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ARTIFICIAL MATERIAL AND PROCESS FOR MAKING SAME

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Heretofore strong sulphuric acid has not been used as coagulating bath in the manufacture of artificial materials, such as artificial threads and films from viscose, because, by means of such acid, the formation of a lustrous thread, band, film or the like has been impossible. (See U. S. Patent 836,452.)

Now I have discovered that this holds good with sulphuric acid containing up to about 55 per cent. of H_2SO_4, but that sulphuric acid containing about 55 per cent. of H_2SO_4, and preferably sulphuric acid containing more than 55 per cent., for example 65 to 86 per cent. of H_2SO_4, when used as coagulating bath for viscose brought into the form of a thread or film or band or the like, yields threads, films, bands or the like which have a good lustre.

But what is still more important: I have discovered that threads, manufactured from viscose by means of sulphuric acid containing at least about 55 per cent. of H_2SO_4, preferably however more than 55 per cent. of H_2SO_4, for example 70 to 85 per cent. of H_2SO_4, with regard to their tensile strength, surpass any artificial thread known hitherto and in some cases approach, in some cases equal, and in some cases even surpass even natural fibres, such as cotton or wool or real silk. For example, it offers no difficulty to produce according to the present process, artificial silk or staple fibre that has a dry tenacity of more than 2 grammes and even more than 3 grammes per denier, for example 3 or even 3,5 to 4 grammes per denier and in some cases even more than 4 grammes per denier and a wet tenacity of 1,5 to 2,5 grammes per denier and more.

A further advantage of the artificial threads manufactured according to the present process over ordinary viscose silk is their higher resistance to aqueous alkalis and to soap.

Considering that, according to the present process, sulphuric acid containing up to 90 and even 95 per cent. of H_2SO_4 gives excellent results, this discovery is extremely surprising for two reasons: First of all, with a view to the well-known fact that strong sulphuric acid, for example such containing 60 per cent. or more of H_2SO_4, have an energetic depolymerizing action on cellulose, just the contrary was to be expected, namely that strong sulphuric acid would decompose the coagulated thread during or after coagulation and thus render it weak, brittle and little resistant to water. In other words: It was to be expected that strong sulphuric acid would produce a thread weaker than a viscose thread made by means of any other coagulating bath known hitherto. All the more amazing is the fact, that strong sulphuric acid yields viscose threads much stronger than any viscose thread known hitherto. The second reason why the present invention is surprising is the lustre of the threads, bands or films, considering that, up to now, it has been believed that the want of lustre in the threads made by means of somewhat stronger acid had been caused by the fact that not enough water had been present in the coagulating bath to dissolve the gaseous by-products.

I have further found that it is advantageous to the tensile strength of the artificial materials, particularly threads, manufactured according to the present process, to stretch the threads either in the coagulating bath or between the coagulating bath and the collecting device or in both places.

The process is carried out in such a manner that viscose is brought into the form of an artificial material and coagulated by means of sulphuric acid, containing at least 55 per cent. of H_2SO_4, for example sulphuric acid containing 55 to 85 per cent. of monohydrate. In most cases, for instance in manufacturing artificial threads or filaments, or films, or bands, or plates, this can be effected in such a manner that the viscose is caused to pass through suitably formed openings into sulphuric acid, containing 55 to 98 per cent. of H_2SO_4, for instance such of 65 to 85 per cent. strength (calculated on H_2SO_4) or into a bath containing 55 to 98 per cent., for instance 65 to 85 per cent. of monohydrate. The strong sulphuric acid may be employed by itself or in admixture with one or more suitable inorganic substances, for example with another strong mineral acid such as

hydrochloric acid, nitric acid or phosphoric acid, or with a neutral or acid salt, such as sodium sulphate, sodium bisulphate, ammonium sulphate, magnesium sulphate, zinc sulphate, sodium bisulphite, sodium sulphite, sodium nitrite, boric acid or the like. To the strong sulphuric acid or its mixture with another strong acid or with one or more of the inorganic substances mentioned above, there may be added one or more organic bodies, such as glycerol or a sugar, for example glucose, or alcohol or a salt of an organic base, for example of aniline, or an organic acid, such as acetic acid or formic acid or lactic acid or oxalic acid. If a salt is added which is capable of reacting with the strong sulphuric acid under formation of an acid sulphate, or which mutually interacts with the sulphuric acid, the strength of the sulphuric acid should be so chosen that, after the amount required for the formation of the acid sulphate or for the mutual interaction is used up, the coagulating bath contains free sulphuric acid of the desired strength, but at any rate not less than 55 parts by weight of monohydrate in 100 parts by weight of the precipitating bath.

The strength of the sulphuric acid depends, ceteris paribus, partly on the time of maturing given the alkali cellulose, partly on the time or ripening given the finished viscose and partly on viscosity or amount of cellulose contained in the viscose. As a rule, to which however the invention is not confined, it may be stated that viscoses which have matured for several days and/or viscoses that have been prepared from alkali cellulose that has been allowed to mature for a longer time, for example 2 to 3 days and/or viscoses that are poor in cellulose or little viscous respectively, do not support so strong a sulphuric acid as viscoses that have not been allowed to mature or have been allowed to mature for a short time (for instance 3 to 48 hours) only and/or have been prepared from alkali cellulose that has been not allowed to mature at all or has been allowed to mature for a shorter time than usual, for example for 3 to 48 hours, and/or are rich in cellulose or viscous respectively. The strength of the sulphuric acid is further, to a certain degree, dependent on the length of immersion in the coagulating bath and the tension given the coagulated material in the acid or outside it, for example between the coagulating bath and the collecting device, for example bobbin.

The precipitating bath may be kept at room temperature or at a temperature exceeding room temperature, for example at 25 to 50° C., or at a temperature lower than room temperature, for example at 0 to 10° C., or even below 0° C., for example at from −1 to −10° C.

The length of immersion of the thread or filament or plate or film or band in the strong sulphuric acid or in the bath containing it may be varied within wide limits, for examples from 3 to 60 centimetres and even more, for instance 1 to 2 meters.

If desired the thread or film or band may be stretched either immediately after its formation, i. e. in the coagulating bath containing, or consisting of, strong sulphuric acid, or subsequently, i. e. between the coagulating bath and the collecting device, such as bobbin or centrifuge, or both in the coagulating bath and between the coagulating bath and the collecting device.

The thread or film or band or plate or coated or impregnated material may, after washing, be heated or steamed before or after drying.

Any viscose prepared according to any process or any method known hitherto may be used in the present process. The process gives excellent results with viscoses that are prepared from alkali cellulose that has been allowed to mature for such a time as it is commonly given the alkali cellulose in the artificial silk or film art and also with viscoses in the manufacture of which alkali cellulose is employed which has been not allowed to mature at all or has been allowed to mature for a shorter time than such as is usual in the artificial silk or film art, i. e. not longer than about 48 hours, for example 1 to 24 hours.

The present process gives excellent results with such viscoses also as are prepared without making alkali cellulose, i. e. by direct mixing the ingredients.

If desired, there may be added to the viscose one or more suitable substances known in the viscose-silk art, for example glycerol or glucose or sodium sulphate or sodium sulphite or sodium bisulphite or ammonium sulphate or an alkali silicate or an alkali aluminate or ammonia.

The process being open to manifold variations, it is not intended to confine the invention to the following examples given for the practical carrying out of the process.

I (a) to (c)

(*a*). 100 parts by weight of sulphite-pulp or linters are impregnated with 900 to 2000 parts by weight of a caustic soda solution of 18 per cent. strength at 15 to 18° C., and the mixture allowed to stand for 3 to 24 hours. Then the mass is pressed to 300 to 400 parts by weight, and the residue comminuted by hand or in a suitable apparatus, for instance in a cooled shredder. The comminuted soda cellulose is then kept for 60 to 72 hours at room temperature, whereafter 30 to 60 parts by weight of carbon bisulphide are added, and the reacting mixture, preferably under stirring (for example in a closed kneading machine) or agitating (for example in a sulphidizing drum), kept for several (for example

4 to 8) hours at 16 to 20° C. Thereafter the excess of the carbon bisulphide is removed by driving off or evacuating, and then the sulphidized mass is dissolved in water or dilute caustic soda solution in such a manner that the viscose contains about 8 to 12 per cent. of cellulose (determined in the known manner by precipitating with acid, washing and drying). If caustic soda solution is used, it may be for example so chosen that, if the amount of caustic soda that was present in the soda cellulose is taken into consideration the final viscose contains 5 to 8 per cent. of caustic soda. The viscose solution thus obtained is then filtered in a known manner through cotton wool or a dense cotton fabric or through both and freed from gas bubbles if any. Immediately after, or after it has been allowed to ripen at 16–18° C. for a shorter (for example 12 hours to two days) or a longer (for example four days to seven days) time, the viscose is spun in a known manner into one of the following precipitating baths:

(1) Sulphuric acid of 50 to 55° Bé., or

(2) Sulphuric acid of 45 to 50° Bé., or

(3) A solution of 13,3 parts by weight of ammonium sulphate in 120 parts by weight of sulphuric acid of 50 to 55° Bé., to which 7 to 9 parts of sulphuric acid of 66° Bé. have been added, or

(4) A solution of 10 to 15 parts by weight of glucose in 190–185 parts by weight of sulphuric acid of 45 to 55° Bé.

The temperature of the coagulating bath is kept at 0 to 16° C., for instance at 8° C., or at −4 to −8° C.

The length of immersion in the coagulating bath of the threads forming therein may be short, for example 10 centimetres or long, for instance from 30 to 100 centimetres and more. They are then collected in a known manner on a bobbin or in a spinning centrifuge which may be placed either by the precipitating bath or at a distance from 20 to 120 centimetres from the bath, whereupon the threads are washed and dried.

The threads may be stretched in any known manner whilst they are in the coagulating bath, or after they have been removed from the bath, i. e. between the coagulating bath and the collecting device. This may be done for example either by choosing a more or less long distance between the bath and the collecting device, or by leading the threads over rods or hooks arranged in the bath or between the bath and the collecting device, or in the bath and between the bath and the collecting device.

The speed of spinning may be varied within wide limits. As a rule to which, however, the invention is not intended to be confined, it may be assumed that the upper limit of speed possible is dependent partly upon the temperature of the coagulating bath, partly upon the length of immersion, partly upon the distance between the coagulating bath and the collecting device, partly upon the degree of stretching given the thread during spinning, and partly upon the quantity of viscose delivered by the pump. The following examples to which, however, the process is not intended to be limited, may be adduced for the purpose of illustration.

(1) If the coagulating bath is used at a temperature below 0° C., for example at minus 5° C., and if the length of immersion is 60 to 120, for example 80 centimetres, and if the distance between the coagulating bath and the collecting device, for example bobbin, is 60 to 150 centimetres, for instance 120 centimetres, and if the stretching given the thread between the coagulating bath and the collecting device is comparatively high, and if the pump delivers about 2 to 3 cubic centimetres of viscose a minute, then excellent results are obtained with a speed of 18 to 26 meters a minute, or of 30 meters a minute, but also a higher speed, for example of 30 to 40 meters a minute may be employed if desired.

(2) If all spining conditions remain the same as in (1), except that the pump delivers 4 to 5 cubic centimetres of viscose a minute, then if desired a speed up to 40 to 60 meters a minute may be employed.

(3) If the spinning conditions are exactly as in (1), except that the temperature of the coagulating bath is plus 4° to plus 25° C., for example 8° C., if desired the speed may be up to 50 to 60 meters a minute.

If in (1) the length of immersion or the distance between the coagulating bath and the collecting device or both are shortened, it is recommendable not to use too low a speed.

Before being washed, the threads may be treated in any known manner with a coagulating bath known in the viscose-silk art, for example a solution of sodium bisulphate or sodium bisulphite or with dilute sulphuric acid or the like.

The washed threads may, before or after drying, be heated (for example at 100–110° C.) or steamed.

They may also be treated with any desulphurating or bleaching agent known in the viscose art.

(*b*). The mode of operation is as in (*a*), except that 75 to 150 parts by weight of carbon bisulphide are employed for the sulphidizing of the soda-cellulose.

(*c*). The mode of operation is as in (*a*) or (*b*), with the difference that the soda-cellulose is pressed to 200 parts by weight.

II (a) to (c)

Mode of operation as in Example I (*a*) or I (*b*) or I (*c*), with the difference

(1) that the sulphidized mass is dissolved

in water or dilute caustic soda solution so as to yield a viscose that contains 5 to 8 per cent. of cellulose, for instance, 6 to 7 per cent. of celulose (determined in the known manner by precipitating with acid, washing and drying).

(2) That as coagulating bath one of the following baths is used:

(1) Sulphuric acid of 55 to 60° Bé., or

(2) Sulphuric acid of 50 to 55° Bé., or

(3) A solution of 13,3 parts by weight of ammonium sulphate by weight in 120 parts by weight of sulphuric acid of 50 to 60° Bé. to which 7 to 9 parts of sulphuric acid of 66° Bé. have been added, or

(4) A solution of 10 to 15 parts by weight of glucose in 190 to 185 parts by weight of sulphuric acid of 55 to 60° Bé.

III (a) to (c)

Mode of operation as in Example I (*a*) or I (*b*) or I (*c*) or as in Example II (*a*) or II (*b*) or II (*c*), with the exception that the viscose is not allowed to mature at all or to mature for 12 to 48 hours only, and that as coagulating bath sulphuric acid of 60 to 64° Bé., for instance sulphuric acid of 62° Bé. at 6 to 10° C. is used.

In using so strong an acid it is preferable to run the spinning at a high speed, for example 40 to 60 meters a minute.

IV (a) to (e)

(*a*). 100 parts by weight of sulphite-pulp or linters are impregnated with 900 to 2000 parts by weight of a caustic soda solution of 18 per cent. strength at 15 to 18° C., and the mixture allowed to stand for 3 to 24 hours. Then the mass is pressed to 300 parts by weight, and the residue comminuted by hand or in a suitable apparatus, for instance in a cooled shredder. Immediately or soon (for example 1 hour) after the comminution has taken place, 30 to 60 parts by weight of carbon bisulphide are added, and the reacting mixture, preferably under stirring (for example in a closed kneading machine) or agitating (for example in a sulphidizing drum), kept for several (for example 4 to 12) hours at 16 to 20° C. Thereafter the excess of the carbon bisulphide is removed by driving off or evacuating, and the sulphidized mass, the weight of which generally amounts to from about 330 to 400 parts by weight, is dissolved in 650 to 900 parts by weight of a caustic soda solution of 6.5 to 10 per cent. strength. The viscose solution thus obtained is then filtered in a known manner through cotton wool or a dense cotton fabric or through both and freed from gas bubbles if any. Immediately after, or after it has been allowed to ripen at 16–18° C. for a shorter (for example 12 hours to two days) or a longer (for example four days or seven days) time, the viscose is spun in a known manner into one of the following precipitating baths:

(1) Sulphuric acid of 60° Bé., or

(2) Sulphuric acid of 55° Bé., or

(3) A solution of 13.3 parts by weight of ammonium sulphate in 120 parts by weight of sulphuric acid of 55 to 60° Bé., to which 9 to 10 parts of sulphuric acid of 66° Bé. have been added, or

(4) A solution of 10 to 15 parts by weight of glucose in 190–185 parts by weight of sulphuric acid of 55 to 60° Bé.

The temperature of the coagulating bath is kept at 0 to 16° C., for instance at 8° C., or at −4 to −8° C.

The spinning and treating of the threads during and after spinning is done in the same manner as described in the foregoing examples.

(*b*). The mode of operation is as in (*a*), with the difference that a caustic soda solution of 3 to 5 per cent. strength is employed for the dissolving of the sulphidized soda cellulose. Accordingly, the viscose is somewhat more viscous.

(*c*). The mode of operation is as in (*a*) or (*b*), except that 75 to 150 parts by weight of carbon bisulphide are employed for the sulphidizing of the soda-cellulose.

(*d*). The mode of operation is as in (*a*) or (*b*) or (*c*), with the difference that the soda-cellulose is pressed to 200 parts by weight.

(*e*). The mode of operation is as in (*a*) or (*b*) or (*c*) or (*d*), with the difference that sulphuric acid of 62 to 64° Bé. at 8° C. is used as coagulating bath, the viscose being 12 to 48 hours old and the speed of spinning about 45 to 60 meters a minute.

V (a) to (e)

The method of preparing the viscose differs from that described in IV (*a*) or (*b*) or (*c*) or (*d*) or (*e*) in that the soda-cellulose, after pressing and comminuting and before being sulphidized, is allowed to ripen for 3 to 24 hours at 16 to 20° C. The amount and concentration of the caustic soda solution used for the dissolving of the sulphidized mass may be equal to those used in Example IV or somewhat lower, for example so as to produce a 10 to 12 per cent viscose (calculated on starting cellulose).

The working up into threads is effected as in IV (*a*) to (*e*).

VI (a) to (d)

(*a*). 80–100 parts by weight of finely divided sulphite-pulp or bleached cotton are mixed with 920–900 parts by weight of a caustic soda solution of 8 to 10 per cent. strength. To this mixture 40 to 150 parts by weight of carbon bisulphide are added and the reacting mixture kept at room temperature for 12 to 72 hours, preferably under continual or temporary stirring or kneading. The viscose solution thus obtained is then

filtered in a known manner through cotton wool or a dense cotton fabric or through both and freed from gas bubbles if any. Immediately after, or after it has been allowed to ripen at 16–18° C. for a shorter (for example 12 hours to two days) or a longer (for example four to seven days) time, the viscose is spun in a known manner into one of the following coagulating baths:

(1) Sulphuric acid of 55 to 60° Bé., or

(2) Sulphuric acid of 45 to 50° Bé., or

(3) A solution of 13,3 parts by weight of ammonium sulphate in 120 parts by weight of sulphuric acid of 45 to 60° Bé., to which 7 to 9 parts of sulphuric acid of 66° Bé., have been added, or

(4) A solution of 10 to 15 parts by weight of glucose in 190 to 185 parts by weight of sulphuric acid of 45 to 60° Bé.

The temperature of the coagulating bath is kept at 0 to 16° C., for instance at 8° C., or at −4 to −8° C.

The spinning and treating of the threads is done as in the foregoing examples.

(*b*). Mode of operation as in (*a*), with the exception that the viscose is prepared according to the method 3 described in the British Patent 212,865, for example according to the following examples of said specification: I (*a*) to (*s*) or II (*a*) to (*c*) or III (*a*) to (*c*) or IV (*a*) to (*e*) or V (*a*) to (*b*) or VI (*a*) to (*c*) or VII or VIII or IX or X or XII or XIII.

(*c*) Mode of operation as in (*a*) or (*b*), with the exception that, before or after the addition of carbon bisulphide takes place, a small quantity of a catalyzer is added to the mixture of the cellulosic body with the caustic alkali solution, for example 0,2 to 1 parts by weight of a soluble chromic salt or nickel salt or iron salt.

(*d*) Mode of operation as in (*a*) or (*b*), with the difference that, before or after the addition of carbon bisulphide takes place, a small quantity of a soluble peroxyde, such as 5 to 20 parts by weight of sodium peroxyde to 100 parts by weight of starting cellulose are added.

VII

100 parts by weight of sulphite-pulp are placed in a cooled shredder admitting of being hermetically closed, whereupon 200 parts by weight of a caustic soda solution of 12 to 20 per cent. strength are added, preferably in small portions. As soon as the mass is homogeneous, 40 to 60 parts by weight of carbon bisulphide are added, whereupon the shredder is closed. After 4 to 8 hours the reaction mass is removed from the shredder and dissolved in 920 parts by weight of a caustic soda solution of 6,15 per cent, strength, the weight of the sulphidized mass being 330 parts. The final viscose contains 8 per cent. of starting cellulose.

The filtering and spinning of this viscose and the treatment of the threads is done as in the foregoing examples.

Examples for producing staple fibre follow automatically from the foregoing examples.

According to the foregoing examples, it is possible to obtain artificial silk or staple fibre which has a considerably higher dry and wet tenacity than silk spun under same spinning conditions, but by means of chemicals customary in the viscose silk-art, for example by using a known precipitating bath. For example: it is not difficult to produce after the present process artificial silk having a dry tenacity of more than 2 grammes per denier, even more than 3 grammes per denier, for example 3 grammes to 4 grammes per denier and a wet tenacity of 1,5 to 2,5 grammes per denier and more.

VIII

A viscose prepared as described in Example I or II or III or IV or V or VI or VII is caused to pass through a suitable hopper or slit in one of the coagulating liquors mentioned in these examples, and after having left the precipitating bath, is washed and dried in any known manner.

IX

A cotton fabric is impregnated or filled or coated once or several times with a viscose prepared according to one of the methods described in Example I or II or III or IV or V or VI or VII, for which purpose any suitable machine, such as a padding machine or a back filling machine or a spreading machine may be employed. To the viscose there may be added a filling substance, for instance talcum or china clay or a colouring matter or a pigment such as mica, or soot or a mineral colour or the like. The impregnated or coated material is, without being dried, optionally in the stretched state, taken through one of the precipitating baths described in Example I, whereupon the material is washed and dried.

Instead of strong sulphuric acid there may be used in the foregoing examples a strong halogen hydracid, such as hydrochloric acid, for example of 25 to 40 per cent. strength, or nitric acid, for example such as contains 60 to 90 per cent. of HNO_3, or phosphoric acid, for example of 1,5 to 1,86 specific gravity, or arsenic acid, for example such as contains from 60 to 90 per cent. of H_3AsO_4.

In the foregoing examples, in the preparation of viscose, instead of wood pulp, there may be used bleached cotton, or cellulose treated in the cold or in the heat with dilute acids, for example hydrochloric or sulphuric acid, in short any kind of cellulosic bodies employed in, or proposed for the manufacture of viscose.

The expression "strong sulphuric acid" or "sulphuric acid containing at least 55 per cent. of sulphuric acid monohydrate" means in the description and claims: sulphuric acid of 55 to 98 or 100 per cent. strength.

The expression: "artificial material" in the description and claims is intended to mean: artificial threads and filaments of any kind, for example artificial silk, staple fibre, artificial cotton, artificial wool, artificial hair, films, plates, coatings and fillings of any kind.

The expression "heating" in the claims is intended to cover any known kind of heating, steaming included.

The statements in the description and claims regarding percentages relate to percentages by weight.

I claim:

1. Process for manufacturing artificial materials from viscose, which comprises bringing a viscose, prepared from an alkali cellulose which has been allowed to mature only from naught to 48 hours, into the form of an artificial material and acting upon it with sulphuric acid containing at least 55 per cent. of sulphuric acid monohydrate.

2. Process for manufacturing artificial materials from viscose, which comprises causing a viscose, prepared from an alkali cellulose which has been allowed to mature only from naught to 48 hours, to pass through suitably formed openings into a coagulating bath which contains at least 55 per cent. of sulphuric acid monohydrate.

3. Process for manufacturing artificial materials from viscose, which comprises causing a viscose, prepared from an alkali cellulose which has been allowed to mature only from naught to 48 hours, to pass through suitably formed openings into a coagulating bath containing at least 55 per cent. of sulphuric acid monohydrate and keeping the bath at a temperature below room temperature.

4. The process for manufacturing artificial material which comprises bringing viscose, prepared from an alkali cellulose which has been allowed to mature only from naught to 48 hours, into the form of an artificial material and acting upon it with a strong mineral acid of a strength equivalent to at least 55 per cent. sulphuric acid monohydrate.

5. The process for manufacturing artificial threads of high dry tenacity which comprises bringing a viscose, prepared from alkali cellulose which has been allowed to mature only from naught to 48 hours, into the form of an artificial thread and acting upon it with strong mineral acid of a strength equivalent to at least 55 per cent sulphuric acid monohydrate.

6. The process for manufacturing artificial threads of high dry tenacity which comprises causing a viscose, prepared from alkali cellulose which has been allowed to mature only from naught to 48 hours, to pass through a suitably formed opening into a bath which contains a mineral acid present in an amount equivalent to at least 55 per cent. sulphuric acid monohydrate.

7. The process for manufacturing artificial thread of high dry tenacity which comprises causing a viscose, prepared from alkali cellulose which has been allowed to mature only from naught to 48 hours, to pass through a suitably formed opening, thereafter contacting it with a liquid which contains a strong mineral acid present in an amount equivalent to at least 5 per cent. sulphuric acid monohydrate and washing the thread.

8. The process for manufacturing artificial thread of high dry tenacity which comprises causing a viscose, prepared from alkali cellulose which has been allowed to mature only from naught to 48 hours, to pass through a suitably formed opening, thereafter acting upon it with sulphuric acid containing at least 55 per cent. sulphuric acid monohydrate and washing the thread.

9. Process for manufacturing artificial materials from viscose, which comprises bringing a viscose, prepared from an alkali cellulose which has been allowed to mature only from naught to 48 hours, into the form of an artificial material and acting upon it with a bath which has a coagulating action on said viscose and a plasticizing action on the coagulated product.

10. Process for manufacturing artificial materials from viscose, which comprises causing a viscose, prepared from an alkali cellulose which has been allowed to mature only from naught to 48 hours, to pass through suitably formed openings into a bath of concentrated acid coagulating agent which bath has a plasticizing action thereupon.

In testimony whereof I affix my signature.

DR. LEON LILIENFELD.