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REGENERATED CELLULOSE DIELECTRIC MATERIAL
AND PROCESS OF PRODUCING THE SAME
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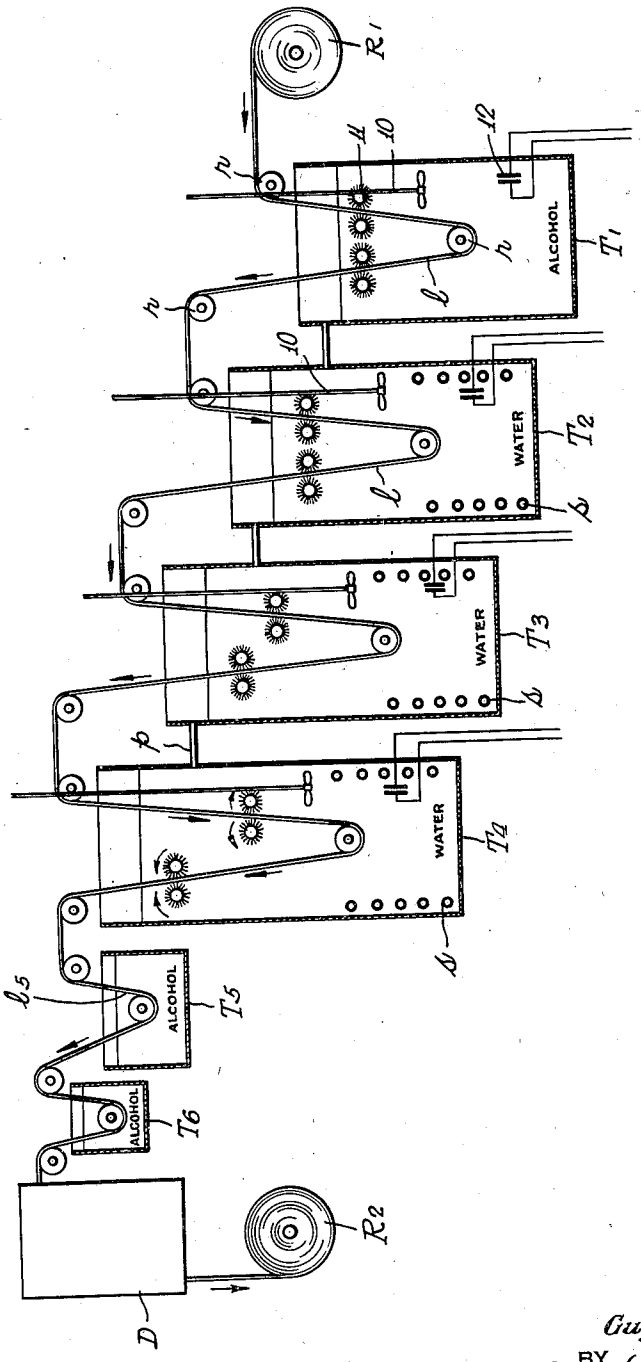

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REGENERATED CELLULOSE DIELECTRIC MATERIAL AND PROCESS OF PRODUCING THE SAME

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7 Claims. (Cl. 18—48)

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The present invention is concerned with electrical insulation or dielectrics and while of more general application is particularly applicable to electrostatic condensers.

Paper as is apparent by examination under a microscope is made up of numerous fibres meshed in a random felt-like fashion. The surface of the paper is rough with irregular hills and valleys due to the criss-crossing of the fibres. There are innumerable voids and crevices throughout the body of the paper due not only to the felting of the fibres but also to the essentially hollow tubular form of the individual fibres. This characteristic of paper renders the use thereof desirable as an interspacer or separator element in electrolytic condensers. The voids and crevices in the paper serve to absorb a substantial supply of electrolyte to permit the required conduction through the interspacer and also to admit of the desired self-healing of the dielectric film.

Though thus suitable for electrolytic condenser separators, even the finest grade of paper is far from ideal (for reasons now to be set forth) as the dielectric for electrostatic condensers. The air entrapped in the voids and crevices of such paper results in loss in capacitance, power losses, corona effects, increase in power factor and other disadvantages. Where such paper interspacer is used as the carrier or interspacer for insulating filler of wax or oil of various types and even where, by elaborate vacuum treatment, such filler is caused thoroughly to impregnate the voids in the paper to the exclusion of all air therein, the resultant condenser depending upon the dielectric constant of the filler, would have more or less capacitance than would a like condenser with the insulating filler alone, assuming of course that it were possible to use such filler in the absence of a paper carrier or interspacer. Moreover the use of such dielectric filler renders the manufacturing process unwieldy, increases the bulk of the condenser and involves many technical difficulties. From theoretical considerations as is also confirmed in practice, the breakdown voltage of a condenser with a combination of paper and filler dielectric is lower than were either of the constituent materials used alone. Furthermore the best grade of paper though elaborately macerated and washed is not pure cellulose, but includes foreign ingredients that are objectionable in a dielectric for condensers.

Regenerated alpha cellulose such as "Cellophane" is substantially pure cellulose, devoid of the impurities in paper. It is moreover characterized by the absence of foreign metallic or carbon particles and by its substantial homogeneity. The substantial absence of air voids in "Cellophane" as contrasted with paper is more or less indicated by the fact that the "Cellophane" has a specific gravity of 1.45 while that of the best grade of highly calendered paper rarely exceeds 1.1. Being in sheet form "Cellophane" would lend itself mechanically as readily as paper for incorporation into a condenser. Though cellulose as such is a good electrical insulator, even the best grades of the regenerated product as commercially made however nevertheless permit fairly ready conduction of electric current therethrough and this fault has heretofore rendered its use unfeasible for electrostatic condensers in which high insulation resistance is of paramount importance.

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The conductivity of "Cellophane" was found to be due to ionizing ingredients left in the "Cellophane" product in the manufacture thereof. The very density of the "Cellophane" that would theoretically render it useful as a dielectric, however interfered with the thorough purification or cleansing thereof to remove such ionizing ingredients. Even minute quantities of easily ionized materials in the "Cellophane" resulted in the conduction set forth and even traces of certain ingredients, such as chlorides also resulted in corrosion of electrodes.

It is an object of the present invention so to process the regenerated cellulose and especially the "Cellophane" of commerce, that it may serve as a useful dielectric of high insulation resistance, the physical structure and the high specific inductive capacity remaining unimpaired as a result of the treatment, so that the material becomes suitable even in the absence of filler as the dielectric of an electrostatic condenser of high quality.

The regenerated cellulose or "Cellophane" is treated according to the invention, with suitable solvent for sufficient length of time and under suitable conditions, to convert the regenerated cellulose or "Cellophane" into the dielectric desired. By such treatment, it is found that the residues of alkaline solvent, of acid bath, of glycerol softener and of any chlorides absorbed from the wash water used may be substantially completely removed from the commercial "Cellophane" sheet, without materially affecting the structure thereof. It is these solvent and acid residues dissolved in the glycerol residue that apparently rendered the "Cellophane" unserviceable for electrostatic condenser dielectric

use, because these residues ionize in the glycerol and accordingly establish a conductive path through the material.

While distilled water is a good solvent for various residual impurities, it does not, even when heated, penetrate the structure of the substantially continuous non-porous "Cellophane" sheet to degree sufficient to leach out these impurities. I have found, however, that where the "Cellophane" is given a first bath in a different solvent or mixture of solvents, desirably a mono-hydric alcohol solvent, preferably ethyl alcohol, the subsequent treatment with distilled water leads to effective removal of the impurities. Were the washing discontinued at this stage the residue of water on the film would tend to collect in small puddles thereon, with resultant wrinkling of the film in drying. To obviate this objection the treatment with distilled water is followed with a final bath of character such as to mix readily with the water remaining on the sheet, and to volatilize promptly in the subsequent drying operation. Such bath may be a suitable solvent, desirably including methyl alcohol, ethyl alcohol or a mixture thereof, which serves to remove further traces of the impurities and promotes more uniform drying, so that when the treated sheet has been dried, the operation is completed and the sheet has been converted into the desired smooth, substantially unwrinkled dielectric sheet.

In practice, the alcohol baths may be at room temperature, but the intervening baths of distilled water are desirably heated to between 175° F. and 190° F. and the drying is conducted in a small drying tower. If the bath is of temperature much above 190° F. the "Cellophane" film has a tendency to buckle.

After a predetermined period of washing in an originally fresh distilled water bath, the resistivity of the bath is measured with a pipette conductivity cell and by reference to any suitable standards that may be determined experimentally, the extent to which the processing of the "Cellophane" has progressed is intermittently determined until a point is reached where the product is satisfactory.

In the accompanying drawings the figure shows one simple diagrammatic illustrative installation of various possible types of equipment for treating regenerated cellulose according to the process above set forth.

Referring to the drawings, the untreated "Cellophane" or the like on reel R_1 at the right is passed through a succession of baths in a series of tanks T, thence to a dryer D and the dried, treated "Cellophane" is collected on a reel R_2. In general conformity with the foregoing description the first tank T_1 contains the alcohol solvent bath; tanks of sequence T_2, T_3 and T_4 each contain a water bath and the final alcohol bath, illustratively shown performed in two stages, is in tanks T_5 and T_6 immediately preceding the heater.

Preferably the alcohol tanks T_1, T_5 and T_6 have no heating equipment, since these baths are at room temperature, while tanks T_2, T_3 and T_4 for the water baths may have steam coils *s* therein with thermostatic control (not shown), to maintain the water warm, desirably, as previously noted, between 175° F. and 190° F.

The continuous web of "Cellophane" is passed over a sequence of rollers *r*, successively through the baths in loops *l*. Each tank desirably has a stirrer 10 therein to maintain the entire depth of

the bath uniform in temperature and composition. Desirably, at least the first few tanks are provided with rotary scrubbers 11 submerged in the bath to expedite the purification process. The brushes have desirably very soft bristles and revolve in direction opposed to that of film movement. Each tank desirably has mounted therein a conductivity cell 12, by which the specific resistivity of the contents is measured. Each cell may have associated therewith an electric signal (not shown) to warn of the need for replacing the bath with a fresh one when the resistivity reaches the minimum value permissible for the particular tank.

It will be understood that the baths at the beginning of the washing process, when the "Cellophane" sheet therefrom still has considerable impurities therein need not be as pure as those at the final stages of the process in which the film is nearly clean and only residual amounts of impurity remain to be washed therefrom. Accordingly it is desirable to arrange all but the final alcohol tanks in cascade as shown, so that tank T_4 slowly feeds through connecting pipe *p* into tank T_3 and similarly tank T_3 feeds into tank T_2 and finally tank T_2 into tank T_1. Thus each tank feeds its less contaminated water to the previous tank where a greater degree of contamination is feasible, triple distilled water being desirably fed continuously into tank T_4 at the rate at which the cascading operation is desired. Thus the percentage of contamination of each tank is continuously being reduced by the overflow from the less contaminated contents of the following tank.

Illustratively the contents of tank T_1 might be replaced when the reading on a given conductivity cell reaches 10 milli-amperes. For tank T_2 such reading might be 4 milli-amperes, for tank T_3 2 milli-amperes and for tank T_4 one milli-ampere. Illustratively the contents of alcohol tank T_5 might be replaced when its conductivity cell reading reaches say 2.5 milli-amperes and the final alcohol tank T_6 when such reading reaches one milli-ampere. The figures show merely illustrative relative figures as to the degrees of permissible contamination in the several baths. These figures are in no sense critical and their absolute value depends of course on the character of conductivity cell used.

Depending on the length of the loop *l* in each tank, the length of washing therein is controlled. For instance the loop l_5 submerged in the alcohol tank T_5 would ordinarily be of length considerably less than that in the water tanks that precede it.

It is desirable to provide an equipment sufficiently large to permit completion of the washing in one run through the machine, because such process is more expeditious and the strain upon the film is less than if the same were passed through a smaller machine several times.

Instead of processing the regenerated cellulose sheets of commerce, the purification of the product might be effected as the final step in the course of manufacture of the regenerated cellulose sheets according to conventional procedure. By way of example, the purified "Cellophane" might be prepared by the following procedure. The cellulose is first treated with a mixture of sodium hydroxide and carbon disulphide to convert it into cellulose xanthate. The cellulose xanthate is then dissolved in a bath of sodium hydroxide and worked to a thick syrupy liquid which is cast through an elongated orifice onto

a moving stream of dilute acid where it congeals into a sheet, the thickness of which is controlled by the orifice. As the sheet forms, it is washed with water to remove excess acid and alkali therefrom. Before the film structure has finally set a polyhydric alcohol, desirably glycerol which serves as a softener is applied to the forming sheet in percentage depending on the properties desired. Instead of stopping with the conventional procedure such as just set forth, the resultant film could then be washed, first desirably with water and then in ethyl alcohol, then in a succession of distilled water baths desirably as above noted between 175 and 190° F. and finally again in alcohol at room temperature, whereupon the product is dried in a drying tower and stored. It is within the scope of the invention to omit the plasticizing step from the conventional manufacture of "Cellophane."

The processed regenerated cellulose is characterized by high specific inductive capacity in the order of about 7.5 as compared to about 4.2 for paper. It has break down strength in the order of 1000 volts/mil and a condenser made therewith has low power factor of less than .33%.

The dielectric sheet material by reason of its smoothness and continuity may be used in electrostatic condensers in the absence of filler, especially where the foil is highly polished. By subjecting the condenser stack to suitable pressure, substantially any remnant of air that might otherwise be entrapped between foil and dielectric is expelled and the resultant condenser structure meets exacting specifications.

Since it has but a single dielectric material, the capacitance of the condenser is considerably higher than where two diverse dielectric materials are used in conjunction, such for instance as paper and oil. Moreover, the processed regenerated cellulose dielectric as above noted, has specific inductive capacity superior to that of paper or of most paper and oil combinations, leading to material reduction in bulk and cost of the condenser.

The use of filler materials in combination with the "Cellophane" dielectric sheet is not however excluded, but where such added filler is used, but a minute amount thereof is recommended, so that the capacitance of the condenser with such filler does not differ markedly from the value thereof were the same structure used in the absence of such filler. The use of mineral oil filler with the "Cellophane" dielectric sheet of the present invention results in only about five per cent change in the capacitance of the same condenser without filler. That is sharply to be contrasted with conventional condensers in which oil or wax of volume much greater than that of the paper must be used, and in which such filler is the controlling factor in determining the capacitance.

Among the fillers that could be used with the regenerated cellulose processed according to the present invention are of course any solid insulating materials such as paraffin, halowax, ceresin wax or the like, or any of the liquid dielectrics such as mineral oils, vegetable oils, chlorinated diphenyls, phosphates, ester compounds etc. If desired, the regenerated, purified cellulose sheet might be plasticized or varnished with any of the above compounds or materials, and this might be accomplished as a final step in the manufacture of the sheet in a continuous process.

It will be understood that the invention lends itself to incorporation in condenser structures whether of the wound, stacked or folded type.

It is also understood that the invention is in no sense limited to use as the dielectric of electrostatic condensers, but is applicable as the dielectric in other electric equipment as for instance, in lieu of impregnated paper wherever the latter is used in the electric arts as for instance for cable, wire and transformer coil insulation.

As many changes could be made in the above process, the resulting dielectric material, and many apparently widely different embodiments of this invention could be made without departing from the scope of the claims, it is intended that all matter contained in the above description shall be interpreted as illustrative and not in a limiting sense.

Having thus described my invention, what I claim as new and desire to secure by Letters Patent is:

1. As a dielectric for electrostatic condensers smooth, regenerated cellulose in sheet form substantially devoid of acid, alkali and softener reagents used in the regeneration of the cellulose and substantially devoid of filler said dielectric being the product resulting from the washing of the regenerated cellulose sheet, first with non-aqueous solvent, then with distilled water, and finally with a solvent that entrains residues of water in drying.

2. As a dielectric for electrostatic condensers smooth, regenerated cellulose in sheet form substantially devoid of acid, alkali and softener reagents used in the regeneration of the cellulose and having one or more insulating filler ingredients plasticised therewith said filler ingredient comprising but a minute proportion by weight of the dielectric sheet said dielectric being the product resulting from the washing of the regenerated cellulose sheet, first with non-aqueous solvent, then with distilled water, and finally with a solvent that entrains residues of water in drying.

3. As a dielectric for electrostatic condensers, smooth regenerated cellulose in sheet form, substantially devoid of acids, alkali and softener reagents used in the regeneration of the cellulose, and having plasticized therewith a material that has dielectric properties, said dielectric sheet being the product resulting from the washing of the regenerated cellulose sheet, first with non-aqueous solvent, then with distilled water, and before drying, washing with solvent that entrains residues of the previous washing operations, and finally applying the dielectric plasticizer.

4. The process of treating commercial "Cellophane" sheets that are substantially free of waterproof coating and of air voids and contain residues of acid bath, alkaline solvent and glycerol softener to convert the same into a dielectric for electrostatic condensers, which consists in passing the same successively through a bath of mono-hydric alcohol miscible with water, through a series of distilled water baths and finally through a second bath of mono-hydric alcohol miscible with water and then drying the sheet, with resultant substantial elimination of the residues of the acid bath, alkaline solvent, and the glycerol softener otherwise present in the "Cellophane."

5. The process as claimed in claim 4 in which the alcohol baths are at substantially room temperature and the distilled water baths are heated.

6. The process as claimed in claim 4 in which the alcohol baths are at substantially room tem-

perature and the distilled water baths are heated to a temperature not exceeding 190° F.

7. The process as claimed in claim 4 in which the alcohol baths are at substantially room temperature and the distilled water baths are at a temperature of 175 to 190° F.

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