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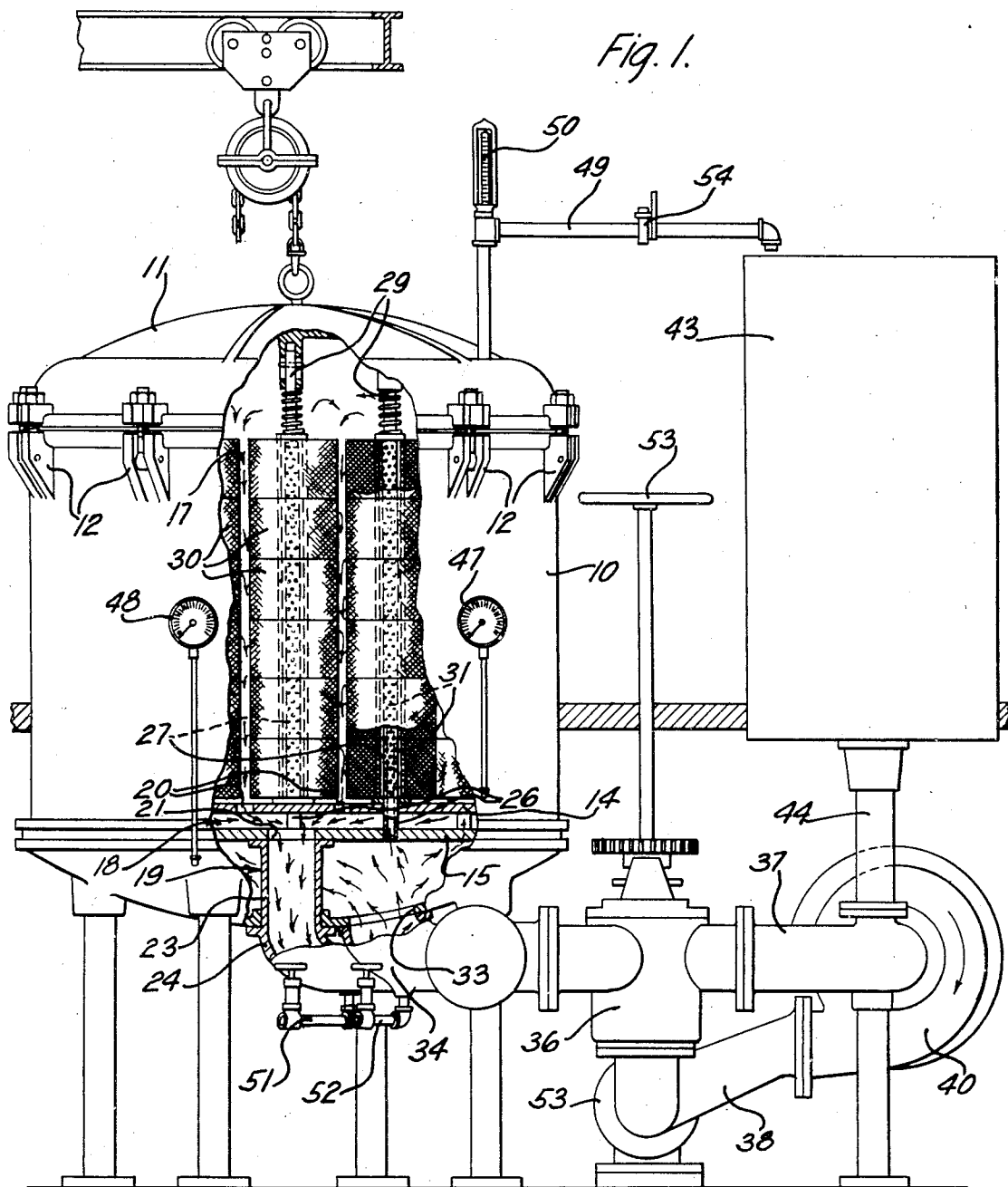
M. C. LEWIS ET AL

1,980,413

INSULATING MATERIALS AND METHOD OF PREPARING INSULATING MATERIALS

Filed Aug. 24, 1930

2 Sheets-Sheet 1



Inventors
Mark C. Lewis
Alger M. Lynn
By H. A. Patterson Atty.

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Fig. 2.

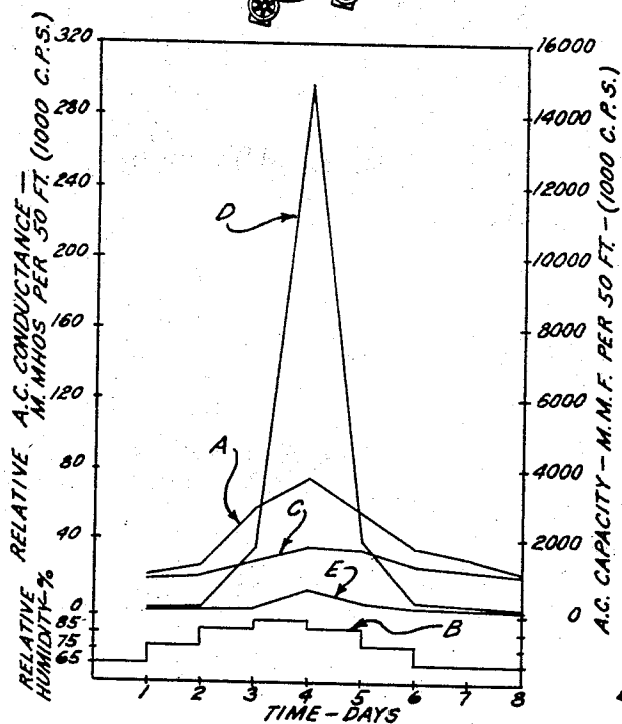
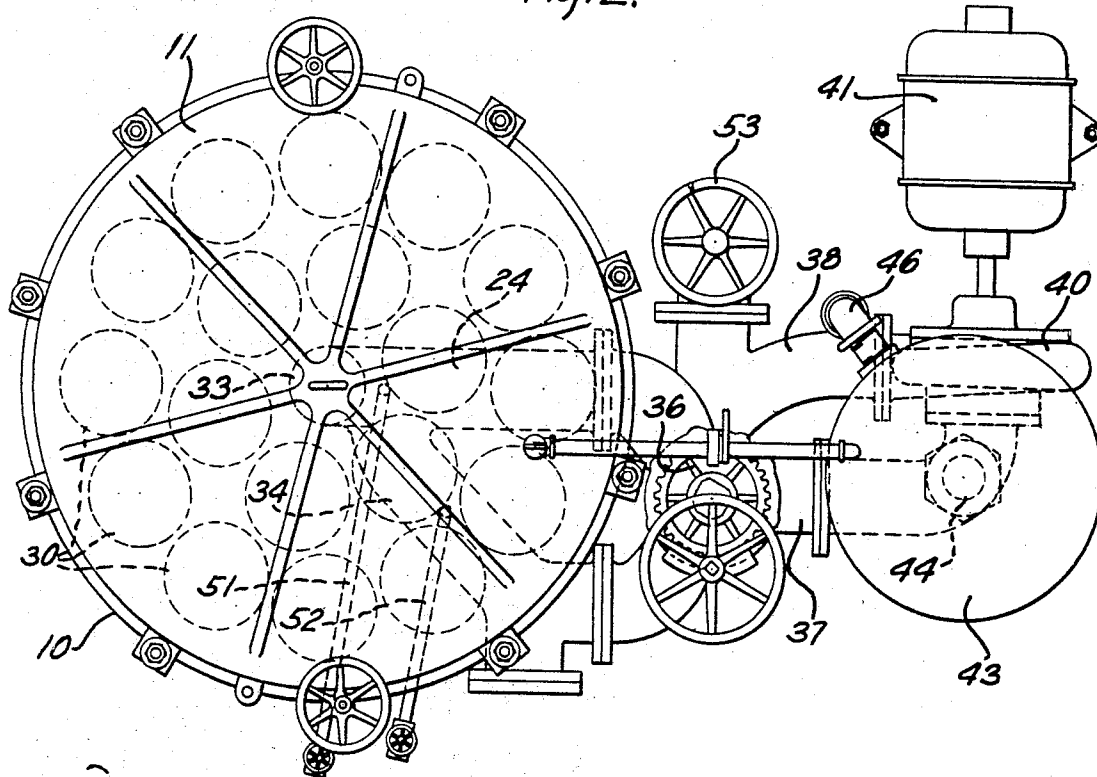


Fig. 3.

Inventors
Mark C. Lewis
Alger M. Lynn
By H.A. Patterson Atty.

UNITED STATES PATENT OFFICE

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INSULATING MATERIALS AND METHOD OF
PREPARING INSULATING MATERIALS

Mark C. Lewis, Cranford, N. J., and Alger M. Lynn, Oak Park, Ill., assignors to Western Electric Company, Incorporated, New York, N. Y., a corporation of New York

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24 Claims. (Cl. 8—2)

This invention relates generally to insulating materials and methods of preparing insulating materials and more particularly to fibrous insulating materials, such as silks, and methods of preparing them.

The characteristics of many insulating materials vary with the conditions under which they are used and it is sometimes necessary to employ an excess of certain insulating materials over the amount required for normal conditions in order to provide adequate insulation under unusual conditions. For example, the electrical characteristics of the usual commercial insulating silks, such as are employed to insulate electrical conductors, vary noticeably with variations in humidity, and in order to insure the desired electrical characteristics under all conditions, it is necessary when using such insulating silks to provide not only the amount of silk required for ordinary conditions, but also an additional quantity of the silk which is sufficient to give the required characteristics under conditions of high humidity.

The economic losses, such as increased costs of materials, additional labor, loss of time, wear on machinery, investments in additional machinery; etc., resulting from using such insulating materials are readily apparent, and furthermore the electrical characteristics of conductors insulated with such materials are also likely to be irregular.

This invention has for its objects the provision of insulating material having substantially uniform characteristics under varying conditions of service, and the provision of simple, expeditious, and economical methods of preparing such materials.

Insulating materials embodying the invention may be prepared in accordance with the invention by subjecting a fibrous material, such as insulating silk, to a plurality of hot washing fluids under pressure, at least one of the fluids including a reagent capable of neutralizing impurities in the insulating material, and the remaining fluids comprising water.

In the preparation of insulating silks by the usual methods one of the steps employed is that of degumming the silk in order to remove the gum (sericin) and other extraneous materials which may be present, from the silk fibre itself. The degumming process varies with the different kinds of silk treated, and depends upon whether the silk treated is a continuous filament silk, such as is known as tussah tram silk, or whether the silk is made of a number of discontinuous fila-

ments spun into a strand, such as is known as tussah spun silk.

In the degumming of silk by one commercial method the usual steps employed are to subject the silk to a hot solution containing an alkaline substance, such as soap, caustic soda, soda ash, sodium phosphate or the potassium compounds corresponding to these sodium compounds, then to wash the thus treated silk with water, then with a dilute solution of sulphuric acid, and finally to wash the silk several more times with water.

Another commercial method of degumming spun silk is to treat the silk with hydrochloric acid to remove soluble impurities, then to treat the silk with a degumming agent, such as one of those enumerated hereinabove, and finally to wash the silk a number of times with water.

Experiments have shown that when silk is degummed by the usual commercial methods the silk may contain small quantities of impurities introduced in the degumming process which may comprise compounds having either acid or alkaline reactions. The usual impurities present in degummed silk are sulphuric acid in the case of continuous filament silk and alkaline compounds in the case of spun silk. Since all of these materials are readily ionizable in water, insulating silks which contain them vary in electrical characteristics in accordance with the amount of moisture present therein, and the electrical characteristics of such insulating silks are likely to be seriously affected under conditions of high humidity.

Insulating silk embodying the invention, from which such ionizable substances have been removed and which in consequence has substantially constant electrical characteristics under varying conditions of service, may be produced in accordance with the methods embodying the invention which will now be described.

The methods embodying the invention may be most effectively practiced by means of apparatus of the type of the so-called "Franklin" dyeing machine disclosed in the accompanying drawings, in which

Fig. 1 is an elevational view partly broken away of a material treating apparatus;

Fig. 2 is a plan view thereof, and

Fig. 3 is a graph showing the electric characteristics of treated and untreated insulating silks.

Referring now to the drawings, in which like numerals designate like parts throughout the several views, the numeral 10 designates a cylindrical vessel having a removable cover 11 which

may be securely fastened in pressure-tight relation to the vessel 10 by means of a plurality of latches 12—12. The vessel 10 is provided with partitions 14 and 15, which act as false bottoms and divide the interior of the vessel into the several chambers 17, 18, and 19. The partition 14 is provided with a plurality of openings 20—20, which permit the flow of fluid from the chamber 17 into the chamber 18, and the partition 15 is provided with a larger central opening 21, which leads into a pipe 23, extending through the chamber 19 and through the bottom of the vessel 10 where it connects with a pipe 24. The partition 15 is provided with a plurality of openings 26—26, which are in alignment with corresponding openings in the partition 14 through which openings hollow, perforated spirals 27—27 extend, so as to permit the flow of a fluid to and from the chambers 17 and 19 without the fluid flowing into the chamber 18.

The cover 11 is provided with a plurality of spring pressed plungers 29—29, one for each of the spindles 27—27 and in alignment with these spindles, so as to exert pressure upon packages of insulating silk 30—30 wound upon springs 31—31 and positioned upon the spindles 27—27 in the manner clearly illustrated in Fig. 1. The plungers 29—29 also prevent the fluid which flows through the openings in the spindles 27—27 from passing out of the top of the channel surrounding the spindles formed by the springs 31—31 and the silk wound thereon, and the plungers thereby cause the liquid to flow through the silk under pressure so as to wash the silk.

The bottom of the vessel 10 has an opening 33 therein, at which point a pipe 34 is connected so as to convey fluid to and from the chamber 19. The pipes 24 and 34 lead from the vessel 10 to a reversing valve 36 from which pipes 37 and 38 lead to the inlet and outlet ports respectively of a centrifugal pump 40 driven by an electric motor 41. An expansion tank 43 is positioned above the pump 40 and a drain pipe 44 leads from the bottom of the tank 43 to the pipe 37 so as to discharge the contents of the expansion tank into the centrifugal pump 40. The expansion tank 43 is also provided with an overflow pipe 46 leading to a waste discharge system, of any suitable type (not shown), to prevent the contents of the expansion tank from overflowing into the room.

Steam inlet pipes 51 and 52 are provided below the vessel 10 to permit the introduction of steam into the pipes 24 and 34, respectively, and the vessel 10 is provided with pressure gauges 47 and 48 which indicate the pressure within the chambers 17 and 19, respectively. The vessel 10 is also provided with an overflow pipe 49 leading into the expansion tank 43, in which pipe is positioned a thermometer 50, by means of which the temperature within the chamber 17 may be ascertained and a valve 54 is provided to permit the flow of fluid through the pipe 49 when desired. A drain valve 53 is also provided adjacent the reversing valve 36 to drain the fluid from the apparatus into a waste discharge system (not shown) whenever it is desirable.

The operation of the apparatus is as follows:

The cover 11 is removed from the vessel 10 and a plurality of packages of silk 30—30 are positioned therein over the spindles 27—27 as shown in Fig. 1. The cover 11 is then placed upon the vessel 10 and clamped tightly in place by means of the latches 12—12, and a washing fluid is introduced into the expansion tank 43.

The pump 40 is then started, whereupon the washing fluid is pumped from the tank 43 through the pipe 38 through the reversing valve 36, through the pipe 34 and into the chamber 19 in the bottom of the vessel 10. The fluid passes from the chamber 19 up through the spindles 27—27, out through the packages of silk 30—30, down through the spaces between the packages, through the openings 20—20 in the false bottom 14 and through the opening 21 in the false bottom 15, then through the pipes 23 and 24 back through the reversing valve 36, through the pipe 37 and to the centrifugal pump 40, which then recirculates the fluid through the apparatus. Whenever it is desired to change the direction of the flow of the washing fluid the reversing valve 36 is actuated to connect the pipe 24 to the outlet port of the pump 40 and to connect the pipe 34 to the inlet port of the pump so that the liquid will enter the vessel 10 through the pipe 24 and leave the vessel through the pipe 34 in a path which is directly opposite that described hereinabove. When it is desired to heat the fluid being circulated through the apparatus, steam may be introduced into the fluid through the pipes 51 and 52 for that purpose.

The above described apparatus may be employed to practice a number of methods embodying the invention, and in order that the invention may be clearly understood one specific method embodying the invention will now be described.

In practicing this method, silk spun into strands is wound upon the springs 31—31, each spring carrying about .75 pound of silk, and the resulting packages are positioned in the vessel 10 over the spindles 27—27 in the manner described hereinbefore. Water at a temperature of about 130° F. is then introduced into the expansion tank 43 until about 1.7 gallons of water per pound of silk has been introduced therein; and the circulating pump 40 is then started, whereupon the water is caused to circulate under pressure through the silk in the manner which has already been described. A quantity of acetic acid equivalent to about .13 pound of 100% acetic acid per pound of silk is then introduced into the expansion tank 43 and forms a solution with the water therein, which is capable of neutralizing the alkaline impurities, such as degumming agents, which are present in the silk. Steam is then admitted through the pipes 51 and 52 into the streams of washing fluid passing to and from the vessel 10, which rapidly heats the solution to a temperature of about 200° F. The hot solution is circulated through the silk for about twenty minutes, during which time the direction of flow of the fluid is changed twice by actuating the reversing valve 36; then the pump 40 is stopped and the washing fluid is drained from the apparatus through the drain valve 53.

Water at a temperature of about 130° F. is then again introduced into the expansion tank 40, and the above steps, except the addition of acetic acid, are repeated. The silk is washed a number of times with hot water under pressure in the manner just described, in order to wash the silk free of ionizable materials, the number of washes depending upon the amount of impurities in the silk. Experiments have shown that a process including one washing with a hot acetic acid solution followed by four washings with hot water gives very satisfactory results.

After the silk has been subjected to the above described washing steps, it is then dried by first

centrifuging it and then heating it in a hot air drier at a temperature sufficiently high to evaporate the water retained by the silk and to volatilize the acetic acid retained therein. Any acetic acid which remains on the silk after the drying operation gradually volatilizes until substantially all of the acetic acid disappears, and in any event the quantity of acetic acid retained on the silk is so small and the acetic acid is such a weakly ionizable substance that it does not materially alter the electrical characteristics of the silk under conditions of high humidity.

It is, of course, to be understood that the method just described is merely an example of numerous methods embodying the invention which may be employed and various modifications may be made without departing from the spirit and scope of the invention. For example, when the silk has been degummed with soap it is sometimes desirable to first subject the silk to hot water under pressure, then to employ a hot washing fluid containing acetic acid, and finally to wash the silk several times with hot water. Furthermore, the number of washing steps employing either acetic acid or water may be varied in accordance with the amount of impurities present in the silk. If the silk contains a large amount of alkaline materials, the step of washing the silk with an acetic acid solution may be repeated; or if the silk contains only a small quantity of impurities, the acetic acid washing step may be omitted entirely; or one acetic acid wash may be employed and followed by less than four water washes. The quantity of acetic used in any one washing step may also be varied in accordance with the amount of alkaline materials in the silk being treated.

Nor is it necessary to use only acetic acid as a reagent for neutralizing the alkaline impurities in the silk, for any weakly ionizable acid or mixture of acids may be employed for this purpose. The volatile fatty acids are preferred, however, because they do not ionize strongly and because they either decompose or vaporize during the drying of the silk, or on standing after the silk is dried, without leaving residues which act as electrical conductors. Among the acids which may be used either alone or in combinations are formic, acetic, propionic, lactic, oxalic, tartaric, citric, carbonic and malic.

It is, of course, not essential in all cases to employ an acid solution to neutralize impurities in the silk, for if the silk should happen to contain impurities having acid reactions, an alkaline neutralizing agent, such as sodium phosphate, or other weakly ionizable alkaline material, or ammonium hydroxide, may be employed for this purpose.

The methods embodying the invention are also not limited to their use with the particular type of apparatus described hereinabove, for other apparatus may be used with comparable results. For instance, instead of employing a Franklin dyeing machine to practice the invention, the apparatus known as the "Theis dyeing machine" and "Obermaier dyeing machine", which work on a similar principle, may be employed.

As examples of the improvements which result from practicing the methods embodying the invention, strands of No. 62 spun silk three quarters of an inch in length, which have been treated in accordance with the invention, have a direct current resistance of from 2000 to 3000 kilomegohms at a relative humidity of 87.5% and a temperature of 100° F., whereas, similar strands of untreated

commercial silk have a direct current resistance of from 5 to 7 kilomegohms under similar conditions. Silk which has been treated with water alone in accordance with the invention has a resistance of about 1000 kilomegohms.

The curves shown in Fig. 3 of the drawing also clearly illustrate the improvements which result from practicing the invention. Curve A in this figure indicates the A. C. capacity of the insulation in micro-microfarads of a conductor insulated with commercial spun silk at 1000 cycles per second plotted as a function of the time in days, during which the insulated conductor was subjected to atmospheres having the relative humidities indicated by the lower curve B. Curve C is a similar curve drawn from the results obtained by employing a conductor which was identical in every respect with that employed in obtaining curve A, except that spun silk treated in accordance with the invention was employed to insulate the conductor. Curve D shows the A. C. conductance of the insulation in millimhos of a conductor at 1000 cycles per second when insulated with commercial spun silk, while curve E shows the A. C. conductance of the insulation of a like conductor insulated with spun silk which has been treated by methods embodying the invention.

The marked differences in the characteristics of silk which has been treated in accordance with the invention and silk which has not been so treated are readily apparent from these curves. It may be seen that as the relative humidity increases the A. C. capacity of a conductor insulated with commercial spun silk increases rapidly, while the A. C. capacity of a similar conductor insulated with spun silk which has been treated in accordance with the invention rises only a comparatively small amount and remains substantially constant. Similarly the A. C. conductance of the insulation of a conductor insulated with commercial silk increases materially as the relative humidity increases, but the A. C. conductance of a similar conductor insulated with treated silk remains substantially unchanged.

The comparative stability of the electrical characteristics of insulating silk which has been produced in accordance with the invention renders such silk especially useful in insulating conductors for use in various types of electrical apparatus, particularly telephone apparatus. Since the currents employed in telephone circuits are alternative currents of audiofrequencies, and as the quality of transmission in these circuits is inversely proportional to the A. C. conductance between adjacent insulated conductors, it may be readily seen that transmission of good quality may be obtained by employing a relatively small quantity of insulating silk embodying the invention; whereas, if ordinary commercial silk is employed a comparatively large quantity of the silk must be used in order to obtain comparable results.

For example, it has been found that, due to the improved properties of insulating silk embodying the invention, one type of insulated conductor employed in telephone apparatus may be insulated with two servings of the improved silk, whereas three servings are usually required when the ordinary commercial silk is used, and in addition the conductors possess satisfactory insulating properties with equal or better transmission characteristics than heretofore obtained. In some cases, it is possible to substitute silk mixed with cheaper materials, such as cotton, for pure

silk and thereby obtain corresponding savings in cost. The silk may, of course, be employed in other fields where its improved characteristics make it desirable and the above described uses are given merely as examples to illustrate some of the desirable characteristics of the material.

What is claimed is:

1. The method of preparing insulating silk, which comprises repeatedly subjecting degummed silk to washing baths under pressure to remove ionizable impurities from the silk, at least one of the baths including a reagent capable of neutralizing impurities in the silk and the remaining baths comprising water.

2. The method of preparing insulating silk, which comprises repeatedly subjecting degummed silk to circulating washing baths under pressure to remove ionizable impurities from the silk, at least one of which baths includes a volatile reagent capable of neutralizing impurities in the silk and the remaining baths consisting of water.

3. The method of improving degummed insulating silk which comprises repeatedly washing the silk under pressure with a plurality of circulating washing baths to remove ionizable impurities from the silk, at least one of which baths contains a fatty organic acid and the remaining baths consisting of water.

4. The method of improving degummed insulating silk having an alkaline reaction, which comprises in circulating a dilute solution of acetic acid through a mass of silk under pressure, and then circulating water under pressure through the silk until substantially all ionizable materials are removed from the silk.

5. The method of preparing insulating silk, which consists in washing degummed silk under pressure with a plurality of circulating washing baths to remove the ionizable materials from the silk, at least one of which baths other than the last one includes a reagent capable of neutralizing impurities in the silk and the remaining baths consisting of water, and drying the silk to remove substantially all of the washing fluids therefrom.

6. As a new article of manufacture, electrical insulating material comprising silk which is substantially free from ionizable impurities.

7. The method of preparing insulating silk, which comprises repeatedly washing degummed silk under pressure with a plurality of circulating washing baths to remove ionizable impurities from the silk, at least one of which baths contains a weakly ionizable reagent capable of neutralizing impurities in the silk and the remaining baths consisting of water.

8. The method of improving degummed insulating silk, which comprises circulating through a mass of the silk a reagent capable of neutralizing reagents employed in producing the degummed silk, and then circulating water under pressure through the silk until substantially all ionizable substances are removed from the silk.

9. As a new article of manufacture, insulating silk which is substantially free from ionizable impurities and which has substantially constant electrical characteristics under varying conditions of service.

10. As a new article of manufacture, insulating silk characterized by being substantially free from ionizable impurities and by having a direct current resistance of at least 1000 kilomegohms per length of three-quarters of an inch at 87½% relative humidity and 100° F.

11. The process of improving commercial degummed insulating silk, which consists in cir-

culating a volatile reagent capable of neutralizing impurities in the silk under pressure through a mass of the silk, washing the silk with water to remove ionizable impurities therefrom, and heating the silk to remove the water and any remaining neutralizing reagent from the silk.

12. The process of improving commercial spun silk, which consists in circulating a hot solution of acetic acid through a mass of the silk under pressure, circulating water under pressure through the silk to remove ionizable impurities therefrom, and heating the silk to remove the water and any remaining acid from the silk.

13. The method of improving commercial degummed tram silk, which comprises circulating an ammonia solution through a mass of the silk, and washing the silk with water to remove the ionizable materials therefrom.

14. The method of improving commercial degummed tram silk, which consists in circulating an ammonia solution through a mass of the silk, washing the silk with water to remove the ionizable materials therefrom, and heating the silk to remove the water and any remaining ammonia from the silk.

15. The method of improving commercial degummed insulating silk, which comprises circulating a neutralizing solution through a mass of the silk, and circulating water through the silk to remove the ionizable materials therefrom.

16. The method of improving degummed insulating silk having an alkaline reaction, which comprises circulating a solution of a volatile acid through a mass of the silk, and washing the silk with water to remove the ionizable materials therefrom.

17. The method of improving degummed insulating silk having an alkaline reaction, which comprises circulating a solution of a weakly ionizable acid through a mass of the silk, and washing the silk with water to remove the ionizable materials therefrom.

18. The method of improving degummed insulating silk having an alkaline reaction, which comprises circulating through a mass of the silk a solution of a compound falling within the empirical class of formic, acetic, propionic, lactic, oxalic, tartaric, citric, carbonic and malic acids, and washing the silk with water to remove the ionizable materials therefrom.

19. The method of improving degummed insulating silk having an acid reaction, which comprises circulating a solution of a volatile basic reagent through a mass of the silk, and washing the silk with water to remove the ionizable materials therefrom.

20. The method of improving degummed insulating silk having an acid reaction, which comprises circulating a solution of a weakly ionizable basic reagent through a mass of the silk, and washing the silk with water to remove the ionizable materials therefrom.

21. The method of improving degummed insulating silk having an acid reaction, which comprises circulating a sodium phosphate solution through a mass of the silk, and washing the silk with water to remove the ionizable materials therefrom.

22. The method of improving the insulating properties of commercial spun silk, which comprises circulating a dilute solution of acetic acid maintained at a temperature of about 200° F. through a mass of the silk under pressure, and then washing the silk under pressure with water

maintained at a temperature of about 200° F. to remove the ionizable materials therefrom.

23. The method of improving degummed insulating silk which comprises neutralizing any unneutralized impurities in the silk and then circulating water through the silk until substantially all ionizable materials of any origin are removed therefrom.

24. The method of improving degummed insu-

lating silk having an alkaline reaction which comprises neutralizing the free alkali in the silk with a volatile organic acid and then circulating water through the silk until substantially all the products of the neutralizing reaction and other ionizable materials are removed from the silk.

MARK C. LEWIS.
ALGER M. LYNN.

10	85
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20	95
25	100
30	105
35	110
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45	120
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60	135
65	140
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75	150