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PROCESS OF PRODUCING WEATHER-RESISTANT GLUE BOND

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This invention relates to the art of gluing, and has particular reference to the fabrication of wood veneers into plywood panels possessing superior water and weather resistant qualities. More particularly, the invention relates to a method of gluing applicable to the manufacture of exterior grade plywood.

Heretofore, exterior grade plywood has been manufactured only by those plywood mills possessing equipment for the simultaneous application of heat and pressure to the assembled panels. In the manufacture of such plywood it is a customary practice to apply a thermo-setting phenolic resin glue to the opposite faces of core veneers and to set these aside until the glue has dried. Thereupon, the several veneers comprising plywood panels are assembled with the layer of adhesive material in the joints between adjacent veneers, and the rudimentary panels are inserted in the openings of a hot plate press for the simultaneous application of heat and pressure. The press is heated to from 240° F. to 320° F., depending upon the character of the glue applied in the joints, while the pressures applied during formation of the bond range from 100 pounds per square inch to 400 pounds per square inch, depending upon the hardness of the wood. The heat of the press so completely drives the moisture from the surface plies of the panel that it is necessary after the pressing operation to subject the panels to a humidifying process during the time the panels are cooling to relieve the stresses in the hot pressed panels for preventing checking and warping.

The present invention provides a process of gluing particularly applicable to the manufacture of plywood, in the practice of which the hot plate press is no longer an indispensable equipment for the production of exterior grade panels. Heretofore, the use of a thermo-setting glue consisting in whole or in part of phenolic resin has been thought to require the simultaneous application of heat and pressure to first liquefy the glue, force it into the pores or cells of the wood, and then to convert it into an inert substance permanently bonding the veneer plies together. In contradistinction to this long used and well known process, applicant has discovered that the thermo-setting resinous glue may be combined with any satisfactory proteinaceous glue such as has heretofore been used in the cold press process, and that the gluing operation may be performed in separate successive steps requiring neither the application of excessive pressure nor an excessive amount of heat, and that the completed panel possesses the high water and weather resistant qualities of exterior grade plywood, as well as being immune to attack by bacteria, molds, fungi and vermin and unaffected by organic solvents.

Briefly outlined, the invention comprises the process of assembling the requisite number of wood veneers to make up plywood panels of the desired number of plies, and forming between adjacent veneers in each panel an interface consisting of a mixture of proteinaceous and thermo-setting resinous glues. This may preferably be done by first applying liquid glue to opposite faces of a sheet of wood veneer and thereupon laying up the panels by placing the veneer sheets to which glue has been applied between other veneer sheets to make panels of the requisite number of plies. Thereupon pressure is applied to the panels as by means of a hydraulic press, and pressure is maintained by the use of suitable clamps until a bond is formed, as in a cold press operation. Thereafter, the panel is heated in the absence of pressure to complete curing of the bond.

It is an object of the invention to provide a process of gluing which will produce a bond possessing superior water and weather resistant qualities, and which may be used in the manufacture of plywood for the production of exterior grade panels.

It is a further object of the invention to provide a process of gluing whereby exterior grade plywood panels may be made without the aid of the hot pressing equipment heretofore deemed necessary for the production of such panels.

It is a further object of the invention to provide a process of gluing whereby exterior grade plywood panels may be made with the use of the conventional cold press equipment in plywood mills.

It is a further object of the invention to provide a process of gluing which will greatly reduce the cost of fabricating exterior grade plywood panels and which will many times increase the capacity of a plywood mill for panels of such grade.

It is a further object of the invention to provide a process of gluing embodying the application of pressure and heat in successive steps and which is productive of a bond of unusual strength in both the wet and dry condition of the panel.

It is a further object of the invention, in a specific application thereof, to provide a protein-resin glue of high water content which is capable, upon the application of pressure and heat in successive steps, of producing a boil-proof bond.

In carrying out the invention, a colloidal sus-

pension or solution of resin is produced by mixing a phenolic body and formaldehyde in an aqueous alkaline solution and heating the mixture until the reaction is at the desired end point. The term "phenolic body" is intended to embrace phenolic bodies such as phenol, cresol, resorcinol, naphthol, xylenol, or their homologs, derivatives or compounds. A phenolic condensation product may be prepared by mixing 100 parts of a commercial phenol (85% phenol) with 100 parts of commercial formalin (40% formaldehyde) and heating the mixture in the presence of from 5 to 10 parts of caustic soda for a period of from 30 to 90 minutes. The amount of heat applied should be sufficient to bring the mixture to the boiling point, and the time required is determined by that which is necessary to produce the desired end point, i. e., whether a solution or a colloidal suspension, and the percent of solids present. A suitable viscosity is obtained by adding water or a dilute caustic soda solution. The proportion of solids is usually adjusted to about 40%. Other phenolic bodies such as cresol, resorcinol, naphthol or xylenol can be substituted for phenol in the foregoing illustration by making suitable adjustments in the proportions as is well understood in the resin making art. The product compounded as above set forth is a liquid composition such as is used in the production of exterior grade plywood panels by the simultaneous application of heat and pressure as in the hot plate process.

The resinous glue compounded as hereinabove illustrated is mixed with a proteinaceous glue prepared as for use in the conventional cold press gluing operation. The glue base for the proteinaceous glue may be any one of the various proteins commonly used for making cold press glues, namely, soluble blood, casein, soy bean flour or any one of the vegetable seed meal flours of which soy bean flour is an example, and any one of the hydrolysis products of collagen referred to broadly as animal glue. It has been discovered also that the process is applicable for use with heat coagulated blood, and a protein-resin glue compounded with this substance is especially attractive, both by reason of the economy of the high water ratio in the proteinaceous glue, and also because of the excellent results obtained as regards the shear strength of the bond and the percentage of wood failure both for the dry panel and after being subjected to the boiling test required for exterior grade panels.

The proteinaceous glue is prepared as for use in the conventional cold press operations. The following formulas are set forth illustrating the process for compounding these glues:

Soluble blood.—To 300 parts of water at room temperature add 100 parts of soluble blood. This mixture requires the presence of an alkali to produce a satisfactory cold press bond. In the instant process the alkalinity is supplied by the caustic soda combined with the resin.

Vegetable seed meal flour.—To 300 parts of water at room temperature is added 100 parts of soy bean flour, or other vegetable seed meal flour. To this mixture is added 8 parts of lime dissolved in 30 parts of water, 9 parts of caustic soda dissolved in 45 parts of water, and 30 parts of silicate of soda. Stirring is continued during and after the addition of each ingredient until thorough mixing of the mass is assured.

Casein.—To 225 parts of water at room temperature is added 100 parts of dry casein. To this mixture is added 11 parts caustic soda dissolved in 30 parts of water, 20 parts of lime dissolved in 45 parts of water, and 40 parts of silicate of soda. Stirring is continued during and after the addition of each of the named ingredients until thorough mixing is assured.

Animal glue.—To 200 parts of water at room temperature add 100 parts of animal glue flakes. After soaking for several hours the temperature is raised to 140° F. and the mass stirred until the animal glue flakes are completely dissolved. Thereupon, there is added 6½ parts by weight of glacial acetic acid and the mixture cooled to 80° F.

Heat coagulated blood.—To 475 parts of water is added 100 parts of dried and ground heat coagulated blood, and the mass thoroughly mixed by a suitable stirring device and heated to 210° F. To the mixture of water and blood is then added 4½ parts of caustic soda dissolved in 25 parts of water and the temperature maintained for approximately 10 minutes. Thereupon, the mixture is rapidly cooled to 80° F. and there is added 8 parts of lime dissolved in 32 parts of water, 4½ parts of caustic soda dissolved in 18 parts of water, and 40 parts of silicate of soda. Each of the ingredients is stirred into the mixture, stirring being continued during and after the addition of each ingredient to insure thorough mixing. This glue is made according to the disclosure in my copending application, Serial No. 413,807, filed October 6, 1941, for Glue and process of manufacture therefor, and requires the use of Type I or Type II blood as therein defined.

The proteinaceous glue compounded as above described is mixed with the resinous glue in a proportion of approximately 60 parts of dry glue base to 40 parts of the phenolic condensation product based on its solids content. The resinous glue is added slowly to the proteinaceous glue, the mixture being thoroughly stirred during the time the resinous glue is being added. The resulting protein-resin glue may be applied to sheets of wood veneer by the use of conventional glue applicator rolls. The wood veneers are assembled with a layer of the protein-resin glue between adjacent plies of the rudimentary panels and pressure is applied as by a conventional hydraulic press and maintained by suitable clamps for a time to insure preliminary setting of the glue. The pressure usually applied in the cold press process for making plywood, namely, of the order of 150 pounds per square inch, has been found adequate to complete the first step in the instant process. It may be said that this step ends when the pressure is released. The bond produced is inferior to a bond formed by the proteinaceous glue alone as regards both dry strength and wet strength. (See Table I.) It is, however, sufficiently strong to withstand the stresses imposed by subsequent handling of the glued panels.

After the formation of the preliminary bond by the cold press apparatus, the panels are heated to a temperature of the order of 200° F. as by putting them through a veneer drier, or placing them in a kiln, or by any other practical means. It is a distinct advantage of the invention that the heat required to cure the bond may thus be applied by standard equipment usually to be found in all plywood manufacturing plants. The application of heat converts the bond into one which is not only of superior dry strength, but

one possessing high water and weather resistant qualities. It appears that all that is required in the heating step is that the temperature of the bond be raised to the order of 200° F. It is evident that the optimum temperature varies according to the glue mixture, and, while, obviously, higher temperatures may be used, panels possessing excellent strength and water and weather resistant qualities have been produced with the use of temperatures ranging from 160° F. to 225° F.

For purposes of illustration, a quantity of each of the proteinaceous glues compounded as hereinbefore described was mixed with a phenolic resin in an aqueous alkaline solution and fir plywood panels were made with each of the glues by the hot pressing process and also by the cold pressing process. One set of panels made by the cold pressing process was heated to a temperature of the order of 200° F., and these panels, as well as the unheated cold pressed panels and the hot pressed panels, were cut into test pieces, half of which were subjected to the shear strength test while dry, and the other half of which were boiled for eight hours and then tested. In the following table each of the figures indicates the average strength and percent wood failure resulting from 40 test pieces tested in the manner specified by the Douglas Fir Plywood Association.

tion, the bond was definitely poor. However, the bond produced by the cold press process was of sufficient strength to withstand the stresses imposed by subsequent handling operations, so that the panels could be removed from the cold press and placed in a conventional veneer drier, where they were heated to a temperature of the order of 200° F. Thereupon, the panels were subjected to the tests prescribed by the Douglas Fir Plywood Association for exterior grade plywood, and it will be noted that both as regards dry strength and wet strength, i. e., after being boiled for a period of eight hours, the bond was definitely superior to even that produced by the hot press process.

It is important to note that, whereas all of the straight protein base glues of no matter how high water resistance fail on weathering, due to the attack of fungi, mold and other organisms, when combined with phenolic resins and subsequently heated to cure the resin, these glues are not attacked by such organisms. Heretofore the addition of an anti-mold agent to protein glues in sufficient amount to be effective in preventing bacterial growth has reacted to very seriously damage the strength and water resistance of the glue bond, so that it has proved impossible to produce a weather resistant bond by the use of proteins in the cold press process. The invention

Table I

Panels	Composition of protein glue						Ratio, dry basis		Hot press				Cold press				Cold press and heat			
									Dry		Boiled 8 hrs.		Dry		Boiled 8 hrs.		Dry		Boiled 8 hrs	
	Glue base	NaOH	Lime	Sil.	CS_2	Total H_2O	Protein	Resin	Shear strength	Percent wood failure	Shear strength	Percent wood failure	Shear strength	Percent wood failure	Shear strength	Percent wood failure	Shear strength	Percent wood failure	Shear strength	Percent wood failure
A	Coagulated blood	9	8	40		550	60	40	231	76	168	46	163	21	74	07	180	92	184	73
B		9	8	40		550	70	30									192	85	158	65
C		9	8	40		550	50	50	203	81	156	68	133	14	66	04	201	93	162	94
D		9	8	40		550	30	70									193	95	175	88
E	Soluble blood	0	0	0		300	60	40	238	96	196	91	147	15	91	19	180	94	189	94
F		0	0	0		300	70	30									181	73	170	68
G		0	0	0		300	50	50	204	92	152	89	134	29	72	15	185	88	143	73
H		0	0	0		300	30	70									181	83	142	72
I	Soy bean flour	9	8	30		375	60	40	202	48	132	21	158	29	36	02	166	87	172	62
J		14	8	40	2	475	70	30									175	87	146	52
K		14	8	40	2	475	50	50	184	67	149	55	136	15	34	02	176	86	157	80
L		14	8	40	2	475	30	70									176	84	153	86
M	Casein	11	20	40		300	60	40	227	81	189	54	169	23	44	0	181	75	177	44
N	Animal glue	(Acetic acid, 6½ parts by wt.)				200	70	30									173	82	98	10
O						200	50	50	0	0	0	0	164	19	46	01	181	97	144	71
P						200	70	30									176	93	161	82

The above table clearly indicates the heretofore unheard of and wholly unexpected results obtained by the instant process as compared to the results produced by the conventional hot press and cold press processes. The several protein-resin glues are compounded as hereinbefore disclosed and applied to the core veneers of the panel by conventional glue applicators comprising horizontal applicator rolls between which the core veneers are fed. On the basis of the dry glue base and dry resin, the glue was compounded in proportions which extended through a range of from 30 parts of resin to 70 parts of resin.

The bond produced by the cold press process of gluing is considered to be entirely unsatisfactory for commercial use, even as regards the dry strength of the bond, and after the boiling test prescribed by the Douglas Fir Plywood Association, lies in the discovery that panels produced by the cold press process with protein-resin glues, when subsequently heated without pressure, acquire a bond of high strength and weather resistance and one which, because of immunity to the attack of organisms such as mold, etc., is weather resistant and suitable for exterior use.

In Table I the ratio of protein to resin (dry basis) is shown as being of varying proportions extending from a ratio of 70 parts of protein to 30 parts of resin to and through a ratio wherein the glue comprised 30 parts of protein to 70 parts of resin. It has been discovered, however, that the ratio of resin to protein may be varied through a range as great as from 15.0 parts resin to 85.0 parts resin and that panels possessing a high degree of water and weather resistant qualities may be produced with such glues by the herein described process. It will be appreciated

that when the proportion of resin to protein is varied the dry strength of the cold press bond before heating will improve as the proportion of protein increases; and that, on the other hand, the weather resistant qualities of the bond, as measured by shear strength tests after boiling, will improve as the proportion of resin is increased. The following data illustrate the progressive improvement of the dry strength of the bond following the cold press operation as the proportion of protein is increased; and also the improvement of the wet strength of the bond following the application of the instant process as the proportion of resin is increased.

Protein	Resin	Cold press, dry		Cold press and heat, boil	
		Shear strength	Percent wood failure	Shear strength	Percent wood failure
70	30	166	27	154	63
50	50	121	04	166	82
30	70	100	04	181	91

Particular attention is directed to the data identified as panels N, O and P in Table I. These panels were fabricated with a protein-resin glue comprising animal glue and an aqueous alkaline solution of phenolic resin. The animal glue was made in the conventional manner having a water ratio of 2 parts of water to 1 part of dry glue base. To prevent gellation of the mixture due to the action of the formaldehyde in the resin upon the animal glue, glacial acetic acid is added to the animal glue before the glue is mixed with the resin. In each case the acid is added in sufficient quantity to neutralize the alkalinity of the resin and give the animal glue-resin mixture a pH slightly on the acid side. When an aqueous alkaline phenolic resin solution is mixed with the acidified animal glue and its alkalinity thereby neutralized, the resin is precipitated. However, the precipitate is peptized by the animal glue in acid solution and remains in suspension in various degrees of colloidal dispersion and the mass remains viscous and spreadable. It will be noted that this mixture, when subjected to cold pressing, produces a sufficient dry bond to permit the panels to be handled, and that when these panels are subsequently heated a very strong and boilproof bond is obtained.

A distinct advantage of the process of gluing herein described is that the moisture content of the completed panels is easily adjusted during the heating step, and a subsequent humidifying process to restore their normal moisture content is not required. The protein resin glue, being an aqueous dilution, imparts to the glue line a considerable moisture content. During the pressing operation, i. e., the time during which suitable clamps are used to maintain pressure on the panels, the moisture migrates from the glue line to the wood and the subsequent application of heat effects a rapid curing of the bond. In the event it is deemed necessary either to increase or reduce the moisture content of the panels, this may conveniently be done by adjusting the humidity of the air surrounding the panels during application of heat thereto.

One of the difficulties encountered in the conventional hot press process is the formation of blisters between the veneers of respective panels by reason of the fact that the heat of the press converts the moisture present in the glue line into steam. The steam is trapped between the veneers and forms steam pockets or blisters which prevent the formation of a bond between the veneers wherever these blisters occur. For this reason a relatively light glue spread, of the order of 40 to 80 pounds of wet glue per thousand square feet of veneer surface, is used to avoid, as far as possible, the presence of moisture in the glue line. In the instant process the fact that the moisture content of the glue line is considerably reduced during formation of the cold press bond, and that the temperature to which the panels are subjected during the heating step is not necessarily sufficient to convert any remaining moisture in the glue line into steam, blistering of the panels is avoided. This enables the use of a considerably greater amount of wet glue per thousand square feet of veneer surface than in the hot press process, and the bond is correspondingly improved.

Having now described my invention and in what manner the same may be used, what I claim as new and desire to protect by Letters Patent is:

1. The process of making plywood comprising assembling wood veneers in face to face superimposed relation with a layer of an adhesive material comprising an admixture of a proteinous glue and an aqueous solution of a thermosetting phenol-formaldehyde resinous glue between adjacent veneers in which the ratio of protein to resin is not less than 1.5:8.5 nor more than 8.5:1.5, applying pressure to the assembled panel for producing a bond between adjacent veneers of sufficient strength so that the rudimentary panels can be handled, and subsequently heating the panels to cure the bond and produce a panel possessing high water and weather resistant qualities.

2. The process of making plywood comprising assembling wood veneers in face to face superimposed relation with a layer of an aqueous dispersion of a protein-resin adhesive material between adjacent veneers, the resin constituent of said material comprising an aqueous solution of a thermosetting phenol-formaldehyde reaction product, applying pressure to the assembled veneers to produce a bond therebetween of sufficient strength to withstand stresses imposed by handling in subsequent operations, and subsequently heating the panels to a temperature not substantially lower than 160° F. to cure the bond and produce a panel possessing high water and weather resistant qualities.

3. The process of forming a glued joint comprising applying between the members to be joined a glue comprising an aqueous dispersion of a proteinaceous cold setting glue admixed with a thermosetting phenol-formaldehyde reaction product, applying pressure to the joint to cause said glue to form a bond with said members, and subsequently heating said joint to a degree sufficient to cure said bond.

4. The process of making plywood comprising assembling wood veneers in face to face superimposed relation, applying between adjacent veneers a glue comprising an aqueous dispersion of a proteinaceous cold setting glue comprising blood in aqueous mixture admixed with a phenol-formaldehyde thermosetting condensation product, applying pressure to the assembled veneers for producing a preliminary bond between adjacent veneers of sufficient strength to withstand stresses imposed by handling in subsequent operations, and subsequently heating the veneers to cure the bond and produce panels possessing high water and weather resistant qualities.

5. The process of making plywood comprising preparing a proteinous glue as for use in the cold press gluing of plywood, preparing an aqueous solution of a thermosetting phenol-formaldehyde resinous glue as for use in the hot press gluing of plywood, mixing the proteinous and resinous glues as by adding one to the other and stirring the mixture, assembling wood plies with an interface of the proteinous-resinous mixture between adjacent plies, applying pressure to the assembled panels as in the cold pressing operation and subsequently heating the panels to effect the setting of the resinous constituent of the adhesive mixture.

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