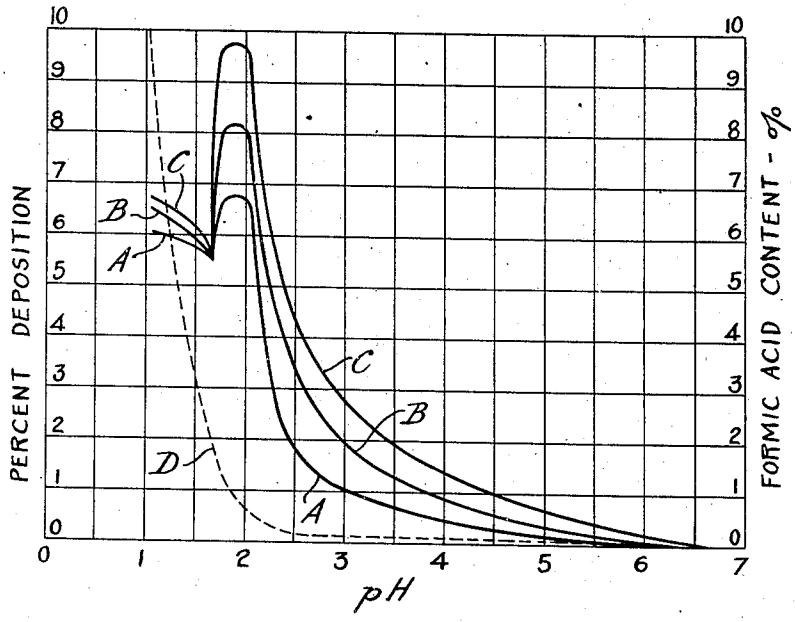
Sept. 19, 1939. H. A. YOUNG 2,173,243

PROCESS FOR TREATING FIBROUS MATERIALS

Filed Jan. 3, 1938 2 Sheets-Sheet 1

Fig. 1.



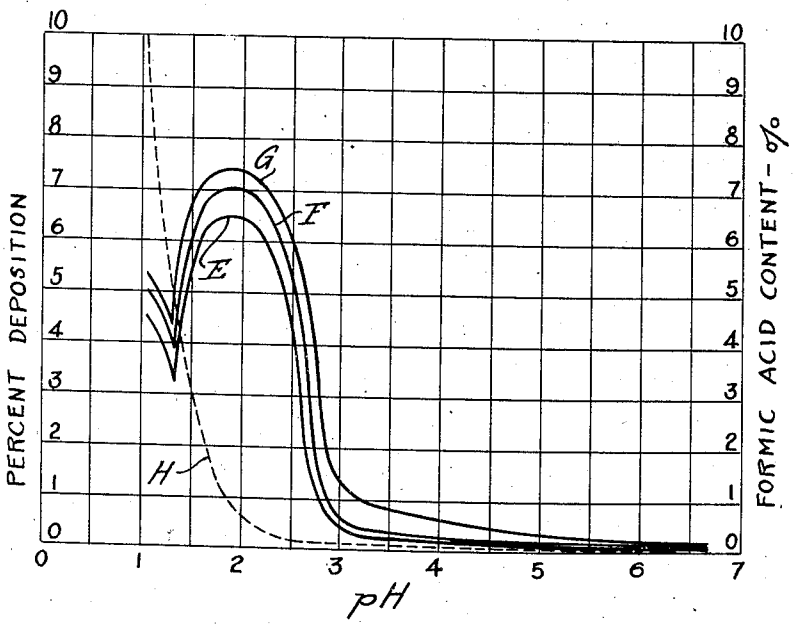


Fig. 2.

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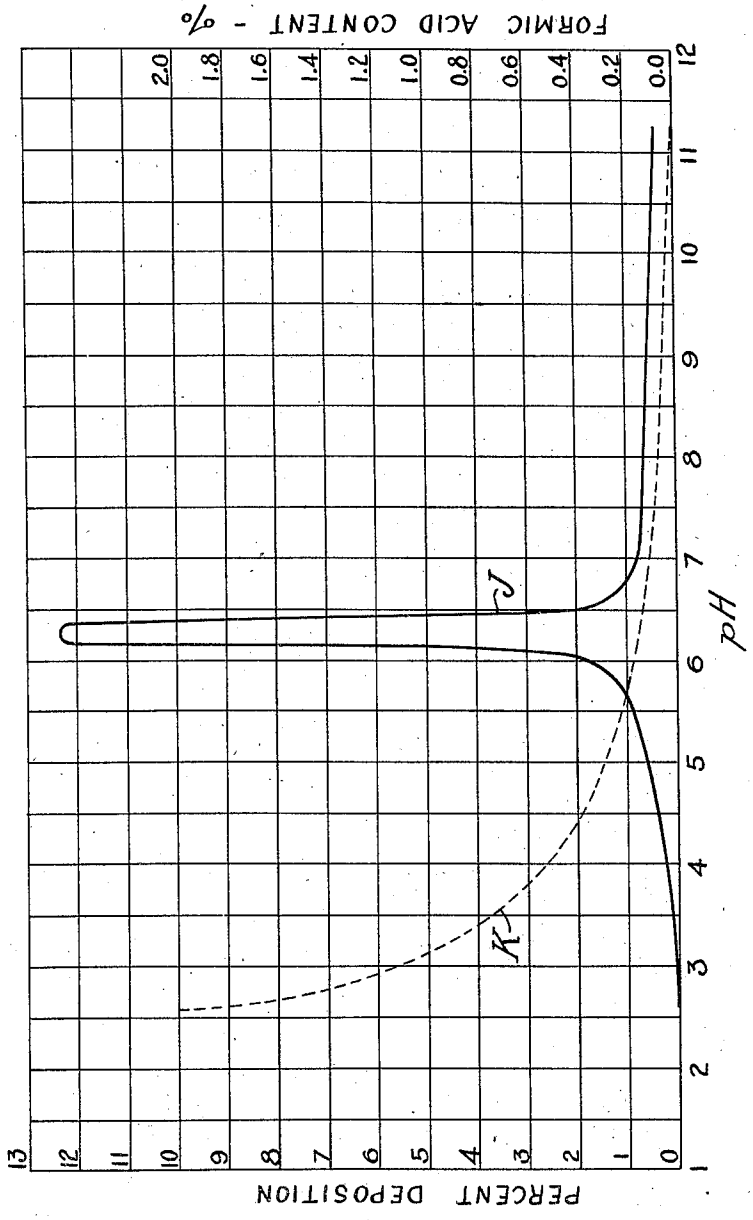

Fig. 3.

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UNITED STATES PATENT OFFICE

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PROCESS FOR TREATING FIBROUS MATERIALS

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Application January 3, 1938, Serial No. 183,092

10 Claims. (Cl. 91—68)

This invention relates to a process for treating fibrous material and more particularly to a process for incorporating treating materials for water-repelling, softening, strengthening, sizing, luster finishing or other finishing purposes in textile fabrics or other fibrous materials.

Various methods are known for incorporating treating substances in fibrous material from colloidal aqueous dispersions of such treating substances. Fibrous materials in the form of loose fibers or in the form of textile fabrics or other association of fibers, have been introduced into a bath containing the desired amount of dispersed colloidal substance and the fibrous material circulated with or through the bath or the bath circulated through the fibrous material until some or all of the colloidal substance has been incorporated in the fibrous material. In these cases, the dispersed material has been deposited on the fibrous material by a coagulative deposition which may be effected by heating the bath with which the fibrous material has been treated in cases where the dispersed particles of treating material can be coagulated by heat, or by the gradual addition of a coagulant or destabilizer to the bath, or by the previous treatment of the fibrous material with a coagulant for the dispersed particles of the bath so that the fibrous material carries an excess of such coagulant, or by the previous preparation of a bath containing dispersed particles having an electrical charge opposite in sign to that carried by the fibrous material so that the dispersed particles become coagulated on the fibrous material by virtue of the neutralization of the particle charges.

According to the present invention, treating material for water repelling, softening, strengthening, sizing, luster finishing or other finishing purposes is deposited on fibrous material by a non-coagulative deposition from an aqueous dispersion of the colloidal treating material which is in its isoelectric zone. Dispersions of colloidal substance generally owe their stability, that is, their inability to settle out or coagulate, to the presence of electrical charges carried by the particles of the dispersed phase. Charged colloidal particles will migrate in an electrical field or towards an oppositely charged body. There is, however, as is well known, a region of hydrogen ion activity where the positive and negative charges on the dispersed particles are equal to each other and where the charges on the dispersed particles are neutralized. This region of electrical neutrality of the dispersed particles is called the isoelectric range or zone and the midpoint of this range is termed the isoelectric point. Dispersed particles having zero charge in their isoelectric zone will generally coagulate unless previously protected by an adequate amount of suitable stabilizer.

In carrying out the present invention, fibrous material is treated with an aqueous dispersion of colloidal material which is at a pH in its isoelectric zone and which contains sufficient protective so that it is stable in the absence of the fibrous material, and the fibrous material is allowed to remain in contact with such an isoelectric dispersion, under normally non-coagulative conditions for the dispersion, that is, under conditions which would not produce coagulation of the dispersion itself were it not in contact with the fibrous material, until the desired amount of colloidal material has been deposited onto the fibers. Since the dispersion is stable and the conditions of treatment of the fibrous material with the dispersion would not coagulate the dispersion itself, and the dispersion is further isoelectric and hence contains no charged particles that will migrate to an electrically charged body, the deposition of the colloidal particles on the fibrous material is a non-coagulative deposition. Such a non-coagulative deposition or union of the dispersed colloidal particles and the fibrous material is termed in this specification "sorption" and the capacity of a given fibrous material to "sorb" a colloidal material from a given dispersion is the "sorptive capacity" of such fibrous material. The dispersion may remain in contact with the fibrous material at room or at elevated temperature providing, of course, that the dispersion contains sufficient protective so that coagulation would not take place in the absence of the fibrous material under the time and temperature conditions of the treatment of the fibrous material with the dispersion. If it is desired to deposit all the colloidal material in a treating bath on fibrous material with which the bath is associated in its isoelectric zone, the quantity of dispersed colloidal material in the treating bath should of course not be in excess of the sorptive capacity of the fibrous material for that particular colloidal material. The greater the amount of protective over the minimum necessary to stabilize the dispersion of treating material in the isoelectric zone in the absence of the fibrous material, the slower will be the rate of deposition of the colloidal particles from the dispersion onto the fibrous material. I have discovered, however, that in all cases the rate of sorption of the colloidal particles from the same treating bath onto the fibrous material is greater in the isoelectric zone than at a pH on either side of the isoelectric zone, regardless of whether the particles are given a positive or negative charge by so shifting the pH of the treating bath out of the isoelectric zone, and regardless of the sign of the charge on the fibrous material. This may readily be seen from the accompanying drawings in which:

Figure 1 is a chart showing the amount of lithopone deposited from a dispersion of lithopone

on woolen fabric in different periods of time at various pH's according to the procedure of Example 1;

Figure 2 shows similar curves for the deposition of lithopone from a dispersion on silk fabric according to Example 2; and

Figure 3 illustrates the amount of zinc oxide deposited from a dispersion on woolen fabric in a given time at various pH's according to Example 3.

Various types of fibrous materials, such as woven, knitted, punched or felted fabrics, yarns, rovings, cords, skeins, and webs, such as waterlaid felts from a paper making machine, or bats of fibers from a garnet or carding machine, or fiber slurries as in paper making, may be treated according to the present invention. Various fibers, as of wool, silk, cotton, viscose or acetate rayon, linen, cellulose and the like, or mixtures of the same, may be used, and these may be undyed, mordanted, dyed or otherwise treated so long as they are not in association with free coagulant which in the absence of the fiber would itself produce coagulation of the aqueous dispersion of colloidal material. Asbestos fibers can also be treated according to the present invention if the coagulants that are naturally associated with the fibers have been removed, or inactivated as by insolubilization.

Various materials which can be dispersed in an aqueous medium, with or without the addition of dispersing agents, may be used for the treatment of fibrous material according to the present invention. Such treating materials which can be colloidally dispersed in an aqueous media include, proteins, such as casein, gelatin, glue, albumen, serecin, and lecithin; carbohydrate gums, such as gum tragacanth, gum acacia, locust bean gum, karaya gum, and gum arabic; waxes, such as montan, carnauba and beeswax; oils such as castor oil, olive oil, mineral oil and neats-foot oil; dialkyl phthalates; fatty acids, such as stearic, oleic and palmitic acids; soaps; petrolatum; paraffin; asphalt; certain hydrogenated bituminous substances; certain halogenated aromatic compounds; certain halogen derivatives of hydrocarbons; water insoluble salts; natural resins, such as rosin, gum dammar and gum copal, and artificial resins such as phenol-formaldehyde resins, acrylic acid resins, glyptal resins, vinyl resins, and urea-formaldehyde resins. As specifically claimed in a copending application filed concurrently with this application, aqueous dispersions of rubber and rubber-like materials may also be used to treat fibrous materials according to the present invention.

The treating materials can be dispersed in water generally with the aid of dispersing agents by methods well known in the art, and the preparation of these dispersions, generally in concentrated form to be diluted in preparing the treating baths, but, if desired, originally in a concentration suitable for a treating bath, forms no part of the present invention. Those of the above listed treating materials which are also protectives may act as their own protectives when bringing the pH of the treating baths into the isoelectric zone, or they may be used to protect treating baths prepared from others of the above listed treating materials. A list of materials commonly used as dispersing agents is set forth below. Liquid water-insoluble treating materials may be dispersed or emulsified in water with the aid of a dispersing agent by mixing in a high-speed stirrer or homogenizing in a so-called colloid mill as well known in the art. Finely divided solid treating materials may readily be ball milled with water in the presence of a dispersing agent until the particles will remain in colloidal suspension. Treating materials that are plastic or which can be made plastic as by "breaking down" on a mill or "kneading" in a mixing device, such as a Werner and Pfleiderer mixer, may have the dispersing agent mixed into the plasticized material and water added until an inversion of phase takes place and the treating material becomes dispersed in the aqueous medium. In preparing a treating bath of an aqueous dispersion of colloidal treating material in its isoelectric zone for use according to the present invention, care should be taken to have sufficient protective present so that the isoelectric dispersion would not itself coagulate under the time and temperature conditions of the treatment with the fibrous material, if the dispersion were not in contact with the fibrous material. At the same time, too much protective should not be used or the rate of deposition of the treating material will be too slow due to the excess of protective, and the sorptive capacity of the fiber for the over-protected colloidal material will be reduced, the bath will not exhaust, and deposition of the desired amount of colloidal material onto the fibers will not satisfactorily take place. With a given treating material and a given fibrous material, it is a very simple matter empirically to adjust the amount of the desired protective so that in the isoelectric zone the otherwise stable bath will readily deposit the required amount of treating material on the fibrous material in a desired time.

A simple method may be used for determining the isoelectric range of the aqueous dispersion of colloidal material with a given protective, namely, by determining the migration or mobility of the dispersed phase of the dispersion at different pH's under the influence of an electric potential and noting the pH range of zero mobility or electrical neutrality. Other well known methods of determining the isoelectric point or zone are based on the fact that at the isoelectric point, the osmotic pressure, viscosity, and conductivity are at a minimum. In adjusting the pH of the aqueous dispersion of colloidal material in order to bring it into its isoelectric zone, the pH may be lowered where necessary by means of acids, such as formic, acetic, tartaric, phosphoric, hydrochloric and sulphuric acids, as well as various acid salts, or mixtures of the same, and the pH may be raised, where necessary, by the addition of ammonia or other alkaline substance.

Two general classes of protectives may be used in preparing the aqueous dispersion of colloidal material, those of a colloidal nature and those of a non-colloidal nature. Different protectives may be used for preparing the dispersion of the treating material and for stabilizing the treating bath made from such dispersion in the isoelectric zone. This will often be the case since many of the aqueous dispersions of treating materials are commercial products and it will be very likely that the protective which would be used to stabilize the treating liquid in the isoelectric zone according to the present invention would not be the same protective that was used to prepare the commercial dispersion. Proteins, gums, soaps, vegetable mucilages, and starches are examples of colloidal protectives that may be used. Various known non-colloidal protectives may be used, such as certain salts of carboxyl (R—COOM), sulphonic (R—SO_3M), sulfinic (R—SO_2M), or

phenolic (R—OM) groups or of ethereal sulphuric ($RO.SO_3M$) radicals, where the R may have a straight chain or ring structure and may contain substituent halogen, amino, nitro or hydroxyl groups. Other organic chemicals are well known protectives as certain benzene sulphonic acids, their homologues and substitution products, naphthoic acids, aliphatic-aromatic acids, derivatives of hydro-aromatic series of acids, phenyl-glycine and derivatives.

In the case of the colloidal type of protective for the dispersed particles, the protective becomes attached to the colloidal particles of treating material and the thus protected particles of treated material assume the charge and to some extent the properties and behavior of the colloidal protective agent and hence the isoelectric of the protective. It is believed that when a non-colloidal protective agent which is capable of ionizing is used to protect a dispersion of colloidal treating material, one of the ionized parts of the protective may fasten itself onto the colloidal material, and in so doing may shift the isoelectric range of the dispersed material somewhat in proportion to the amount of protective used. When a colloidal protective agent is used and the dispersion assumes the isoelectric of the colloidal protective, the deposition of the dispersed particles of treating material will be accompanied by a deposition of the colloidal protective and especially when an excess of coloidal protective over that merely necessary to stabilize the bath in the isoelectric zone is used. This may or may not be desirable. If it is objectionable for the colloidal protective to be deposited with the treating material, then a non-colloidal protective agent where possible should be used. Frequently both the colloidal and non-colloidal protectives may become modified by association with other ingredients of the treating bath, such as the colloidal treating material itself or alkaline or acidic materials or salts which may be added for this purpose or because it is desired to include them in the composition, with the result that the complex associated colloids in the dispersion exhibit an isoelectric point different from that of the protectives. However, it is always a simple matter to locate the isoelectric range in terms of hydrogen ion activity by means of cataphoretic tests, i. e., the conventional observations on colloid movement or migration under an impressed electrical force.

Various examples of the treatment of different fibrous materials with aqueous dispersions of various treating materials according to the present invention are set forth below, but these are merely exemplary of the invention and are not intended to be limitations thereon. The pH of the dispersions in these examples was lowered by means of formic acid because it is a volatile acid and has little effect upon the color and characteristics of fibrous materials, but other electrolytes as above described may be used satisfactorily for this purpose.

Example 1

In this case lithopone was deposited on a woolen fabric. A lithopone paste of the following composition was first prepared, the casein being solubilized in a part of the water by means of a small amount of sodium fluoride and borax in a known manner, and the thus solubilized casein added to the lithopone with the remainder of the water and the whole ball milled for over twelve hours.

	Parts by weight
Lithopone	30
Casein	3
Water	76

Various treating baths of the following composition containing amounts of formic acid ranging from 0 to 10% were made from the above lithopone paste:

	Parts by weight
Solids of the lithopone paste	.5
"Aquarex D"	.1
Formic acid	0 to 10
Water	To make 100

The dispersed lithopone particles were protected by the solubilized casein and the casein in turn protected against coagulation on the addition of the formic acid by the "Aquarex D". "Aquarex D" is a commercial product and is the mono sodium sulphate ester of one-half lauryl and one-half myristal alcohol. Among other commercial protectives or stabilizers which may successfully be used in preparing dispersions of colloidal material for treatment of fibrous materials according to the present invention may be mentioned "Mertanol" which is a naphthalene sulphonic acid derivative, "Gardinol WA" which is the sodium salt of sulphated lauryl alcohol, "Gardinol LS" which is the sodium salt of sulphated oleyl alcohol, "Igepon T" which is a fatty ester of a sulphonated aliphatic compound, "Arctic Syntex AX" which is an oleic acid ester of sulphated aliphatic compounds of the formula $R—COOXSO_3Na$, "Arctic Syntex T" which is an oleic acid ester of sulphated aliphatic compounds of the formula $R—CONHXSO_3Na$, "Emulphor O" which is a condensation product of ethylene oxide and higher alcohols, "Sulphanol" which is a sulphated fatty alcohol, and "Laurel Supersulphate" which is a hydrogenated oil which is sulphonated.

Pieces of woolen fabric were immersed in about 20 times their weight of various treating baths at 90° F. containing formic acid in amounts from 0 to 10% of the bath, and agitated in the baths for different lengths of time, namely, 5, 10 and 15 minutes, before removing from the baths, rinsing and drying. The percent deposition of lithopone on the fabrics under various pH conditions of the treating baths in the 5, 10 and 15 minute periods were determined, and these results are shown in the chart of Fig. 1. Throughout the specification, the percent deposition of the various treating materials on the fibrous material is calculated as percent gain based on the untreated fibrous material. The isoelectric zone of the treating bath extended from pH 1.7 to 2.1 and its midpoint at pH 1.9 was the isoelectric point as determined by cataphoretic tests. The amount of lithopone deposited on the woolen fabrics in the 5, 10 and 15 minute periods of treatment in the various treating baths which were in the pH range of 7 to 1.1, corresponding to formic acid contents of the bath ranging from 0 to 10%, are shown on the curves marked A, B and C respectively. The curve D shows the decrease in pH of the treating bath with increase of the formic acid content of the bath, and is typical of curves where acid content is plotted against pH. The amount of formic acid added to the bath is considered the formic acid content of the bath. From each of the curves A, B and C, it will be seen that the deposition of lithopone is con-

siderably greater in the isoelectric zone than on either side of it. The amount of lithopone deposited at the isoelectric point in 5 minutes was about 6.7%, in 10 minutes about 8.1% and in 15 minutes about 9.7%, which latter figure represents deposition of substantially all the lithopone in the bath. In this latter case there was substantially complete exhaustion of the bath.

Raising the pH from the isoelectric zone results in the assumption of negative charges by the dispersed lithopone particles and greatly reduces the amount of deposit of lithopone on the fabric in a given time. At pH's higher than 4 substantially no lithopone will deposit on the fabric from a dispersion by simple treatment of the fabric with the dispersion. Shifting the pH below the isoelectric range results in the assumption of positive charges by the dispersed particles and at first likewise reduces the amount of lithopone deposited on the fabric in a given time. This reduction in the amount of dispersed particles sorbed on the fabric in a given time continues with a decrease in pH from the isoelectric point 1.9 to a pH of about 1.65, corresponding to an acid content of the bath of about 2%. On further reduction of the pH below 1.65, as to 1.1 by increasing the acid concentration from 2% to 10%, the amount of lithopone deposited in a given time increases somewhat above the amount deposited at a pH of 1.65, although it does not in this case approach the amount of deposition obtained in the isoelectric zone. When the acid content of the bath is increased over 2%, that is, when the pH is lowered below 1.65, the bath becomes destabilized and flocks and coagula appear in the bath. When the fabric is immersed in such a bath below a pH of 1.65 visibly coarse particles of lithopone are deposited on the wool. It is also obvious from the change in the shape of the curve that a different mechanism is operating to deposit the lithopone at pH's below 1.65 than at pH's above 1.65. The undesirable deposition of visibly coarse particles of lithopone on the wool at pH's below 1.65 is believed to be the result of the chemical reaction of the excessive amounts of acid in these low pH ranges with the protectives on the lithopone particles, with the result that the protectives lose much of their protective power, and the consequent loss of stability permits the formation by coagulation of coarse particles of lithopone which then attach themselves to the fibers when the fabric is immersed in such a treating bath.

Example 2

In this case lithopone was deposited on silk fabric from treating baths of the compositions used in the treatment of the woolen fabric in Example 1. Pieces of silk fabric were immersed in about 20 times their weight of the various treating baths containing formic acid in amounts from 0 to 10% of the bath at 90° F. and agitated in the baths for similar lengths of time as in Example 1, namely, 5, 10 and 15 minutes, before removal from the bath, rinsing and drying. The percent lithopone deposited on the fabrics under various pH conditions of the treating baths in the 5, 10 and 15 minute periods were determined, and these results are shown in the chart of Fig. 2. As may be seen from Fig. 2, the isoelectric zone of the treating bath was, as to be expected, the same as in Example 1 and extended from a pH of 1.7 to 2.1 with the isoelectric point at a pH of 1.9. The amount of lithopone deposited on the silk fabrics in the 5, 10 and 15 minute periods of treatment in the various treating baths in the pH range of 7 to 1.1, which corresponds to the range of formic acid content of the baths of 0 to 10% are shown on the curves marked E, F and G respectively. The curve marked H shows the change in pH of the treating bath with the formic acid content of the bath and is the same as the corresponding curve D of Fig. 1. A comparison of the charts of Figs. 1 and 2 shows that the woolen fabrics of Example 1 had a greater capacity for the dispersed lithopone particles than the silk fabrics of Example 2. The amount of lithopone deposited on the silk at the isoelectric point in 5 minutes is about 6.5%, in 10 minutes about 7%, and in 15 minutes about 7.4%. Due to the lower rate of deposition of the lithopone particles on silk fabric than on woolen fabric, the rate of change in deposition with changes in pH is less with deposition on silk than on wool, and hence curves E, F and G display wider optima than than curves A, B and C. The maximum deposition, however, in every case is in the isoelectric zone. The deposition of lithopone clearly falls off in the case of silk fabrics as the pH is shifted in either direction from the isoelectric zone but not as rapidly as in the case of the woolen fabrics of Example 1. Raising the pH out of the isoelectric zone results in the assumption of negative charges by the dispersed lithopone particles and reduces the amount of deposited lithopone on the fabric in a given time. Shifting the pH below the isoelectric range results in the assumption of positive charges by the dispersed particles and at first likewise reduces the amount of lithopone deposited on the fabric at a given time. The reduction in the amount of dispersed particles sorbed on the fabric in a given time continues with a decrease in pH to a pH of about 1.35, corresponding to an acid content of the bath of about 5%. On further reduction of the pH from 1.35 to 1.1, by increasing the acid concentration from 5% to 10%, the amount of lithopone deposited in a given time increases over the amount deposited at a pH of 1.35. This is similar to the change which takes place in the deposition of the lithopone on woolen fabrics as shown in Example 1, except that in case of woolen fabrics, the change in the shape of the deposition curve takes place at a pH of 1.65 rather than at a pH of 1.35 as in the case of silk fabrics.

Example 3

In this case zinc oxide was deposited on a woolen fabric from a dispersion of zinc oxide protected by a colloidal protective, namely, sodium caseinate and sodium oleate. A protected colloidal zinc oxide paste of the following composition was first prepared by ball milling for over twelve hours:

	Parts by weight
Zinc oxide	1,000
Sodium oleate	150
Casein	25
Sodium hydroxide	1.25
Water	1,760

Various treating baths of the following composition containing amounts of formic acid ranging from 0 to 2% were made from the above zinc oxide paste:

	Parts by weight
Solids of zinc oxide paste	.5
"Aquarex D"	.1
Formic acid	0 to 2
Water	To make 100

Samples of woolen fabric were immersed in about twenty times their weight of the various treating baths at 90° F. containing formic acid in amounts from 0 to 2% of the bath, and agitated in the baths for 15 minutes before removal from the baths, rinsing and drying. The percent zinc oxide deposited on the fabrics under the various pH conditions of the treating baths were determined, and these results are shown in the chart of Fig. 3. The isoelectric zone of the treating bath extended from 6.20 to 6.35 and its midpoint at pH 6.28 was the isoelectric point as determined by cataphoretic tests. The amount of zinc oxide deposited on the fabric in the various treating baths which were in the pH range of 11.25 to 2.6, corresponding to formic acid content of the baths ranging from 0 to 2%, are shown on the curve marked J. The curve K shows the decrease in pH of the treating bath with increase of the formic acid content of the bath. The curve J illustrates the very rapid decrease in deposition of zinc oxide as the pH of the bath is shifted away from the very sharply defined isoelectric zone. The curve J clearly demonstrates again that sorptive deposition is a maximum in the isoelectric zone and falls off as the pH is shifted away from the isoelectric zone in either direction. As may be seen from curve J, the deposition of zinc oxide in 15 minutes at 90° F. at a pH of 11.2 where no acid had been added was only .4%. In order to bring the treating bath into the isoelectric zone, it was necessary to add only .05% formic acid, whereupon in 15 minutes at 90° F., 12.3% of zinc oxide, which was substantially all the zinc oxide in the bath, deposited on the fabric. The "Aquarex D" in the treating bath formulae was used to prevent coagulation of the sodium caseinate and sodium oleate protective on acidification of the various treating baths made from the zinc oxide paste on addition of the formic acid.

Curves similar to those of Figs. 1 to 3 may be obtained for the deposition of various other colloidal treating materials on various types of fibrous materials and such curves will all show that the sorptive deposition of colloidal material is a maximum when the treating bath is in the isoelectric zone. In the remaining examples, however, it will be sufficient to illustrate the deposition of various colloidal treating materials upon various fibrous materials at a single pH in the isoelectric zone of the treating bath, and in some cases to compare such isoelectric deposition with a deposition under similar conditions at a pH outside the isoelectric range.

Example 4

In this case, the lithopone paste of the formula shown in Example 1 containing 30 parts by weight lithopone, 3 parts solubilized casein, and 76 parts water, was made into an approximately 10% treating bath which was used to treat a silk fabric. This is a treating bath of much higher concentration than those used in the previous examples and was used in a padding procedure. The treating bath had the following composition:

	Parts by weight
Solids of lithopone paste	10
"Aquarex D"	1
Formic acid	1
Water	88.6

The above treating bath was padded onto a silk fabric by passing the fabric through the above treating bath and squeezing through rollers so that the fabric contained about 100% of its weight of the treating bath, that is, a weight of liquid bath in the fabric about equal to the weight of the fabric, or a so-called "bath-to-goods" ratio of 1:1. The pH of the treating composition which was in the isoelectric zone was 1.90, as in Examples 1 and 2. The fabric with the thus absorbed bath was allowed to rest at room temperature for 20 minutes, in which time substantially all the dispersed material deposited on the fabric, as shown by a clear rinse. After rinsing and drying, the fabric showed a gain in weight of 11.6%.

Example 5

In this case woolen fabric was treated with a dispersion of casein. The casein was first solubilized by means of a small amount of sodium fluoride and borax in water in a manner well known in the art, and the thus solubilized casein was made into a colloidal treating bath having the following composition:

	Parts by weight
Casein	.5
"Aquarex D"	.2
Formic acid	3.8
Water	95.5

The pH of this bath which was in the isoelectric zone was 2.1. The woolen fabric was immersed in about 20 times its weight of this bath and agitated for 10 minutes at 90° F. and then rinsed and dried. It showed a gain in weight of 7.6% casein. The treating bath without the formic acid, and hence not in its isoelectric zone, with a similar 10 minute immersion at 90° F., and rinsing and drying, showed only a 1.6% pick-up of casein. The "Aquarex D" was used to protect the casein against coagulation in the isoelectric zone.

Example 6

A commercial wax known as "Sunproof" wax was deposited from an emulsion onto wool fabric. The Sunproof wax dispersion was made by emulsifying 88 parts of Sunproof wax in 300 parts of water by means of 9 parts of oleic acid and 3 parts of triethanolamine as follows: The Sunproof wax was melted and the oleic acid added to it and the whole heated to 75° C. This was added to the triethanolamine dissolved in 100 parts of water at 75° C. in a thin stream under a high speed stirrer and the stirring continued for 10 minutes after the wax and oleic acid had been added. The stirring was continued while 200 parts of cold water was added in a thin stream, and this was stirred for 30 minutes after all the water had been added.

The treating bath was prepared from the above emulsion by diluting and acidifying in the presence of a stabilizer and had the following composition:

	Parts by weight
Solids of the above dispersion	.5
"Aquarex D"	.1
Formic acid	5.
Water	94.4

The pH of the bath which was in the isoelectric zone was 1.66. The woolen fabric was immersed in about 20 times its weight of the treating bath and agitated for 10 minutes at 90° F., and the fabric was then removed leaving a slightly hazy bath. After rinsing and drying the fabric showed a gain in weight of 10.5%. The gain in weight of fabric treated in the same manner but without the addition of the formic acid to bring the treating bath in its isoelectric zone was only 5.6% under the same treating conditions.

Example 7

Woolen fabric was treated with an emulsion of "Nujol" which is a white mineral oil. 93 parts of "Nujol" were emulsified in 100 parts of water by means of 5 parts of oleic acid and 2 parts of triethanolamine, and a treating bath of the following composition was made up from this emulsion.

	Parts by weight
Solids of "Nujol" emulsion	.5
"Aquarex D"	.05
Formic acid	5.
Water	94.45

The pH of the above emulsion which was isoelectric was 1.68. After immersion in about 20 times its weight of the treating bath and agitating for 15 minutes at 70° F., rinsing and drying, the fabric showed a gain of 7.2%. A similar treating without the formic acid necessary to bring the bath into the isoelectric zone, showed only 3.5% gain.

In the next three examples, various fabrics were treated according to the present invention with a commercial dispersion of Bakelite known as "Bakelite Emulsion XW-9060", the composition of which was not known. A treating bath was made from the "Bakelite Emulsion XW-9060" having the following formula:

	Parts by weight
Solids of "Bakelite Emulsion XW-9060"	.5
"Aquarex D"	.1
Formic acid	18.
Water	81.4

The pH of this treating bath which was isoelectric was 1.14.

Example 8

Woolen fabric was immersed in about 20 times its weight of this treating bath and agitated for 10 minutes at 80° F., and after rinsing and drying showed a gain of 7.8%. A similar treatment of the woolen fabric but without the addition of the formic acid to bring it into the isoelectric zone, showed a gain of only 4.1%.

Example 9

Viscose rayon fabric was immersed in about 20 times its weight of the treating bath made from the "Bakelite Emulsion XW-9060", as shown above, and agitated for 15 minutes at 80° F., and the bath completely exhausted. The gain in weight of the fabric after rinsing and drying was 12.2%.

Example 10

So-called "Immunized cotton" fabric was immersed in about 20 times its weight of the treating bath made from the "Bakelite Emulsion XW-9060" above and agitated for 15 minutes at 80° F. The bath again exhausted. After rinsing and drying, the fabric showed a gain of 10%.

In the next three examples various fabrics were treated according to the present invention with dispersions of soap. In each of the following examples the treating bath had the following composition:

	Parts by weight
Potassium oleate	.5
"Aquarex D"	.1
Formic acid	15.
Water	84.4

The pH of this bath which was in the isoelectric zone was 1.38.

Example 11

Woolen fabric immersed in about 20 times its weight of the above treating bath showed a gain of 9.4% after agitating for 10 minutes at 85° F., rinsing and drying. Without acidification and consequent reduction of the pH to the isoelectric, in 10 minutes immersion at 85° F., rinsing and drying as above, the same fabric showed a gain of only .23%.

Example 12

Silk fabric immersed in about 20 times its weight of the above potassium oleate treating bath and agitated for 10 minutes at 80° F., rinsed and dried, showed a 13.1% gain. The treating bath in this case was exhausted.

Example 13

Tanned viscose rayon fabric immersed in about 20 times its weight of the above dispersion and agitated for 10 minutes at 80° F., rinsed and dried, showed a gain of 9.1%. The bath in this case also exhausted.

The above examples clearly illustrate the present invention as applied to various treating materials for fabrics. Further examples showing the treatment of fibrous materials with isoelectric rubber dispersions will be found in the application filed concurrently with this application referred to above. It will be obvious to those skilled in the art that various other treating baths than those illustrated in the above examples may be developed for the treatment of various fibrous materials using the principles of the present invention. In view of the many changes and modifications that may be made without departing from the principles underlying the invention, reference should be made to the appended claims for an understanding of the scope of the invention.

This application is a continuation-in-part of application Serial No. 149,269, filed June 19, 1937.

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

1. The process of treating fibrous material which comprises associating a bath of an aqueous dispersion of colloidal treating material which is at a pH in approximately its isoelectric zone with fibrous material under normally noncoagulative conditions for the dispersion, said dispersion being stable in the absence of fibrous material but capable of depositing substantially all of its dispersed particles of treating material on fibrous material under normally non-coagulative conditions for the dispersion, and maintaining the fibrous material associated with said bath under normally non-coagulative conditions for the dispersion until the desired amount of treating material has been sorbed on the fibrous material.

2. The process of treating fibrous material which comprises associating a bath of an aqueous dispersion of colloidal treating material which is at a pH in approximately its isoelectric zone with fibrous material under normally non-coagulative conditions for the dispersion, said dispersion containing sufficient protective to stabilize it in the isoelectric zone in the absence of fibrous material but insufficient protective to prevent substantially all the dispersed particles of the dispersion from being capable of depositing on fibrous material under normally non-coagulative conditions for the dispersion, and maintaining the fibrous material associated with said bath

under normally non-coagulative conditions for the dispersion until the desired amount of treating material has been sorbed on the fibrous material.

3. The process of treating fibrous material which comprises associating a bath of an aqueous dispersion of colloidal treating material which is at a pH in approximately its isoelectric zone with fibrous material under normally non-coagulative conditions for the dispersion, said dispersion containing an acid and sufficient protective to stabilize it in the isoelectric zone in the absence of fibrous material but insufficient protective to prevent substantially all the dispersed particles of the dispersion from being capable of depositing on fibrous material under normally non-coagulative conditions for the dispersion, and maintaining the fibrous material associated with said bath under normally non-coagulative conditions for the dispersion until the desired amount of treating material has been deposited on the fibrous material.

4. The process of treating fibrous material which comprises associating a bath of an aqueous dispersion of colloidal treating material which is at a pH in approximately its isoelectric zone with fibrous material under normally non-coagulative conditions for the dispersion, said dispersion containing an acid and sufficient protective to stabilize it at elevated temperature in the isoelectric zone in the absence of fibrous material but insufficient protective to prevent substantially all the dispersed particles of the dispersion from being capable of depositing on fibrous material at elevated temperature under normally non-coagulative conditions for the dispersion, and maintaining said fibrous material associated with said bath at elevated temperature under normally non-coagulative conditions for the dispersion until the desired amount of treating material in the dispersion has been deposited on the fibrous material.

5. The process of treating fibrous material which comprises associating with fibrous material a bath of an aqueous dispersion of colloidal treating material which is at a pH in approximately its isoelectric zone, said bath containing sufficient protective so that it is stable in its isoelectric zone in the absence of the fibrous material but containing insufficient protective to prevent substantially all the dispersed particles in the bath from being capable of depositing on the fibrous material under normally non-coagulative conditions for said dispersion, and maintaining said fibrous material associated with said bath under normally non-coagulative conditions for said dispersion until at least part of the treating material has been sorbed on the fibrous material.

6. The process of treating fibrous material which comprises associating with fibrous material a bath of an aqueous dispersion of colloidal treating material which is at a pH in approximately its isoelectric zone, said bath containing sufficient protective so that it is stable in its isoelectric zone in the absence of the fibrous material but containing insufficient protective to prevent substantially all the dispersed particles in the bath from being capable of depositing on the fibrous material under normally non-coagulative conditions for said dispersion, and maintaining said fibrous material associated with said bath under normally non-coagulative conditions for said dispersion until substantially all the treating material in the dispersion has been sorbed on the fibrous material.

7. The process of treating fibrous material which comprises associating with fibrous material at room temperature a bath of an aqueous dispersion of colloidal treating material which is at a pH in approximately its isoelectric zone, said bath containing sufficient protective so that it is stable in its isoelectric zone in the absence of the fibrous material but containing insufficient protective to prevent substantially all the dispersed particles in the bath from being capable of depositing on the fibrous material in the absence of free coagulant on the fibrous material, and maintaining the bath associated with the fibrous material at room temperature and in the absence of free coagulant on the fibrous material until at least part of the treating material has been sorbed on the fibrous material.

8. The process of treating fibrous material which comprises associating with fibrous material at room temperature a bath of an aqueous dispersion of colloidal treating material which is at a pH in approximately its isoelectric zone, said bath containing sufficient protective so that it is stable in its isoelectric zone in the absence of the fibrous material but containing insufficient protective to prevent substantially all the dispersed particles in the bath from being capable of depositing on the fibrous material in the absence of free coagulant on the fibrous material, and maintaining the bath associated with the fibrous material at room temperature and in the absence of free coagulant on the fibrous material until substantially all the treating material in the dispersion has been sorbed on the fibrous material.

9. The process of treating fibrous material which comprises associating with fibrous material at elevated temperature a bath of an aqueous dispersion of colloidal treating material which is at a pH in approximately its isoelectric zone, said bath containing sufficient protective so that it is stable in its isoelectric zone in the absence of the fibrous material at elevated temperature but containing insufficient protective to prevent substantially all the dispersed particles in the bath from being capable of depositing on the fibrous material in the absence of free coagulant on the fibrous material, and maintaining the bath associated with the fibrous material at elevated temperature and in the absence of free coagulant on the fibrous material until at least part of the treating material has been sorbed on the fibrous material.

10. The process of treating fibrous material which comprises associating with fibrous material at elevated temperature a bath of an aqueous dispersion of colloidal treating material which is at a pH in approximately its isoelectric zone, said bath containing sufficient protective so that it is stable in its isoelectric zone in the absence of the fibrous material at elevated temperature but containing insufficient protective to prevent substantially all the dispersed particles in the bath from being capable of depositing on the fibrous material in the absence of free coagulant on the fibrous material, and maintaining the bath associated with the fibrous material at elevated temperature and in the absence of free coagulant on the fibrous material until substantially all the treating material in the dispersion has been sorbed on the fibrous material.

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