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PROCESS FOR IMPREGNATING PAPER WHILE PARTIALLY DRY WITH A QUATERNIZED RESIN POLYELECTROLYTE AND A CLAY COATING Trygve C. Lucken, Milford, N.J., assignor to Riegel Paper Corporation, New York, N.Y., a corporation of Delaware

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The present invention relates to conductive papers and conductive paper making, and, more particularly, to a new and improved conductive paper especially well suited for use as a backing layer in electrostatic recording tive or photoconductive materials and to an improved method of making such paper.

In the formation of an image upon an electrostatic recording element, it has been found desirable for an electrostatic charge to be efficiently conducted from the 20 electrosensitive or photoconductive image forming layer. Accordingly, in the production of such recording elements, a conductive base sheet has been used in conjunction with an active surface coating which, typically, may include zinc oxide as a primary constituent. The de- 25 velopment and improvement of such conductive base sheets has therefore been a necessary adjunct to the recently developed and improved electrostatic printing methods and machines which have gained widespread ac-30 ceptance.

According to an important objective of the present invention, a new and improved commercially acceptable, conductive paper is provided having desirable and advantageous physical and electrical properties and which 35 may be produced on a practical and economical basis. More specifically, it is an object of the invention to provide a base sheet having the requisite conductivity to be employed in an electrostatic reproduction process and also having the requisite compatibility with and a high receptivity of commonly employed electrosensitive or 40 photoconductive coatings, especially those which include zinc oxide. In addition to providing a sheet which readily accepts the desired coatings, it is an object of the invention to provide a backing sheet which is non-deteriously affected by and offers substantial hold-out to sol- 45 vents commonly used in the application of the coatings, which solvents often include toluol or toluene.

A further object of the present invention is the provision of new and improved economical methods for manufacturing a conductive backing sheet having a high 50 conductivity, high acceptance of zinc oxide coatings. high toluol holdout, and desirable surface characteristics including smoothness, color, etc., which sheet is especially well adapted for commercial use in so-called electrostatic reproduction processes. 55

In accordance with the present invention, a conductive paper having the aforementioned desirable characteristics may be produced on a papermaking machine by highly refining or highly beating a papermaking stock, forming a wet web on a traveling screen from said stock. 60 reducing the moisture content of the web to a first predetermined level, applying to one side of the web through the use of rolling pressure a special electrolyte solution including a polyelectrolyte composition (and to particular advantage the commercially available Ionac XAX929. 65 to be described in more detail), reducing the moisture content of the web to a second predetermined level, applying a treating solution consisting of a special clay wash, thereafter completely drying and calendering the treated web. The clay wash advantageously may in- 70 clude, in addition to clay, such additives as binders, flow modifiers, optical whiteners, and lubricants, in predeter-

mined proportions to adjust the properties of the finished web to desired values in accordance with the ultimate specific electrostatic reproduction process in which it is to be employed. The electrolyte solution may similarly include predetermined proportions of additives to achieve certain of the requisite properties of the finished sheet. Variations in the proportions of the additives employed allow the physical properties of the sheets to be easily adjusted, as desired or found necessary, to fulfill opti-10 mally the varying requirements of different commercial electrostatic reproduction processes.

It should be appreciated that there are many requisite properties for a commercially acceptable conductive paper in addition to conductivity and that these properties elements of the type which are coated with electrosensi- 15 have been realized in the new and improved paper along with the desired conductivities. Thus, in accordance with an important aspect of the invention, there has been no significant sacrifice of desired color, electrosensitive coating receptivity, toluol holdout, or flexibility in the new paper for the sake of conductivity, as has often been necessary heretofore.

For a better understanding of the invention, reference should be made to the following detailed description and to the accompanying drawing, which is a simplified, schematic representation of the new method.

Manufacture of the new product, in accordance with one aspect of the invention, may be carried out with generally conventional papermaking equipment. Thus, with reference to the drawing, the equipment may comprise a traveling Fourdrinier wire 10, onto which a highly refined furnish (e.g. approximately 250-400 cc. Schopper Riegler) of cellulosic or like papermaking fibers 11 may be flowed from a head box 12 to form a highly refined web 16, a press section 13 just beyond the dry end of the wire 10, and a dryer section 14, typically including a large number of dryer drums 15.

The highly refined, wet paper web 16 leaving the wire 10 is quite weak and its strength is enhanced somewhat by the removal of some of its excess moisture by passing it through the opposing rollers of the wet press section 13. The still very moist and plastic web 16 is then passed through a small portion of the dryer system 14, for example, only eight dryer drums, to reduce further its moisture content. At this point in the process and in accordance with a preferred practice of the invention, the highly refined web has a special electrolyte solution 17 applied thereto under rolling pressure in the nip of a size press 18 which, because of its location in the system, will be referred to herein as a "plastic" press.

The electrolyte solution 17 is a water solution including, advantageously, from 8-30 percent of the polyelectrolyte composition. In accordance with one specific aspect of the invention, the polyelectrolyte composition is a water soluble quaternized condensation product of epichlorohydrin and dimethylaminopropylamine, commercially available under the trade designation Ionac XAX929 from the Ionac Chemical Company. A more detailed description of Ionac XAX929 may be obtained from the United States patent application No. 353,227 of Albert H. Greer for "Soluble Condensation Products of Epihalohydrins and Polyamines," filed on March 19, 1964. For convenience of description, the polyelectrolyte composition Ionac XAX929 and its equivalents may hereinafter be referred to as resin polyelectrolytes.

Certain additives may be included in various amounts in the solution as found desirable or necessary. Thus, to increase the viscosity of the polyelectrolyte solution 17 and to enhance the toluol holdout of the finished sheet, a film former such as carboxy-methyl cellulose should be added. When necessary or desirable to have a supercalendered finish on the conductive sheet, a suitable

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lubricant may be incorporated with the electrolyte solution for that purpose. Similarly, the color and coatability of the finished sheet may be enhanced by the inclusion of suitable pigments. In addition, the electrolyte solution 17 may also comprise an electrolyte "extender" such as 5 sodium chloride or sodium nitrate. For the optimum results and in accordance with one aspect of the invention, it has been found desirable to limit the total amount of additives in the solution to approximately 1 to 100 percent of the basic polyelectrolyte weight. 10

After being treated at the plastic press 18, the web has its moisture content significantly reduced by being passed through a major portion of the dryer section, for example, through a total of twenty-seven drums, at which point it is subjected to further treatment in a second size 15 press 19 which, because of its location in the system, will be referred to herein as a "starch" press. A clay wash 20, comprising titanium dioxide and binder (starch, alpha protein, etc.) in addition to clay, is applied to one or both sides of the web in the nip of the press 20 19. The wash 20 may also include such additives as flow modifiers, optical whiteners, lubricants, and the like.

Upon leaving the starch press 19, the web is coursed through the remaining dryer drums (e.g., eight drums in a 35 drum dryer section) whereafter it is subjected to 25 a machine calendering operation to impart surface smoothness and to reduce the web to its finished caliper. Alternatively to machine calendering, the web may be dampened and supercalendered using conventional techniques.

It has been determined, for its desired end use as a backing sheet for a subsequently applied electrosensitive coating for a commercially acceptable copy paper, that the amount of applied polyelectrolyte composition in the dry web should not exceed ten percent, with four to five 35 procedures and proportions specifically described herein percent being optimum for economically achieving the requisite properties. Similarly, limits on the amount of clay coating applied have been determined to be not in substantial excess of ten percent, dry basis, with four to five percent being typical of the percentages being 40 employed for commercially acceptable products.

While the preferred method outlined hereinabove has proved most acceptable, contemplated alternatives include the application of the electrolyte solution to one or both sides of the web in one step at the starch press, along 45 with the clay wash, etc. One specific example of these alternatives which has proven to be highly successful is the application of surface sizing agents, such as carboxy methyl cellulose and the like, to one side of the web at the plastic press followed by a one or two sided applica- 50 tion of resin polyelectrolyte at the starch press.

Conductivities of the new and improved paper can be easily and carefully controlled in accordance with the invention, by varying the percentage of conductive resin applied to the wet web. Thus, with an electrolyte solu- 55 tion comprising a resin polyelectrolyte such as Ionac XAX929, it has been found that with an approximate four percent (dry solids) pickup by the web, the finished sheet will have a surface ohmic resistance of 10³ to 10¹¹ ohms per square, as measured by a pair of one centimeter 60 wide electrodes spaced one centimeter apart, at relative humidities of 50 percent to 20 percent, respectively, which range of resistance is especially desirable for electrostatic reproduction processes of the so-called "Electrofax" type. Moreover, it has been determined that great control of 65 specific surface resistance may be obtained, since an increase or decrease of one to two percent from the preferred four percent pickup range will effect a corresponding change of one to two decades in ohmic resistance. Conductivities of papers treated with Ionac XAX929 may 70 range from 10⁵ to 10¹² ohms per square centimeter at relative humidities of from 90 percent to 20 percent, respectively.

In addition to the requisite conductivities, the finished

to the combinative effects of the highly beaten fibers with the on-machine wash coatings and other surface sizing materials or additives. These hold-out properties of the papers are not only important in the subsequent application of electrosensitive coatings to form electrostatic recording elements or copy paper, but are of additional importance when the copy paper is to be used in socalled "wet" electrostatic reproduction processes in which liquid toners having solvents in the nature of kerosene, mineral spirits, and the like are employed. As will be understood, the hold-out properties of the new and improved conductive paper will resist penetration of the toner solvents into the backing sheet of the copy paper, particularly from the back (uncoated) side.

The finished conductive paper of the invention advantageously has a basis weight of 30 to 55 pounds per ream (3,000 sq. ft.) for single copy paper, although it may be somewhat greater for special applications, e.g., 75 pounds per ream, for lithographic masters.

The present invention provides a method of making an improved, commercially acceptable conductive paper having especially desirable properties for use in electrostatic reproduction processes. Moreover, it provides a process by which a conductive paper can be produced efficiently and economically "on machine" and with readily available equipment. It should be appreciated that the new and improved product of the invention may have its electrical resistance carefully and easily controlled, possesses high toluol holdout for subsequent coat-30 ing, good flexibility, and is relatively inexpensive. Accordingly, it is especially well suited for use as a base paper or backing sheet for electrostatic recording elements.

Inasmuch as certain departures may be made from the without departing from the clear teachings of the disclosure, reference should be made to the following appended claims in determining the scope of the invention. I claim:

1. A method of manufacturing conductive backing paper for recording elements for use in wet development electrostatic reproduction processes and the like, comprising

- (a) highly refining a furnish of papermaking fibers to a substantially low value of freeness in the range of approximately 250 to 400 cc. Schopper Riegler;
- (b) forming on a paper machine a weak wet web from said furnish of highly refined fibers;
- (c) reducing the moisture content in said web after formation on the paper machine by a partial drying thereof:
- (d) impregnating at least one surface of said web with a solution of a water soluble quaternized condensation product of an epichlorohydrin and dimethylaminopropylamine in a manner to provide said web with approximately 4%, based upon the dry solids weight of said papermaking fibers, of said condensation product;
- (e) further reducing the moisture content of said impregnated web of highly refined fibers by additional drying thereof;
- (f) impregnating at least said one surface of said web with a clay coating not in substantial excess of 10%, based upon the dry solids weight of said papermaking fibers, while subjecting said web to rolling pressure, whereby the highly refined fibers and the clay coating combine to provide substantial holdout with subsequently applied electrostatic sensitive coatings at one of the surfaces of said web and to provide a substantial holdout of solvents used with electrostatic development toners at the other surface of said web; (g) substantially completely drying said impregnated

web of highly refined fibers.

2. A method of manufacturing conductive backing papers exhibit a highly satisfactory toluol hold-out due 75 paper for recording elements for use in wet development electrostatic reproduction processes and the like, comprising

- (a) highly refining a furnish of papermaking fibers to a substantially low value of freeness in the range of approximately 250 to 400 cc. Schopper Riegler;
- 5 (b) forming on a paper machine a weak wet web from said furnish of highly refined fibers;
- (c) reducing the moisture content in said web after formation on the paper machine by a partial drying thereof:
- 10 (d) impregnating at least one surface of said web with a solution of water soluble quaternized resin polyelectrolyte in a manner to provide said web with not substantially more than approximately 5%, based upon the dry solids weight of said papermaking fibers 15 of said resin polyelectrolyte;
- (e) further reducing the moisture content of said impregnated web of highly refined fibers by additional drying thereof;
- (f) impregnating at least said one surface of said web 20 with a clay coating not in substantial excess of 10% of the dry solids weight of said papermaking fibers, while subjecting said web to rolling pressure, whereby the highly refined fibers and the clay coating combine to provide substantial holdout of subsequently ap- 25 S. LEON BASHORE, Examiner. plied electrostatic sensitive coatings at one of the

surfaces of said web and to provide substantial holdout of solvents used with electrostatic development toners at the other surface of said web;

- (g) thereafter completely drying said impregnated web of highly refined fibers.
- 3. The process of claim 2 which includes the step of
- (a) calendering said substantially completely dried web.
- 4. The process of claim 2 which includes the step of
- (a) subjecting said weak wet web to wet pressing pressures after it leaves the paper machine and before it is partially dried.

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