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PREPARATION OF COATING COMPOSITION

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In finishing surfaces of more or less porous character, such for instance as wood surfaces, filling and staining occupy an important position, since all subsequent materials must depend, for clearness of color, uniformity and smoothness of surface, upon the foundation of fillers and stains. If these are defective, no subsequent material, however excellent its quality, can be successful. If the pores of open-grain woods are properly filled a large saving can also be made in the subsequent shellac, lacquer, varnish or enamel. Again, it is found necessary to stain and fill wood to gain uniformity without unreasonable cost. It frequently happens that a piece of wood is blemished in part; for instance, a beautiful piece of walnut desirable for a table top may have light sap streaks in ten percent of its surface. If there were a staining material that could be applied to the entire surface and stain the sap streaks without darkening the remainder of the surface, the natural beauty of the wood may be enhanced and a tremendous saving be realized. In the usual procedure, a filler-material of some sort in liquid suspension is applied in considerable quantity over the surface to be treated and is then spread out upon the surface in a fairly uniform thick film. This film of filler-material is now allowed to evaporate and stiffen for a period of time, and is then carefully but vigorously worked into the surface of the wood. After the working operation, the excess is rubbed off until the surface is smooth. After this, the filler is allowed to dry, and finally the finishing coats, shellac, varnish, lacquer, enamel, or the like, are applied.

In this procedure, the extent to which the filler-material "pads" or forms a good layer on the cloth is determined largely by the characteristics of the material. The "slip", or the ease with which the rag or burlap is pushed across the surface during the rubbing-in operation is likewise contingent upon the properties of the filler, and the slip is bettered generally in proportion as the padding properties are favorable. Water-borne fillers, as contrasted to oil fillers, have

heretofore had quite a tendency to roughen the surface of an initially smooth piece of wood, or raise the grain. For this reason, oil fillers have had general preference, notwithstanding certain objectionable tendencies which characterize them. Oil fillers are vigorously attacked by pyroxylin lacquers. Where an oil-soluble stain is relied upon, there is generally a lack of desirable depth and uniformity of color, and if a lacquer-finish-coat be attempted, there is a tendency for the stain to bleed into or be dissolved by the lacquer. This tendency is even to be observed with shellac and varnish. A greater objection to the oil-type of stain-filler also, from the trade, is that the staining materials usually lie on top of the surface in a paint-like manner, making uniform wiping difficult. The drying-time, or period of time after the excess of filler has been rubbed off and before the final coat of finish can be applied, must be as short as possible compatible with a reasonable working time. By "working time" is meant the time required between the initial application of the filler and the rubbing off of the excess. Such working time must be long enough that a workman can finish a reasonably large section as a unit before the material sets to such an extent as to impede operations. The working time should not be so long that it becomes incompatible with a short drying time. In the case of oil fillers, the working time is usually ample, but on account of the tendency of these fillers to set hard, more or less inconvenience is encountered at the end of a day's run. With oil fillers, the piece must be finished rather than be allowed to stand over until the next day, since by that time it will have set so hard as to preclude removal. Over-time work is thus frequently necessitated where oil fillers, heretofore customary, are employed. Again, if the drying time required be unduly long, it results in an economic loss due to slowing down in production and an increase in the storage space required for the articles in treatment. For this reason, it is desirable to have a filler requiring a minimum of drying time. With oil fillers here-

tofore customary, the drying time requirement has seldom been less than four hours. A composition then, which is capable of cutting down the drying time to a short range, while also providing a working time range free from handicap by undue setting, is an important desideratum in the art and highly desirable. In accordance with the present invention, a composition capable of efficient application for usages in filling, staining, etc., and with such desirable properties, may be had, besides affording highly advantageous padding and slip characteristics, and without undesired tendency to raise the grain, although containing water.

To the accomplishment of the foregoing and related ends, the invention, then, comprises the features hereinafter fully described and particularly pointed out in the claims, the following description setting forth in detail certain illustrative embodiments of the invention, these being indicative however, of but a few of the various ways in which the principle of the invention may be employed.

In accordance with the invention, we provide a water-containing composition including a silicious preparation. As raw material, we ordinarily employ sodium silicate solution, although other soluble silicates amenable may likewise be employed, and are contemplated as equivalents correspondingly. Starting with a sodium silicate solution, we treat this with an agent capable of producing a controlled thickening, without withdrawal of water. This may be accomplished by adding to a soluble silicate solution another solution which is sufficiently acid with respect thereto to cause a colloidal precipitation of silicic acid or where desired it may be accomplished by adding a metallic compound which will produce a solid suspension or a gelatinous precipitate of an insoluble silicate of the metal, as for instance aluminum, barium, calcium, cadmium, copper, iron, lead, magnesium, titanium, zinc, etc. For the acid-type of precipitation it is desirable to employ such weakly acid agents as sodium acid phosphate, sodium bicarbonate, etc. In general, the rate of such action should not be too rapid. The rate depends upon concentration. If the treating agent is employed too concentrated for instance, the solution will precipitate almost at once, and the resulting material will lack the most desirable characteristics. If the treating agent be employed in too low concentration on the other hand, effective action is not had. Besides concentration, other factors influencing the properties of the resultant product are temperature, agitation, particular composition of the solutions, and the length of time they are allowed to act. This latter "ageing time" is found to bear an important relation to the final properties of the materials, since they only develop their maximum physical properties after a more or less definite time which we thus call the "ageing time". Depending upon the particular composition of the solutions and the treatment to which they are subjected, the ageing time may vary from several minutes to two weeks or more.

Temperatures of about 70° F. are desirable, and vigorous agitation is beneficial. Higher or lower temperatures are less advantageous, particularly higher temperatures since they increase the reaction rate. In the acid-type of precipitation, the action is brought about by the addition of a dissociable agent of generally acidic character, as already noted, and in this relation the

hydrogen ion concentration becomes of importance. Where silicic acid is precipitated, the pH varies from about 7.8 to about 13, if sodium bicarbonate be employed as treating agent. For this, the average pH is about 10 or 11. When using sodium acid phosphate, the pH will average very nearly 7. The concentration of the solutions may be about the same for both the first and second types of reaction. It has been found possible to produce results with solutions containing as low as ½ of 1 percent of sodium silicate and about 1 per cent of sodium bicarbonate or its equivalent amount of sodium acid phosphate. The concentration of sodium silicate may be as high as 20 per cent or more, and the sodium bicarbonate or its equivalent in sodium acid phosphate as high as 5 per cent or more. For example, a 4 per cent sodium acid phosphate solution (based on the weight of the phosphate to the weight of water), and a 6 per cent sodium silicate solution (based on volume parts of 40° Bé. sodium silicate to volume of water) may be used. Desirably, a 5 per cent phosphate solution and 6 per cent silicate solution may be used. On being well mixed and allowed to stand, the character of the mixture changes, and a marked thickening occurs. If such a solution be applied to a porous surface, in accordance with customary procedure in finishing-operations, it is found that unlike sodium silicate, which would dry to a coherent hard coating, the present material provides a finely divided filling which lodges in the pores, but the surplus is freely removable from the outer surface. In making use of the second type of reaction, a metallic compound, for instance an oxy-compound as zinc oxide or basic lead carbonate, is agitated along with the rest of the solid material in the solution of sodium bicarbonate or sodium acid phosphate, and the sodium silicate is then added. The metallic compound, such as the zinc or lead compound, is generally used in the proportion of 1 pound of solid to 1 gallon of solution.

Along with the improved filler base, we may also combine stains, such as aniline dyes, natural dye stuffs, pyrogallol and the like suitably treated with oxidizing agents, pigments, etc. In fact, by causing precipitation of the silica from the sodium silicate in the presence of such dye or pigment, it is found that the action of the latter is much improved, possibly due to the extremely finely divided and adsorbent character of the ultimate precipitate which is left on the drying of the filler. In such stain, water-soluble or oil-soluble stain-materials may be employed, water-soluble stains being preferable. One desirable manner of combining the stain with the filler may be realized for instance, by employing a stain made up of pyrogallol, with sodium bicarbonate (the latter in the proportion of about 21 pounds and 3 ounces per 100 gallons of water), and subsequently the addition of about 7-10 pounds of sodium dichromate. The amount of pyrogallol will vary, depending upon the depth of color desired. In making up any such compositions, it is desirable to mix all of the ingredients together, except the silicate solution. This is stirred in last.

We may also incorporate an oil, that is a drying oil, preferably linseed oil, this being in a minor proportion. Furthermore, it is generally advantageous to also include finely divided mineral matter such as quartz, silica, mica and the like.

As illustrative of compositions within the pur-

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view of our invention, the following examples may be noted:—

5	I.	Sodium silicate solution 40° Bé.....	parts..	15
		Sodium bicarbonate solution (2½ percent).....	do.....	60
5		With this may be incorporated, if desired, as much as 25 parts by weight of finely divided minerals, such as quartz, mica, silicx and the like. On the initial mixing no particular change is observed. On standing however, for 3 or more days the solution is found to have thickened up very materially. It is then ready for use.		
10	II.	Sodium silicate solution, 40° Bé.....	cc.....	15
		Stain solution (comprising 2 percent pyrogallol and about 2½ percent sodium bicarbonate in water).....	cc.....	60
		Bone black.....	grams.....	2
		Quartz (400 mesh).....	do.....	15
		Amorphous silica (silicx powder).....	do.....	5
15	III.	Mica (160 mesh).....	do.....	5
		Sodium silicate solution, 40° Bé.....	cc.....	12
		Stain solution (including sodium bicarbonate 2½ percent).....	cc.....	40
		Water.....	do.....	15
		Glue.....	grams.....	2
		Starch.....	do.....	2
		Sodium salicylate.....	do.....	15
		Boiled linseed oil.....	cc.....	7
		Turpentine.....	do.....	6
		Quartz (400 mesh).....	grams.....	20
20	IV.	Sodium silicate solution, 40° Bé.....	cc.....	15
		Stain solution (including sodium bicarbonate 1 to 5 percent).....	cc.....	60
		Casein.....	grams.....	3
		Boiled linseed oil.....	cc.....	6
		Bone Black.....	grams.....	1
		Quartz (400 mesh).....	do.....	25
		Finely divided amorphous silica (silicx).....	do.....	10
25	V.	Mica (160 mesh).....	do.....	5
		Sodium silicate solution, 40° Bé.....	cc.....	15
		Stain solution (including sodium bicarbonate 1½ to 5 percent).....	cc.....	60
		Boiled linseed oil.....	do.....	4
		Varnish.....	do.....	3
		Bone black.....	grams.....	2
		Quartz (400 mesh).....	do.....	15
30	VI.	Finely divided amorphous silica (silicx).....	do.....	5
		Mica (160 mesh).....	do.....	15
		Sodium silicate solution, 40° Bé.....	cc.....	5
		Stain solution (including sodium acid phosphate 5 percent).....	cc.....	60
		Boiled linseed oil.....	do.....	4
		Varnish.....	do.....	3
		Bone black.....	grams.....	2
35	VII.	Quartz (400 mesh).....	do.....	15
		Finely divided amorphous silica (silicx).....	do.....	5
		Mica (160 mesh).....	do.....	15
		Zinc oxide.....	do.....	5
		Sodium silicate solution, 40° Bé.....	cc.....	15
		Stain solution (including sodium bicarbonate 1½ to 5 percent).....	cc.....	60
		Boiled linseed oil.....	do.....	4
40	VIII.	Varnish.....	do.....	3
		Bone black.....	grams.....	2
		Quartz (400 mesh).....	do.....	15
		Finely divided amorphous silica (silicx).....	do.....	5
		Mica (160 mesh).....	do.....	15
		Zinc oxide.....	do.....	5
		Basic lead carbonate.....	do.....	5

Other modes of applying the principle of the invention may be employed, change being made as regards the details described, provided the features stated in any of the following claims, or the equivalent of such, be employed.

We therefore particularly point out and distinctly claim as our invention:

1. A process of making an improved liquid wood filler, comprising colloiddally thickening, by the precipitation of colloidal silicic acid, a one-half to twenty per cent solution of a 40° Bé. commercial sodium silicate, by the admixture therewith of a one to five per cent solution of an acid salt of an alkali metal, in such an amount that pH value of the mixture is reduced to a value within the range pH 13 to pH 7.

2. A process of making an improved liquid wood filler, comprising colloiddally thickening, by the precipitation of colloidal silicic acid, a one-half to twenty per cent solution of a 40° Bé. commercial sodium silicate, by the admixture therewith of a one to five per cent solution of sodium bicarbonate, in such an amount that the pH value of the mixture is reduced to a value within the range of pH 13 to pH 7.

3. A process of making an improved liquid wood filler, comprising colloiddally thickening, by the precipitation of colloidal silicic acid, a one-half to twenty per cent solution of a 40° Bé. commercial sodium silicate, by the admixture therewith of a one to five per cent solution of sodium acid phosphate, in such an amount that the pH value of the mixture is reduced to a value of about seven.

4. A process of making an improved combined wood filler and stain comprising colloiddally thickening, by the precipitation of colloidal silicic acid, a one-half to twenty per cent solution of a 40° Bé. commercial sodium silicate, by the admixture therewith of a mixture of a coloring material adapted for use as the coloring ingredient of a wood stain and a one to five per cent solution of an acid salt of an alkali metal, in such an amount that the pH value of the mixture of silicate solution, coloring material and solution of the acid salt is reduced to a value within the range of pH 13 to pH 7.

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