be treated by lamination as described in Example 1.

By operation without using pressure and even by working with a reflux condenser at the boiling point of the ethyl-acetate, it is impossible to obtain a gel under the conditions set forth.

Example 3

	Parts
Cellulose tri-acetate, (acetyl content 61,5) --	100
Acetone -----	60
Benzene -----	96
Methyl alcohol -----	44
Chlorinated o-phenyl-phenol -----	100

are heated for some hours under pressure at 120° C and the gel obtained is worked as described in Examples 1 and 2.

By operating with the same charge but without using pressure it is possible to obtain gelling of the cellulose acetate.

Example 4

The method of working described in the preceding examples is applied to a mixture constituted by:

	Parts
Cellulose tri-acetate, (acetyl content 61,5) --	100
Benzene -----	100
Acetone -----	100
Chlorinated butyl phenol -----	100

The triacetate can be employed in the form of blocks previously compressed in a press, which increases the apparent density to 0.6.

Without working in an autoclave it is impossible to obtain a gel under the conditions set forth.

Example 5

	Parts
Cellulose tri-acetate, (acetyl content 61,5) --	100
Benzene -----	100
Methyl alcohol -----	110
Chlorinated butyl phenol -----	100

are malaxated for some hours under pressure at 120° C and the gel obtained is treated as described in examples 1 and 2.

Example 6

	Parts
Primary cellulose acetate having an acetyl content 58.5 -----	100
Ethyl-toluene-sulphamide -----	16
Tri-acetine -----	34
Acetone -----	150

are charged in an autoclave and malaxated for eight hours at 80° C. The mass becomes transformed into a homogeneous gel which is treated as described in Examples 1 and 2.

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