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METHOD OF CALENDERING MINERAL COATED PAPER

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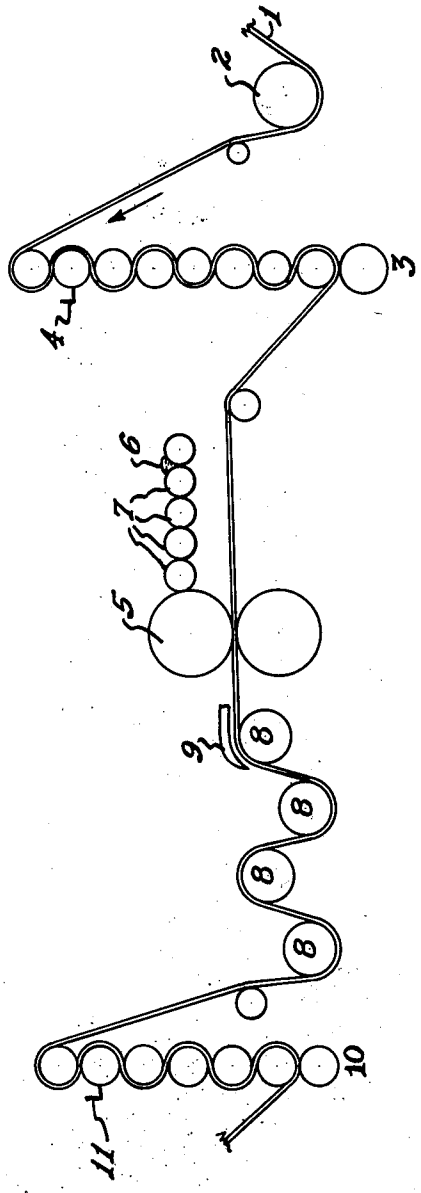

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METHOD OF CALENDERING MINERAL COATED PAPER

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Our invention relates to the problem of producing wet-type calendered finishes on paper or boxboard coated with adhesive-mineral coatings, and has for its objects the obtaining of superior finishes, improved physical properties and new effects, all as will be pointed out hereinafter.

In the processes of coating paper or board with finely divided mineral matter to enhance its surface characteristics and appearance, the coating mixture itself comprises a dispersion of the mineral matter in a water solution of adhesive. The mineral matter usually is clay with or without admixture of other finely divided mineral matters, pigments and the like. The adhesive can be casein, animal glue or other proteinaceous material, starch, dextrine and similar substances. The adhesive is prepared by dissolving it in water, using chemicals to assist as may be necessary; and the mineral matter, also in water, is dispersed in the adhesive solution by mechanical means. The dispersion may be assisted by the use of dispersing agents. Other materials may be included in the coating mixture to smooth it and give it body.

The physical properties of the coatings formed when these mixtures are applied to and dried on the surface of paper or board are dependent in part on the nature of the surface to which they are applied, upon the ratio of adhesive to mineral matter and also upon the intrinsic strength of the adhesive. If the ratio of adhesive to mineral matter is small, the porous character of the minerals prevails in the applied coating, whereas with increasing adhesive content the absorptive power of the minerals decreases. The adhesive not only binds the minerals to the paper surface in the dried coating but also reduces their porosity.

Depending upon the method used to apply the wet coating mixture to the paper or board surface and upon the coated results desired, the concentration of solids in the coating mixture may vary from a few per cent, say, around 10%, up to around 60% or 70%, more or less; this is well understood in the art.

In United States Patent No. 2,419,207 to Harry C. Fisher there are disclosed steps in a process of making coated board or paper wherein the freshly made web is treated with a plasticizable,

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film forming, water-borne substance, for example starch, or starch and polyvinyl alcohol, while on equipment such as a breaker stack of calenders, so that the surface is smoothed by plasticization and working. Then, immediately, a mineral coating is applied to it in an aqueous vehicle. The web is then dried and calendered in one or more stacks of finishing calender rolls. Where the coating is applied by printing, as with a roll, the solids concentration in the coating mixture is preferably high, say, around 50% and upwards; and a very thin mineral coating may be produced.

Where a coating mixture is imprinted upon the surface of paper or board as by a roll, a characteristic "patterned" surface of the coating results due to the manner in which the wet film of coating breaks away from the imprinting roll. The brush distribution of applied coating and smoothing rolls, used in some processes, has in part the purpose of breaking up patterns. If the coating mixture is low in viscosity, the pattern is minimized by a partial flowing together of the wet mixture. If the coating mixture is stiff, the applied film may be strongly patterned. In general, the heavier the thickness of the applied film the more prominent is its surface pattern. Absorptivity of the paper surface plays its part. Thus there are optimum conditions to meet to produce a coating of usable thickness on the surface of paper or board and with a minimum of pattern. The patterning difficulty is one reason for the desirability of a further after-treatment for the coated board.

The tendency of a mineral coating, especially one containing moderate amounts of adhesive, is to dry with a matt or dull surface. For some uses a harder, glossier and denser surface is desired. The coated board of the patent referred to above, when finished in a dry calender stack, has had a coating which has a matt or dull surface, comparatively, and which is excellent for many purposes. It can be printed with oil or varnish base inks to a dull or glossy film of dry ink, or to a glossy finish with overprint varnish depending upon the quality of coating mixture. It is, however, desirable in many instances to harden the coating to reduce the possibility of marking when passed over equipment for handling and processing the coated board, and to

produce a smoother and glossier surface which will lend itself better to the production of highly uniform matt printed surfaces or of a glossier and smoother printed film with gloss inks and varnishes.

The mineral coated boards of commerce ordinarily cannot be given a wet finish in a calender stack. The coated board of the patent just described, when coated with a non-waterproof starch-clay mixture has hitherto received only a dry finish by passing it through the nips of one or more finishing stacks of steel calender rolls, with the exception that steam, issuing at gentle pressure from a perforated pipe extending across the calender stack at an appropriate nip and blown onto the coating of the web just before it enters said nip, has been employed with an improvement of the finish and without picking or sticking difficulties in the stack. But the introduction of water into the calender box of a stack of finishing calenders through which the coated paper or board is passing soon results in a dissolving off of coating substances into the water, a picking off of the coating onto the rolls, and sticking together of the coating itself over the surface and back of the board.

The copending applications of Harry C. Fisher and Brainard E. Sooy, Serial No. 558,042, filed October 10, 1944, now abandoned, and Serial No. 738,722, filed April 1, 1947, now Patent No. 2,515,340, teach procedures by which the starch of a starch-clay coating on paper or board can be insolubilized and rendered waterproof. There is also a known process of coating webs in which the adhesive has to be intrinsically water resistant. In this process the coating mixture is applied and doctored onto the still damp web early in the heated dryer roll section of the paper machine, and the coated web is passed around and contacts the alternate remaining dryer rolls, passing thence through a calender stack. Starch does not perform well in this process because even though it be treated to become insoluble in water eventually, it remains soluble in the coating for some little time, and fouls up the heated dryer rolls of the board machine. The process employs casein or other proteinaceous adhesive material of a kind which is intrinsically water-resistant but can be dispersed for coating purposes; the dispersion reverts to the water-resistant condition as soon as the water of the coating mixture is reduced or removed, and complete insolubility is achieved by use of an agent like formaldehyde.

The coated web of the latter process, as well as the coated webs of the copending applications referred above, may be calendered in the presence of water without fouling the calender stack rolls if the coatings have first been dried and sufficiently insolubilized. However, if the coating, or rather its adhesive, is completely insoluble in water, water is ineffective on the calender stack and the wet calendering action does not produce the highly smooth, even glossy, surface frequently desired. Only to the extent that the surface of the coating is softened and dissolved, more or less, by the liquid used on the calender stack can the surface become plastic and workable into a smooth glossy condition by the action of the calender roll nips. This is especially true with insolubilized starch, and to a lesser extent with casein, which latter is more plastic by nature.

Aside from the difficulties and expense of employing adhesives which are or become substantially completely insoluble in water, since wet finishes could hitherto only be applied to mineral coated boards which had been thoroughly dried and had lost whatever plasticity the coating as such possessed, it has not hitherto been possible to apply a water finish to coated boards in such manner as to take advantage of the smoothing action of calender finishing as applied to a coating having resident plasticity. In the process of this invention we have found a way in which a water finishing operation may be correlated with coating operations in such manner as to obtain the ultimate in smoothness, removal of surface pattern, hardness and glossiness of coating, whether ultimately insoluble in water or not, and without the fouling of the stack of calender rolls.

Prior attempts to calender a mineral coated sheet in which starch or other binder is not completely water resistant, in the presence of a calender box full of water, have been disastrous either immediately or after a short time has elapsed. The coating mixture may be loosened from the surface of the sheet thus destroying the coated surface. Often stickiness of the adhesive in the coating develops at once, and this causes the coating to pick off the web, stick to the calender rolls and adhere to other parts of the coated web on the stack. Methods have been suggested in which a rubber doctor is placed against the surface of a steel calender roll between its nip and the lower calender box containing the water, whereby the doctor could control the amount of water entering the nip against the coated web surface. It was thought that a strict control of the amount of the applied water would reduce the sticking troubles. The success of this procedure depended upon the skill with which it was employed; it was infrequently successful; and in many instances the amount of water which could be tolerated at the nip of the rolls was not sufficient to enhance the smoothing effect and produce commercial improvement in the coating.

In a copending application for patent of Harry C. Fisher and Brainard E. Sooy, Serial No. 613,436, filed August 29, 1945, we taught the procedure of sealing the surface of an insolubilized mineral coating on board by coating it with a thin layer of starch, protein, sodium silicate, cellulose compound, etc., applied from water solution as from the water box of the finishing stack of board machine calenders. We did this to reduce the absorption of the vehicle of oil or varnish base printing inks into the mineral coated surface, which, itself, remains non-glossy, thus to enhance the gloss of the dried ink or varnish film printed onto the coated surface. The process operates only with coating that is substantially completely insolubilized.

We have now discovered, in one aspect of this invention, that a slippery, gelled solution of starch, preferably but not necessarily containing a small admixture of dispersed wax to enhance its slipperiness, can be used in the ordinary calender box of a stack of calender finishing rolls against the mineral coated surface of a web or board, where the adhesive of the mineral coating is a starch soluble in water, without producing stickiness of the coated surface and without harming the sheet in any way. Our procedure results in producing a smooth, glossy, sized, coated surface on the web, in which the pattern of the coating is largely reduced, the coating having been softened by the slippery

starch solution, then smoothed and compacted by the calender roll nips. The resulting sheet has greatly enhanced printing qualities, good appearance, and can be as bright and white as desired, or can be colored to desired hues. It will have other properties as dictated by the nature of the mineral coating itself. A mineral coated web so calender-treated on its surface tends naturally to be better suited for gloss ink printing and varnishing than for flat effects because of the continuous film of starch deposited on surface of the coating. However, it has been discovered also that flat ink printing is enhanced, too, if the starch calender solution is slightly acidified as by acid, ammonium chloride, potassium chrome alum or other suitable chemical. Penetrating and wetting agents such as those disclosed in United States Patent 2,419,206 to Harry C. Fisher, or others, are also suitable for use.

Soluble or dispersed dyestuffs or other coloring matters may be included in the slippery calender solution to color the surface of the coating or to modify it if already colored by dyestuffs or coloring matters therein, or thereunder as taught in U. S. Patent 2,370,344, "Imparting Color to Coated Paper Board," to Harry C. Fisher, or by various combinations of these procedures.

In a typical test of our procedure, where the introduction of water into the calender box of a stack of steel finishing calender rolls through which the coated web was passing resulted in a dissolving off of the coating substances into the water and a picking off and sticking together of the coating itself over the surface and back of the board, we substituted for the calender water a gelled solution of starch in increasingly greater concentrations. The picking and sticking effects persisted until the content of starch in the calender water solution was great enough to give the solution a slippery character. Then these difficulties ceased.

By gelled starch we mean any of those known chemically or otherwise treated starches or starch products capable of dissolving in cold or heated water to form a solution of high solids content accompanied by practicable viscosity. In the particular test referred to, a chlorinated corn starch was used, and sufficient slipperiness developed in the gelled solution at around 4½% of the starch by weight in a solution used at a temperature of about 140° F. on the calender stack. Starch solutions up to about 15% concentration by weight may be found useful. The concentration will vary with the particular starch used. With some starches an upper limit of 9% concentration may exist. Calender box solutions are used at temperatures ranging up to 160° F., more or less. In such a starch solution it is helpful but not essential to add a gallon, more or less, of wax dispersion of 50% wax content to each 100 gallons of the starch solution to enhance its non-adhesive quality. The type of starch used and its content by weight in the solution is critical only to the extent that it must make a slippery solution in water, with practicable viscosity at relatively high solids contents, and this is a property of the treated or modified starches such as those converted with enzymes, those chlorinated, oxidized, and otherwise chemically treated, all of which we may employ. Starch from corn develops much slipperiness and that from potato, tapioca, etc. is useful too. We find that starches treated to gel in cold water develop very high slipperiness, of great utility in our process. We are not limited as to the nature of the wax dispersion, if any, employed, but may use dispersions of paraffin, microcrystalline wax, carnauba or any of the natural waxes, depending, of course, upon the extent to which their nature might affect the desired color, brightness, or water-resistance, etc. of the coating. Small admixtures of the synthetic water dispersible wax-like substances of the class of polyhydric alcohol esters are useful.

Other substances which make slippery solutions and which are useful alone, or preferably in small amount as modifiers of our slippery starch solutions are alginic materials like Irish moss (Chondrus), the monogalactan mucilages as from locust bean gum or guar, and that natural substance, slippery elm (ulmin). We do not limit ourselves to these substances.

It will be evident from the above that the amount of starch in the calender water solution, as a measure of its slipperiness, is critical. The reason why a moderately concentrated starch solution will not produce sticking of the coated board, even when the adhesive of the mineral coating is not waterproof, while less or more highly concentrated starch solutions or plain water do produce sticking and picking is complex. We do not wish to be bound by theory but observe that by keeping the calender nip full of the proper starch solution its slipperiness prevails to the extent of preventing sticking. If the starch solution itself is initially too thick it already is sticky or tacky, while if the starch solution is too dilute, or if plain water is used, the slipperiness of the solution is absent, while the water of the solution acts to develop stickiness of the adhesive of the coating itself in the calender nip, by dissolving adhesive from the coating. Perhaps the rate of solubility of the adhesive of the mineral coating in a solution already containing a concentration of starch as solute enters into this phenomenon, since the starch solution of the proper slipperiness already has its capacity for solute fairly well satisfied, and perhaps this retards the dissolving away of the adhesive in the mineral coating itself, thus minimizing the development of stickiness in the coating proper in the nip of the rolls.

The fact remains that in our process the use of a gelled solution of starch containing approximately from 4½% up to 15% by weight of starch, and with or without admixture of other substances, as described, in the water box of a calender stack prevents the sticking and picking difficulties hitherto encountered with attempts to water calender mineral coated boards; and the ability to water finish a mineral coated board is no longer dependent on the solubility of the adhesive employed in the mineral coating. Our solution operates on mineral coated boards in which the adhesive is untreated water soluble starch as well as with coatings in which the adhesive is insolubilized starch or starch derivatives or insoluble casein or proteinaceous substances. Furthermore, the calendering action is not dependent upon any specific previous state of dryness, hardness or plasticity in the coating itself. Any of the hitherto known mineral coatings may be calendered in accordance with our process. A great advantage of our process is that the finishing action occurs while the coating at least retains a degree of plasticity which assists the smoothing action. Nevertheless, the coating as the result of the treatment has enhanced hardness and gloss as well as enhanced smoothness.

In another aspect of the invention we have discovered a method for calender-treating coated board, whether the coating is water-soluble or water-insoluble, on a finishing stack of calender rolls, to give coated surfaces varying as desired from merely slightly smoother and harder than the dry finished surface to smooth and glossy almost to a mirror-like degree and greatly harder, and without the deposition of a surface film of substance, like starch is deposited from the slippery starch solution and without fouling the calender stack.

Preliminary to our discovery, we reasoned that an enhanced smooth glossy surface of coating could result only if surface of the coating were softened and dissolved, as by plain water, at the calender nip and then re-smoothed and compacted by it and the following calender nips. Further, it was reasoned that by substituting for the water on the calender stack, a liquid in which the coating is insoluble, e. g., methyl alcohol, no enhanced smoothing and glossing of the coated surface could result. Both premises were found to be true. We have now discovered that for any given coating some simple combination of liquids, two or more, containing a solvent and a non-solvent for adhesive of the coating, are usable on the calender stack to produce a smooth glossy finish of coated surface as desired, and without fouling the calender rolls or the coated surface itself. These liquids should be ultimately volatile to leave the calendered coated surface without residue or discoloration.

By way of example, when a solution made by mixing 60 parts water and 40 parts methyl alcohol by volume was used in a water box on the top nip of a finishing stack containing seven polished metal calender rolls through which a freshly coated, dried web of paper board was passing, the adhesive of the coating being water soluble, and the coating comprising starch and minerals, the velvety matt surface of coating ordinarily obtained was enhanced to become highly smooth and glossy, almost to a mirror-like degree. The calender rolls remained clean, and the coated surface did not stick to them, pick or become marred. The calendered surface was dried in a normal manner. In the use of a solution of methyl alcohol and water, the water dissolves and softens the adhesive of the coating, while the alcohol in which the adhesive is insoluble acts as a diluent to retard and control the solvent action, we believe.

Results similar to those just described were obtained with water-methyl alcohol liquid on starch-mineral coated board in which the process of insolubilizing the starch with urea formaldehyde resin was in progress, as in the copending applications of Harry C. Fisher and Brainard E. Sooy, Serial Nos. 558,042 and 738,722 referred to above. Here the retarding action of alcohol or equivalent substance remains important but plays a lesser role because the proofed starch adhesive coating already has lower solubility in water and in consequence less alcohol or other non-solvent for the coatings adhesive may be needed.

While it is possible and useful to include a soluble substance in our alcohol-water calender solution and from it deposit a very thin film of said substance over the surface of the coating on the web while it is being calendered, it is not our primary desire to do so. We prefer to improve the smoothness and glossiness of surface of a coating without the imposition of anything to alter the printing characteristics of the particular starch-mineral coating mixture itself. Of course, we can color or dye our alcohol-water solution and use it to color the mineral coating itself by the procedures and combinations thereof as described in the discussion above of our slippery starch solution.

We do not confine ourselves to methyl alcohol in our alcohol-water type of calender solution, but do state that it has properties valuable for our use. It dissolves in water, is not sticky in solution, does not dissolve the starch-adhesive of the coating, is not too volatile but evaporates away from the calendered coated surface without leaving a residue of stain or odor thereon, and is not too expensive. Acetone can be used but is too volatile and is also expensive. Ethyl alcohol is expensive, but is satisfactory otherwise. On the other hand, glycerine does not dissolve the coating but does not evaporate off at all, leaves a stain on the coating and makes a sticky solution in water.

As an illustration of a procedure in accordance with our invention, reference is now made to the drawing wherein a web of paper or paperboard **1** is shown leaving the last roll **2** of the heated drying rolls of a paper machine and passing to a calender stack indicated generally at **3**. A plasticizing solution such as a solution of starch or starch and polyvinyl alcohol is applied to the surface of the web on the calender stack **3** as by means of one or more water boxes **4**; and the result of the action of the calender roll nips of the stack on the treated surface of the web is to plasticize it and prepare it for the reception of the mineral coating. The mineral coating is applied promptly while the surface of the web retains its plasticity, by means of an imprinting roll **5**, receiving the coating mixture from a supply **6** by means of metering and doctoring rolls **7**. The coated board then passes to a series of heated drying rolls **8**. Adjacent the first of these there may be if desired a hood **9** where hot air may be blown against the coated surface of the board to accelerate the initial drying before that surface contacts the second one of the drying rolls **8**.

The procedure thus far described is characteristic of the procedure of Patent 2,419,207 referred to above; but we are not restricted to it. Other procedures of imposing the coating may be employed; but we prefer to practice our finishing treatment upon freshly coated board and while the coating retains a resident plasticity. The adhesives employed in the coating mixture may be water-soluble, or water-insoluble, or may be treated to render them water-insoluble in accordance with the procedures of the copending applications referred to above or in other manners.

The freshly coated web of paper is next led to a finishing stack of polished steel calender rolls indicated generally at **10**, and the gelled solution of starch of the proper strength as described above is applied by means of one or more water boxes **11** the exact location of which may be varied as seems desirable with a given quality of coated web. We believe that the slippery character of the solution, acting as described above, is the factor which carries the softened coated surface of the web through the nip at which the solution is applied, without picking and sticking. The quantity of solution applied is small, and the applied substance either does not pass through a strongly adhesive stage, or dries so quickly as to be non-tacky at the next nip, which is preferably a dry nip. Where resident tackiness is

feared, it may be desirable to move water box 11 down the stack to decrease the number of calender nips following it. Absorption of the applied solution into the relatively porous mineral-adhesive coating beneath may be a factor. The treatment may be repeated at later nips.

The calendering is done at the pressures usually characteristic of finishing stack calendering, say, varying up to some 300 pounds per lineal inch of nip line of polished steel rolls, depending upon the calender stack and the calender nip concerned. The treatment with slippery starch solution or the like results in a sizing or sealing action as well as giving the effect of a water-calendering, whereas this sizing or sealing action does not occur where the alcohol-water type of liquid is used. The coated board acquires the desired hard, smooth, high gloss, water finish which has been described. We may use a plurality of finishing stacks if desired, and practice our treatment in connection with one or all of them, any stacks upon which our treatment is not applied serving to impart a further dry finish to the web.

When our alcohol-water solution is used instead of the slippery starch solution substantially the same procedure as described for it is followed. However, since the alcohol-water solution is more volatile than the slippery starch solution, and solutions in calender water boxes become hot from the hot web of paper or board, it must be handled in such manner that its alcohol-water ratio is preserved. This is done by balancing the flow of solution to the calender nip with its consumption in the nip so that no excess stands to be subject to differential evaporative effects; alternatively, alcohol lost by evaporation can be replaced.

We have found that the addition of other substances of quality and in quantity which do not in themselves affect the character of the slippery starch solution or increase its tackiness, does not in any way inhibit that action which permits us to apply a water finish to mineral coated webs. For example, the slippery starch solutions may contain wetting or penetrating agents, acidic or alkaline materials, soluble or dispersible dyestuffs or coloring matters, as well as small amounts of film-forming substances that promote gloss in printing, such as polyvinyl alcohol, soluble celluloses like carboxymethyl cellulose and hydroxyethyl cellulose, alginic substances, proteins, etc. and by the methods herein taught such solutions can be applied over the mineral coating in a calender finishing operation which forms a wet finish and employs the useful effect of the plasticization of the mineral coating itself. The application of the slippery starch solution in itself has a sizing and glossing action on an otherwise porous mineral coating as has been indicated. The film-forming agents mentioned above enhance the sealing of the pores of the coating in the finished coated board as treated in accordance with our process.

Again we may include other materials in the calender box solution. Where it is desired to enhance the waterproof characteristics of the coating, as in the case of coatings which must stand rough handling under moist conditions or in the case of coatings which are colored and must not smudge or offset if handled under conditions of dampness, we may include a small amount of resinous material in the finishing calender solution with or without any of the several substances mentioned above, in order further to waterproof and harden the surface coating.

Heretofore limited utility has been found in a colored mineral-starch coating which is not waterproof, since such a coating on paper or boxboard is undesirable because the color comes off on wet hands, and when the board itself becomes moist. In a board surfaced with mineral-starch, non-waterproof coating, with or without the underlying calender color treatment of United States Patent No. 2,370,344, an overlying calender application from an appropriate slippery starch solution may be employed to impart the moisture resistance or waterproofness to the mineral coating. Thus the slippery starch solution may contain a resinous material such as urea-formaldehyde or melamine resin sufficient in amount to give water resistance to the coating. A catalyst for the reaction may be carried by the mineral coating, the slippery starch solution, or even by the initial plasticizing solution, or any plurality of them. Dyes, pigments or other coloring matter may be contained in any of the applied coatings or any plurality of them.

Discovery has been made also that a finishing calender surface application of slippery starch solution or of the alcohol-water type of solution, as herein taught, but with the solution slightly acidified, over a starch-mineral coating, waterproof or not waterproof, promotes flat printing with oil or varnish base inks without sacrificing the high quality of gloss ink printing or over-varnishing where otherwise obtainable. A similar action appears to take place with paper or board which has not been coated with mineral-adhesive coatings; but a critical feature here is that acid treatments against a cellulosic surface may be undesirable, especially as the board or paper ages. An acid treatment applied over the coating as herein taught, however, does not adversely affect the quality of the board, while producing the printing effects which have just been described.

Other advantages are inherent in our process. Whereas starch-mineral coated surfaces, particularly those which are not waterproof, when given the heretofore possible dry calender finishes, marred and marked easily when rubbed or passed over mechanical parts of machinery, scored with the fingernail, and the like, the same coating, calender finished with the slippery starch solution or alcohol-water type of solution in accordance with our invention, with or without a content of other substances in such solutions, no longer can be marked easily and is more valuable for that reason. The coated surface has become harder, smoother and glossier, and in such condition performs better where two coated surfaces must rub and turn upon each other.

Modifications may be made in our invention without departing from the spirit thereof. Having thus described our invention in certain exemplary embodiments, what we claim as new and desire to secure by Letters Patent is:

1. A process of producing water finishes to attain smoothness on webs of paper or paperboard bearing a set but water-softenable coating comprising mineral substance and a water soluble adhesive which is starch, said process comprising passing said webs through a stack of calender rolls, and applying to the coated surface of webs while on said calender rolls an aqueous solution of gelled starch having a solids content substantially between 4½% and 15% by weight.

2. A process of producing water finishes to attain smoothness on webs of paper or paperboard bearing a set but water-softenable coating com-

prising mineral substance and a water soluble adhesive which is starch, said process comprising passing said webs through a stack of calender rolls, and applying to the coated surface of webs while on said calender rolls an aqueous solution of gelled starch having a solids content substantially between 4½% and 15% by weight, the said solution being applied by means of at least one water box on said calender rolls, at a temperature of substantially 140° F.

BRAINARD E. SOOY.
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