UNITED STATES PATENT OFFICE.

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PROCESS OF SIZING PAPER, &c.

SPECIFICATION forming part of Letters Patent No. 307,607, dated November 4, 1884.

Application filed February 6, 1884. (No specimens.) Patented in England May 3, 1883, No. 2,251.

To all whom it may concern:

Be it known that I, CHARLES WEYGANG, a subject of the Queen of Great Britain, residing at 7 Hermitage Villas, Childs Hill, in the county of Middlesex, England, manufacturer's agent, have invented a certain new and useful Improved Process for Sizing Paper, which is also applicable to the manufacture of material to be employed in the place of leather, parchment, and for many purposes for which wood, bone, horn, and stone are used, (for which I have obtained a patent in Great Britain, No. 2,251, bearing date May 3, 1883,) of which the following is a specification.

The principal object of this invention is to incorporate animal glue, starch, or flour with a fibrous pulp prepared by paper machinery by precipitating the glue, starch, or flour in the fibrous pulp and treating these materials in such a manner as to render them more or less impervious to water, while not materially injuring their valuable binding property, so that the fibers with which they are combined may be caused to unite, and by varying the proceedings as hereinafter described the invention may be applied to the sizing of ordinary paper or for the production of material applicable for some of the many purposes for which parchment, leather, wood, horn, bone, and stone, &c., are employed.

For the purpose of sizing with animal glue I dissolve the same in any convenient manner, and I add the solution to the fibrous pulp in the beating-engine or other suitable mixing apparatus. I then add one or more astringent metallic salts of nature appropriate to the kind of material to be produced. I then add a caustic alkali or an alkaline earth, or a salt of an alkali or an alkaline earth, taking care that the acid of the metallic salt is in excess, or that otherwise an acid is in excess, the object being to cause the formation of a nascent oxide or an acid or an interchange of elements in the glue in solution, whereby this is energetically attached and precipitated in the fibrous pulp.

Instead of first adding the acid metallic salt, I may in some cases add the alkaline salt first, and under certain circumstances I can mix the separate chemical agents together and add the mixture to the pulp and size in the beating-engine.

In some cases I mix one or more of the chemical agents employed with the size before I introduce the latter into the beating-engine, and sometimes I mix the glue in a precipitated form with the pulp in the beating-engine. Sometimes I mix the glue with certain chemical agents which do not precipitate it, but only thicken it, and I incorporate this thickened glue or size with the fibrous pulp while this is being formed into sheets.

Instead of employing metallic salts, I may for some purposes use non-metallic elements which have chemical affinity for the glue.

Having in the above given an outline of my invention, I now describe the various methods for carrying the same into effect.

For sizing ordinary paper the practical manufacturer will be able to proceed from the following details: For special purposes the fibers and the quality of the glue are chosen according to the nature of the material to be produced. Thus, for very strong purposes—such as for substitutes for leather—the strongest classes of fibers are employed, such as hemp, china-grass, &c. The fibrous materials are treated in the same manner as in paper-making, and in beating are kept of greater or shorter length, according to the material to be produced. Thus, if the material is to stand great tensile strain, the fibers are kept of as great a length as possible. The glue is also chosen according to the material to be produced. For instance, for material to take the place of leather I prefer to employ such as is made from skin and hide pieces. For almost all other purposes it is chosen according to the color required. I prefer to use the glue in the form of size, the quantity varying for the different purposes. For instance, for the purpose of making the material to take the place of leather, a good proportion is about three hundred-weight of size of average consistency to about one hundred-weight of fibrous pulp, reckoning the latter in dry weight. For material to be used in the place of horn, bone, and such like purposes I use from five to six hundred-weight of size to about one hundred-weight of fibrous pulp, dry weight. For most other purposes from one and one-half to two and one-half hundred-weight of size is usually employed. However, as such a variety of ma-

terial can be produced, I do not confine myself in any way to the proportions given above. When the glue has been dissolved, I incorporate it with the fibrous materials in the beating-engine after the fibrous material has been beaten to the requisite condition, and I then proceed to precipitate the glue by either of the following processes:

No. 1.—I add for every hundred-weight of size in the beating-engine about five pounds of an acid salt of zinc or alumina in a dry state or in solution. I prefer the sulphates of these metals, and I can use them, either separately or combined, in a proportion of about two pounds of the sulphate of zinc and about four pounds of the sulphate of alumina. I now add a salt of an alkali until no more precipitate will form, and I can employ a caustic alkali—such as soda hydrate—in a proportion of about one pound to every five pounds of metallic sulphate employed; but I prefer to use a sulphate or a sulphate of an alkali, and I find that the sulphate of ammonia in a proportion of about eight pounds to every hundred-weight of size gives the best result. I prefer to add the chemicals in a state of solution; but they may be added in a dry state, in which case I first reduce them to powder. Instead of mixing the metallic salts with the size in the beating-engine, the same results are obtained by mixing them with the size previous to its introduction into the beating-engine; also, the same results are obtained by mixing one of the metallic salts with the size and combining the other with the alkali. Thus I would add the sulphate of alumina with the size, and to a solution of sulphate of zinc I would add a caustic alkali until the precipitate formed is redissolved, and this mixed solution I use in the same manner as the alkaline salt alone.

No. 2.—I add a solution of ammonia chloride to the size in the beating-engine, or I incorporate the ammonia chloride with the size before putting the latter into the beating-engine. I employ a proportion of about seven pounds of ammonia chloride for every hundred-weight of size, and when the size and the salt are thoroughly mixed with the pulp I add about three pounds of bichromate of potash in solution for every hundred-weight of size. I then add a sulphate of an alkali in a dry state or in solution, and I prefer to employ the sulphate of ammonia, which is added until the size is all precipitated, the proportion being about seven pounds of sulphate of ammonia for every one hundred-weight of size. For the purpose of improving the precipitate I add a small quantity of an acid salt of alumina in solution, such as the sulphate of alumina in a proportion of about two pounds to every one hundred-weight of size.

Nos. 3 and 4.—Instead of employing bichromate of potash, as just described, I can employ with advantage sulphate of copper or iron alone or together, or either one combined with an acid salt of zinc or alumina. The proportion of the copper or iron salt is about five pounds of either for every one hundred-weight of size, or, if used together, about two and one-half pounds of each; or, if employed with zinc or alumina, the proportion of the copper or iron salt would be about four pounds and two pounds of the zinc or three pounds of the alumina salts. I precipitate with a salt of an alkali, as before mentioned, and I prefer to employ the sulphate of ammonia or soda hyposulphite, or these two combined—that is, I first add a solution of soda hyposulphite in about the same proportion as the acid metallic salts employed, and the ammonia sulphate in a proportion of about one-third of the soda hyposulphite employed.

No. 5.—I dissolve ammonia chloride with size, as before mentioned, and then sulphate of alumina in a proportion of about six pounds to every one hundred-weight of size, and when thoroughly incorporated with the pulp in the beating-engine I add for every one hundred-weight of size about two pounds of an alkaline chromate, such as chromate of potash in solution. For the purpose of improving the precipitate, I may finally add a small quantity of ammonia sulphate in a proportion of about two pounds for every hundred-weight of size.

No. 6.—Instead of employing ammonia chloride alone, as described, I sometimes combine it with calcium sulphate and hydrochloric acid. For this purpose I first dissolve the ammonia chloride in water, and then I add about six pounds of calcined calcium sulphate to every six pounds of ammonia chloride, and then I add about three pounds of hydrochloric acid. I allow the mixture to remain a few hours before using, stirring it from time to time, and I then employ it as I would the ammonia chloride alone, precipitating the glue in the same way as already described. In employing this mixture, however, I precipitate with very good results by using ammonia sulphate alone in a proportion of about twelve pounds for every one hundred-weight of size, without employing an astringent metallic salt. In all cases described above the ammonia chloride is principally added for the purpose of rendering the glue less brittle. Where hardness is no object—such as for material to be used in the place of horn, bone, wood, &c.—the ammonia chloride is omitted.

No. 7.—To a strong solution of hydrochloric acid I add a dilute solution of an alkaline silicate until the slightest signs of precipitation appear. Of this solution I add from one and one-half to two and one-half gallons to every four gallons of size contained in the beating-engine. I then add for every gallon of acid silica used about an equal quantity of soda hyposulphite in a saturated solution, and when thoroughly mixed I add a small proportion of ammonia sulphate, for the purpose of improving the precipitate, for this purpose a proportion of two to three pounds being sufficient.

No. 8.—Instead of employing an acid silicate,

I may, under certain circumstances, employ an alkaline silicate, for which purpose I prefer to proceed as follows: I mix with every hundred-weight of size from one-fourth to three-fourths hundred-weight of an ordinary solution of an alkaline silicate, such as silicate of soda. I incorporate the size and silica with the pulp in the beating-engine, and I now add a compound of chloride, by preference ammonia chloride, of which I add a solution to the matter in the beating-engine until no more precipitate forms. Instead of employing a salt of chlorine, I can use with very good results a small quantity of hydrochloric acid in a proportion of about one-eighth of the weight of the silicate employed, and I then add ammonia sulphate until no more precipitate will form.

No. 9.—I employ a salt of an alkaline earth, and I prefer to use the calcium chloride with hydrochloric acid in excess—that is, to a saturated solution of ordinary calcium chloride I add from one-half to an equal quantity in bulk of hydrochloric acid. Of this solution I add about one and one-half gallon to every four gallons of size contained in the beating-engine. I then add a saturated solution of soda hyposulphite, in a proportion of about two gallons to every one and one-half gallon of calcium chloride and acid employed, and when thoroughly mixed with the size and the pulp I add a small quantity of ammonia sulphate, for the purpose of improving the precipitate.

No. 10.—In cases where it is desired to make a very opaque material of a good color I employ a soluble salt of baryta or calcium in conjunction with a sulphate or a sulphite. For this purpose I add to every hundred-weight of size, either before or after its introduction into the beating-engine, from six to ten pounds of the soluble salt of the alkaline earth, and I prefer to employ for this purpose barium chloride, and after being thoroughly mixed with the size in the beating-engine I add, either in a dry state or in solution, a sulphate or a sulphite. I prefer to employ sulphate of alumina in a proportion of about four pounds to every six pounds of the salt of the alkaline earth employed, and, after being thoroughly mixed, I add a small quantity of a caustic alkali in solution—such as soda hydrate—in a proportion of about one-half pound to every six pounds of baryta chloride employed; or, instead of employing a caustic alkali, I can employ an alkaline sulphate, in which case I prefer to use the ammonia sulphate.

No. 11.—Instead of precipitating the glue in the beating-engine, I may precipitate it, but not with such good results, before it is incorporated with the fibrous pulp in the beating-engine by any of the above-named processes, or with bichromate of potash in conjunction with any other astringent metallic acid salt, such as the sulphate of alumina or zinc. In this case I can add the precipitating agent separately to the dissolved glue in any suitable apparatus, or I can mix the precipitating solution together before adding them to the size. Thus, in employing process No. 2, I can first add a solution of three pounds of bichromate of potash to every one hundred-weight of size and then a solution of sulphate of ammonia until the whole is precipitated; or I may mix the two solutions in a proportion of three pounds of bichromate of potash and from eight to ten pounds of ammonia sulphate together and add the same until all the size is precipitated. I add the solid precipitate formed to the pulp in the beating-engine and allow it to rub or beat fine with the pulp.

No. 12.—Instead of precipitating the glue in the pulp, or previous to its introduction into the beating-engine, I may, in the case of paper or boards, which are made in thin layers upon a drum, mix the chemical agents with the size in such a manner that the size becomes very thick without precipitating. I prefer for this purpose an alkaline chromate and an acid salt of alumina—for instance, chromate of potash and sulphate of alumina. I mix a solution of the two salts until the mixture becomes turbid—that is, until the mixed solution is almost neutral, the acid, however, supervening. Of this mixture I add about one gallon to every four gallons of size in solution, stirring well. I then introduce the thickened size into a conical-shaped box fitted with a steam-pipe, for the purpose of keeping the size fluid. The box is placed above the drum of the paper-machine, extending its whole length with the narrow end downward. The box is open at the bottom, the aperture being covered by a roller which rests upon the drum and revolves with the same, giving off the glue taken from the box to the layer of the pulp upon the drum. Flaps attached to the box at each side of the roller are drawn together or opened by means of screws, and thus by opening or pressing on the roller regulate or stop the flow of size from the box The glue in material made in this manner being homogeneous, such material does not require the treatment with heat, as hereinafter described. Of course the above-described process may be varied or blended according to circumstances, and although to the best of my experience they are the most applicable, I do not strictly confine myself to them. Taking, for instance, process No. 2, I could substitute, but not with such good results, sulphate of zinc alone or with other astringent acid salts in the place of the sulphate of ammonia. Instead of adding the chemical agents separately to the size, I may in some instances add all the chemicals together and precipitate with the mixed solution. Thus in the case of chromate of potash and sulphate of alumina I can mix a solution of each salt together until the mixture becomes turbid, and I then add this mixed solution to the size in the beating-engine until all the size is precipitated, which may be ascertained in all cases by filtering some of the liquid from the pulp into a glass and test-

ing it with some of the precipitating solution employed. If the liquid remains clear upon the addition of some of the precipitating solution, all the size is precipitated; but if it becomes cloudy more of the precipitating solution must be added. Instead of employing chromate of potash, very fair results are obtained by employing an acid salt of zinc and alumina in combination, and adding to a solution composed of equal parts of these salts a caustic alkali until the mixture is very turbid but still of an acid reaction. I would employ for this purpose the sulphate of zinc and of alumina and the soda hydrate. Although the precipitated glue is in an insoluble or very difficultly soluble condition in the fibrous pulp, it retains in most cases its strong adhesive property, and I make use of this for the purpose of rendering the material manufactured perfectly homogeneous and very strong, as required for the various purposes described.

For the purpose of merely sizing paper in the ordinary sense the usual treatment which paper receives in the course of manufacture is sufficient; but for special purposes I proceed as follows: In order to render the particles of glue contained in the fibrous material adhesive, I heat the sheet or board directly or soon after it leaves the machine—that is, while fresh and wet—to about 180° to 220° Fahrenheit in any convenient manner. I may expose the material for this purpose on heated surfaces or place it in heated chambers fitted with suitable shelves. When the material is thoroughly heated throughout, I subject it to pressure between felts or other porous fabrics for the purpose of expelling the water and allowing the particles of glue to unite with each other. The pressure is brought to bear very gradually upon the material, so as to allow the water to pass out without disturbing the felting of the fibers. The material is allowed to remain in the press until no more water escapes, and it is then hung up and dried in suitable rooms, which may be slightly warmed for the purpose of causing a dry atmosphere. The processes described under Nos. 1, 2, 3, 4, 5, 6, 7, 9, and the corresponding processes under Nos. 6 and 11 are adaptable to this method of rendering the material homogeneous. The omission of the ammonia chloride as mentioned does not hinder the material from becoming adhesive. Glue treated by the processes Nos. 8 and 10 does not become adhesive by heat, but in drying the particles of glue set to a solid mass. Therefore material made by processes Nos. 8 and 10 need not to be subjected to heat, but it is well to subject it to a moderate pressure; also in cases where I precipitate with a salt of an alkaline earth instead of with a salt of an alkaline metal. For instance, if I substitute hydrate of lime for sulphate of ammonia in process No. 1, the material produced is not affected, and such material need not be subjected to heat, but merely to pressure. The processes under Nos. 1, 7, 8, 9, and 10, and the corresponding processes under Nos. 6 and 11 produce a colorless or almost colorless sizing, and these are therefore adapted where a white material is to be produced and for sizing white papers in general. Of these the processes No. 1 and the corresponding processes under Nos. 6 and 11 produce the strongest sizing, and are adaptable for purposes accordingly, such as parchment-paper. In cases where the salts of chrome, copper, or iron are employed the size is distinctly colored.

For the purpose of making a substitute for leather the processes Nos. 2 and 5 and the corresponding processes under Nos. 6 and 11 give the best results, so far as my experiences go. These processes are also adapted for making substitutes for horn.

Material manufactured to take the place of horn is subjected to an extra pressure after it is dry. For this purpose I heat the material, when perfectly dry, to about 200° to 240° Fahrenheit, and I then place it between metal plates and into a hydraulic press and subject it to pressure of one hundred and sixty tons or more, and I have the material under full pressure for three hours or upward. Such material, when finished, can be bent to any shape, when heated, like natural horn. With all the processes described the glue is rendered more or less insoluble, but with the exception of Nos. 7 and 8, and in cases where hydrate of lime has been employed for precipitating the glue, it absorbs, when placed in water, a certain amount of the same, without, however, losing its strength.

No. 7 produces a sizing which, when dry, is perfectly foreign to water or damp, and is therefore applicable for all kinds of building purposes, being especially adapted for such purposes, as this process renders the material almost fire-proof. It is also useful for a great number of purposes where wood, bone, stone, &c., are employed.

For the purpose of imparting great softness to the material, such as is required for most leather purposes, I subject it to a bath of glycerine diluted with from three to five times its bulk of water. It is of course understood that the material is subjected to this bath when it is finished and dry. It is allowed to remain in the bath until saturated, and it is then again dried in any convenient manner. The material produced by such processes as Nos. 7 and 8 is not adaptable for this purpose, as will be naturally understood, being quite unaffected by moisture; also, material which has been heated and pressed for making substitutes for horn is not adapted for this purpose.

Material manufactured to take the place of leather after the processes described may be curried in a similar manner as natural leather.

Instead of heating and pressing the material as described for the purpose of rendering it homogeneous, I sometimes omit these operations, and merely dry the material as it comes from the machine. Such material is porous and slightly spongy, and may, if re-

quired, be saturated with oils or fats by immersion in oils or fat, or by rubbing these with the material.

For the purpose of sizing with starch or flour I suspend the starch or flour in water, and I add a solution of a caustic alkali until the mixture becomes transparent and gelatinous, and I add of this dissolved starch or flour the requisite quantity to the fibrous pulp in the beating-engine. Instead of dissolving the starch with a caustic alkali, it may be dissolved by boiling.

For ordinary sizing purposes, the practical paper-maker will be able to adjust the proportion of starch or flour to his requirements. For special purposes rather more will be required than for ordinary sizing. Thus, if the material is to be employed for some purposes where wood and bone are used, a good average proportion is about ten pounds of dry starch or flour for every hundred-weight of dry pulp.

Instead of dissolving the starch or flour before its introduction into the beating-engine, I may mix it in a dry state with the pulp in the beating-engine, and when thoroughly mixed with the fibrous pulp I add about five pounds of caustic soda in solution for every ten pounds of starch or flour, and I proceed as follows: I add to the starch or flour and the pulp in the beating-engine about twenty pounds of a solution of silicate of soda or other alkaline silicate for every ten pounds of starch or flour employed, and I then add a solution of a salt of an alkaline earth—such as calcium or barium chloride or magnesia sulphate—until no more precipitate will form; or, instead of an alkaline earth, I may employ a salt of chlorine—by preference ammonia chloride—for the purpose of precipitation. For purposes where a stronger sizing is required I precipitate with an acid metallic salt, omitting the silica. I prefer to employ the chloride of zinc or the sulphate of copper, a solution of either of which salts I add to the starch or flour and pulp in the beating-engine until no more precipitate will form. I may also precipitate with very good results with an acid solution of silica prepared with hydrochloric acid, as hereinbefore described. If the processes are employed for sizing paper in the ordinary sense, the paper is finished off in the usual manner; but for special purposes, when the material is to be employed for some of the purposes named, I subject it to a very gradual pressure while it is still in a wet state—that is, soon after it leaves the machine—and I then dry it by exposing it in any suitable rooms in the same manner as the glue-sized material. Still better results are obtained by partially drying the material before subjecting it to any pressure, and when about one-half or three-fourths dry to subject it to a strong pressure between soft porous material, allowing it to remain under full pressure for three hours or upward.

In employing copper sulphate heat may be used to facilitate the adhesion of the particles of starch in the same way as described under the animal-sizing process. The copper sulphate colors the starch or flour a bright green or blue. The other processes produce almost a colorless sizing.

I claim—

1. An improved process or method for treating fibrous material, which consists in precipitating glue, starch, or flour in the fibrous pulp, and then treating the materials in the manner described, so as to render them more or less water-proof without injuring their binding property, for the purposes specified.

2. The improved process or method herein described for treating fibrous materials, consisting in applying dissolved glue to fibrous pulp, then adding one or more astringent metallic salts or non-metallic elements, and then adding a caustic alkali or alkaline earth or a salt of the latter, for the purposes described.

3. The improved process or method herein described for treating fibrous materials, consisting in applying dissolved glue to fibrous pulp, then adding one or more astringent metallic salts or non-metallic elements, then adding a caustic alkali or alkaline earth, or a salt of the latter, and then pressing and drying the pulp, for the purposes set forth.

4. The method of incorporating animal glue with fibrous material by precipitating the glue with the pulp in a mixing apparatus, then adding one or more astringent metallic salts and one or more salts of an alkali or alkaline earth, as described.

In testimony whereof I have hereto set my hand this 30th day of November, 1883.

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