HEAT TREATMENT OF EXTERIOR PARTICLE BOARD

Thesis for the Degree of M. S. MICHIGAN STATE UNIVERSITY RICHARD C. ENLOW 1968 THESIS



ABSTRACT

HEAT TREATMENT OF EXTERIOR PARTICLE BOARD

by Richard C. Enlow

The objective of this study was to determine the effect of a heat treatment on the performance of a threelayer exterior particle board. A heat treatment may affect board performance in three ways:

- 1. Reduction of hygroscopicity of wood flakes
- 2. Reduction of compressive stresses within the board by stress relaxation at high temperatures
- 3. Reduction of mechanical or strength properties by thermal deterioration.

The proposed tests were designed to detect these components at two temperature levels and three heating periods. The ideal treating method would be a compromise between the beneficial and adverse effects of a heat treatment.

Three-layer exterior particle boards were manufactured in the lab and then heat treated. Half of the boards were heated to 350°F and the remaining half to 425°F. Heating periods were for 30 minutes, 1 hour, and 2 hours. Tests were run to determine the effect of heating on modulus of elasticity, internal bond strength, thickness swelling, hygroscopicity or water absorption, and surface smoothness. Color changes were also noted. Depending upon the properties examined, samples were tested at room conditions, at equilibrium conditions of high humidity, at room conditions after redrying, and after soaking for 24 hours.

Heat treating reduces modulus of elasticity by about 12% at the most severe conditions, but this was not considered critical since most exterior applications of particle board are relatively nonstructural. Internal bond strength was increased for all levels of treatment over the control value due to additional curing of the resin. Boards arranged in increasing order of severity of treatment were also in increasing order of darkening and dimensional stability, except for two treatments that result in approximately the same stability.

Heat treating to a temperature of 425°F for 1 to 2 hours produces the most desirable results with 40 to 50% reduction in thickness swelling. However, water absorption was approximately the same for all samples. The reduction in thickness swelling was due primarily to a relaxation of compressive stresses within the boards. Little, if any, change was noted in hygroscopicity. Surface profiles taken of samples treated and untreated indicated a definite improvement in smoothness of heat treated boards over the untreated boards after exposure to high humidity and soaking.

A heat treatment of exterior particle board appears to be a promising method of dimensional stabilization to make particle board more competitive with exterior plywood and other exterior wood based products.

HEAT TREATMENT OF EXTERIOR

PARTICLE BOARD

Ву

Richard C. Enlow

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CHAPTER I

INTRODUCTION

Particle board is a wood based product, manufactured from wood chips or flakes and a resin. The properties of particle board are functions of material and process variables. Commercial standards have been adopted that specify maximum and minimum values for these properties, regardless of the raw material or process used. Because of the nature of particle board and the processes used in manufacturing it, no two boards are alike in every respect.

Particle board is classified as interior or exterior, depending upon the resin used as a binder in the manufacturing process. Exterior particle board is manufactured with a completely water proof resin, usually phenol formaldehyde. Interior particle board is manufactured with a resin that is water resistant only, usually urea formaldehyde. The classification of particle board manufactured with a water proof resin as exterior is misleading. The performance of particle board manufactured with a water proof resin, when subjected to exterior applications, is not comparable to that of other wood based products bonded with an exterior resin (15).

Built-in compressive stresses within the board are the result of pressing a relatively thick mat of material into a thin board and holding it there by curing the resin with heat while under pressure. There is a natural tendency for the compressed material to spring back to its original thickness immediately following the release of pressure, but the cured binder prevents it from doing so.

The net effect of this condition on the performance of particle board is excessive thickness swelling when subjected to conditions of high humidity. Some areas within a particle board may have a density exceeding 1.0 gms/cm³ and will swell excessively pulling less dense areas along with it (16). The more plastic condition of the wood flakes, when wet from exposure to high relative humidity, allows springback to occur along with natural swelling. The natural swelling is almost entirely recoverable when redried, but swelling attributed to springback will not be recovered.

Due to excessive thickness swelling, there are various shortcomings common to interior and exterior particle board, but they are more pronounced in exterior particle board because of the extreme environmental changes it is subjected to. Some of these shortcomings are as follows: (1) poor dimensional stability including thickness swelling, edge flare, and linear movement; (2) surface roughening and telegraphing through veneers and finishes;

(3) screw and nail popping; (4) reduced strength properties such as bending strength and internal bond strength.

Because of these pronounced shortcomings and unsatisfactory performance, the current annual production of exterior particle board in the United States is only 0.5% of the total particle board production. Research in the direction of improving the performance of exterior particle board seems necessary in light of the market potential that exists for exterior wood based products.

The approach this thesis will take is that of reducing compressive stresses within particle board and reducing hygroscopicity of the wood flakes through a heat treatment similar to that used in the tempering of hardboard in an oven after pressing. Any heat treatment of particle board is limited to exterior-type board because of the temperature sensitivity of urea resins. Because the manufacturing process of particle board is centered around shortening the length of the press cycle, any treatment that would lengthen press time is not economically feasible. Therefore, a post treatment in an oven is the approach taken.

The objective of this thesis is to determine the effect of a heat treatment on the performance of a threelayer exterior particle board. A heat treatment may affect board performance in three ways.

- 1. Reduction of hygroscopicity of wood flakes.
- Reduction of built-in compressive stresses
 by stress relaxation at high temperatures.
- Reduction of mechanical properties by thermal deterioration.

The proposed tests are designed to detect these components at two temperature levels and three heating periods. The ideal treating method would be a compromise between the beneficial and adverse effects of the heat treatment.

CHAPTER II

THEORY OF DIMENSIONAL STABILIZATION

<u>A Look at the Need for Dimensional</u> Stabilization

The primary reason for trying to stabilize exterior particle board is the need to reduce excessive thickness swelling. Heebink (4) defines degradation in exterior particle board as, "any change that seriously reduces the ability of the board to meet its use requirements." However, degradation that results in strength losses, color change, and warping is probably secondary to excessive dimensional changes since most exterior uses of particle board are essentially nonstructural, requiring only moderate strength values (4). Warping is usually not a technical fault of the board itself, but a fault of the manufacturer producing an unstable product. Color changes appear to be secondary because the major applications of exterior particle board are in the housing field for such products as sheathing, siding, and soffits. In most of these applications a finish would be applied, or in the case of sheathing the board would be concealed. These products vary somewhat in their essential requirements, but they have one common denominator -- they must maintain

their integrity when exposed to the most severe conditions for their particular use (4).

General Approaches to Stabilizing Wood

Stamm (12, 13, 14) lists four major approaches to stabilizing wood:

- 1. Chemical reduction of hygroscopicity of wood,
- Baulking the fibers to limit the amount of water that can be taken up or lost,
- Cross-linking of the fiber structure so that the separation of the structural units is limited,
- Reduction of hygroscopicity of wood through a heat treatment.

An appreciable amount of stabilization can be obtained using any or a combination of these approaches, but certain drawbacks have led to limited use in special instances only. Strength losses, embrittlement, loss in toughness, and loss in abrasion resistance are usually associated with stabilizing treatments. One or more of these adverse effects are a result of any of the methods used. Perhaps the biggest drawback to stabilizing wood is cost. The first three approaches listed above require additional materials such as chemicals, bulking agents, and formaldehyde cross-linking agents as well as additional processing. Products stabilized in any of these three ways usually have a prohibitive cost that makes them economical only in special use instances. The stabilizing of wood through a heat treatment shows promise from the economic point of view since there is usually no addition of materials that become a part of the product itself. Due to this economic advantage, heat treating of wood to achieve stability can be used in more instances. A good example of this is the tempering of hardboard with heat and oil to obtain stability.

Particle board has certain material variables that can be adjusted to obtain a limited amount of dimensional stability. Picking a species with low radial and tangential shrinkage factors can reduce excessive thickness swelling (4). Increasing the resin content will give stability, but only at prohibitive cost since the glue cost is approximately 60% of the total material cost. Wax emulsions can reduce water absorption and swelling, but because they affect gluability their use is limited.

Since a heat treatment appears the most promising to produce any high degree of stability at a reasonable cost, the following discussions will be restricted to efforts toward stabilizing wood with heat.

Heat Stabilization of Solid Wood and Veneer

An early attempt by Seborg, Millett, and Stamm (8) to stabilize solid wood (Staypack) involved compressing and heating. Wood is compressed under pressure and heat

sufficient to cause plasticizing of the wood. It is believed that a flow of lignin causes a plastic yielding and relaxation of internal stresses from compression (8, 14). This relaxation of stresses eliminates the springback effect encountered in the usual densified wood. Since lignin is a thermoplastic material, the panels must be cooled under pressure to prevent springback when the pressure is released (8, 14). Recovery from compression can be reduced by increasing the temperature or length of exposure to pressing temperature (14). Moisture content of the wood also has an effect. Higher moisture contents increase plasticity of wood and reduce recovery from compression (14).

Samples are heated to temperatures around 300°F, but not for a length of time sufficient to cause dimensional stabilization comparable to that of heat stabilized wood. Staypack swells as much as the original uncompressed wood, but with considerably less springback from compression. The Staypack process results in wood that is appreciably darkened in color. This is probably a result of some chemical change in the lignin under flow conditions (8). The color change is very sensitive and specimens arranged in increasing order of darkness are also in the order of increasing stability (8).

Stamm, Burr, and Kline (10) stabilized uncompressed wood (Staybwood) by heat alone. Temperatures from 100°C

to 320°C for periods from 15 minutes to 7 hours were used. Specimens were heated in the presence of air as well as other gases and in a molten lead-tin-cadmium alloy. The nature of the gas had little effect on the resulting hygroscopicity. An oxidizing atmosphere was only slightly more effective than a reducing atmosphere. Wood was heated in the alloy to minimize oxidation and make possible rapid heat transfer.

The net effect of the heat treatment on uncompressed wood was reduced hygroscopicity and thickness swelling. This was permanent for three humidification and dehumidification cycles tested as well as three 24-hour soak tests. The light softwoods used were darkened to about the color of walnut when heated to temperatures and for periods sufficient to give an appreciable stabilizing effect.

Strength properties were affected by heat treating due to a loss of wood substance through thermal decomposition. In the Staybwood experiment (10), the modulus of elasticity was reduced, but to a lesser degree than the modulus of rupture. The toughness and abrasion resistance was one-third to one-half that of the original untreated wood when a 40% reduction in equilibrium swelling was attained (9). There was also a reduction in hardness and embrittlement of the fibers (10, 11, 13).

When wood has been heat treated sufficiently to attain a 40% reduction in thickness swelling, it has been noted by Stamm (14) that there is no weight loss as a

result of decay organisms in decay resistance tests. This resistance results largely from the inertness of the material rather than a toxic ingredient. It is possibly due to the reduced hygroscopicity, whereby sufficient water is prevented from entering the cell wall structure to support decay organisms, and or to some chemical change (10).

Initial thermal decomposition of wood takes place in the following order of priority: hemicelluloses, alphacelluloses, cellulose, and lignin (7, 14). Accompanying a weight loss of dry wood is a reduction in hygroscopicity. The dimensional stabilization that takes place as a result of reduced hygroscopicity from heat treating was at one time believed to result from the formation of ether linkages between adjacent cellulose chains due to loss of water of constitution (11). This has since been discredited on the basis that wood so heated swells more in dilute alkali and pyridine than unheated wood. If cross bonds were formed, the swelling should be reduced in all swelling agents that cannot break the ether linkages (11, 14). The alternate explanation offered is that the more hygroscopic hemicelluloses undergo degradation into furfural polymers of breakdown sugars which are less hygroscopic in water, but swell more than untreated wood in dilute alkali solutions and pyridine (11, 14). Some restricted decomposition of

lignin has been observed by Sandermann and Augustin (7) at relatively low temperatures. However, in the further course of heating, a condensation of the lignin takes place and thermally largely stable macromolecules are developed.

The rate of thermal decomposition is greater in the presence of air than in an inert medium. This applies primarily to wood polyoses, not to the relatively slowly oxydated cellulose (7). At temperatures ranging from 120°C to 180°C, the heat stabilizing reaction rate can be increased by using an acid salt catalyst. Stamm (11) found that wood containing 2.5% zinc chloride catalyst develops dimensional stability from the thermal reaction about 17 times faster than catalyst-free wood. Heat stabilization has been shown to reduce swelling of both solid wood and paper to about half the normal amount of untreated wood. By using heat, acid catalysts, and a formaldehyde cross-linking agent, a 90% reduction in swelling has been obtained (11).

Stamm (14) has ascertained that the reduction in thickness swelling is almost always greater than the reduction in hygroscopicity. Also, the square of the reduction in swelling is proportional to the weight loss occurring during the reaction (11, 14).

Adsorption and desorption isotherms have been determined by Kollmann and Schneider (5) for wood heat treated at temperatures from 70°C to 200°C for 6, 24,

and 48 hours. Specimens were placed in a vacuum-sorption apparatus and the average sorption values obtained. No change was noted for woods heated to 70°C, but at 100°C, 130°C, and 150°C the sorption capacity was significantly decreased. An even greater decrease was noted for specimens heated to 180°C for 6 and 24 hours, but the sorption capacity at 48 hours was greater than that at 24 hours. Kollmann and Schneider (5) concluded that a sintering of the lignin took place somewhere between 24 and 48 hours at the 180°C level that caused the increase at 48 hours. The sorption changes that occur during the heat treatment can be explained by the sorption behavior of the chemical components wood polyoses, cellulose, and lignin and their proportions in the chemical composition of wood as well as their different thermal stability. Kollmann and Fengel (6) determined that in wood heated from room temperature to 180°C for 24 and 48 hours, the most critical factor affecting deterioration was temperature. The duration of heating had a less significant effect, especially after 24 hours.

Heat Stabilization of Particle Board

Much of the discussion on the effect of heat treatments of solid wood also applies to particle board, especially that of thermal decomposition and resulting strength losses. A significant reduction in thickness swelling can be obtained by heat treating exterior

particle board at temperatures ranging from 180°C to 220°C in a post treatment after pressing. Ernst (2) found this type of a treatment to be more effective and economical than a thermal pre-treatment of material in a dip tank process.

In heat treating flakes before manufacturing of the board, Tomek (17) found the adsorption properties of the resulting urea boards reduced and thickness swelling decreased by 45 to 50%. Flakes were treated at temperatures from 240°C to 270°C for periods up to 4 minutes. An increase in bending strength of 20 to 25% was noted, but no explanation was given. Boards were pressed to a uniform thickness and an average density of .65 gms/cm³. Perhaps the strength increase was a result of replacing water lost in treating with additional material in boards made with treated flakes to the same average density as untreated boards.

El'bert (1) describes a method developed by Solecnik and Kirov in which homogeneous and three layer urea particle boards were heated in a dry stream of air to temperatures of 160°C to 180°C for up to 3 hours. The treatment resulted in reduced hygroscopicity and increased strength at the lower temperatures due to a more complete polymerization of the urea resin. At the higher temperatures, strength was reduced due to degradation of the glue. The explanation given for reduced hygroscopicity was the

ether linkage theory which has since been discredited. After thirty days of soaking, an average thickness swelling value for a three layer board treated at 180° C for 3 hours was 16.6% as compared to 40.1% for an untreated board. El'bert (1) concluded from this that boards could be made 2 to 3 times as water resistant with a 10 to 12% increase in glue cost.

Hart, Rice, and Snyder (3) also did post conditioning of a homogeneous urea particle board at a temperature of 150° F for 1, 4, and 8 days. After soaking in water for 24 hours, post conditioned samples had significantly reduced thickness swelling and decreased internal bond strength. Bending strength was not affected at this lower temperature.

A patented process by Hann of the Forst Products Laboratory (18) describes a method of steam treating particle board while the boards are restrained between platens. Steam is injected at a pressure of 30 to 100 pounds per square inch and at temperatures ranging from 275° to 340° F for 1 to 10 minutes. A relative vapor pressure of at least 90% is maintained to allow particles to adsorb heat and moisture sufficiently to cause internal swelling and promote plastic flow. The board is allowed to achieve a moisture content of at least 90% of the fiber saturation point and is then redried. Heebink (4) is currently doing extended work on a severe steam treatment for exterior particle board.

CHAPTER III

EXPERIMENTAL PROCEDURE

Design of Experiment

This experiment was designed to determine the effect of a heat treatment on the performance of a three-layer exterior particle board. Of specific interest were the effects on hygroscopicity, compressive stresses within the boards, and mechanical properties. The tests were designed to detect the effects on these three components at two temperature levels and three heating periods. The temperatures of 350°F and 425°F were selected from a temperature range suggested by Ernst (2). The heating periods of 30 minutes, 1 hour, and 2 hours were selected with the severity and economics of the treatments in mind.

To detect thickness swelling and absorption, the standard ASTM D1037-64 soak test and a special test, where swelling was observed over a period of time in a controlled climate, were used. The special test was more illustrative of the effects of a heat treatment since it more closely duplicated actual exposure of exterior particle board to environmental conditions.

Mechanical or strength properties that were of particular interest and have been tested were modulus of elasticity and internal bond strength. Internal bond specimens were tested at room conditions and after reaching equilibrium conditions at $90^{\circ}F$ and 90% relative humidity. Modulus of elasticity samples were tested dynamically at room conditions, at equilibrium conditions with $90^{\circ}F$ and 90% relative humidity, and at redried conditions. These two strength properties appeared to be of most significance in exterior applications of particle board. A total of 28 boards were made with 4 boards for each treatment and the control. For a summary of the experimental design refer to Table 1.

The smoothness or roughness of boards was of interest since in some exterior applications a finish is applied and surface roughening would degrade the finish. A test was designed to make a comparison between untreated boards and boards heated to 425° F for 2 hours. Samples were compared after exposure to room conditions, to 90° F and 90%relative humidity and then redrying, and to soaking for 24 hours then oven drying.

Manufacture of Boards

In order to make this experiment realistic, boards were manufactured that closely simulate commercial threelayer particle boards. The boards were flat pressed in a $20" \times 20"$ platen hydraulic press. The platens were heated by circulating hot oil.

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1Summary
TABLE

Board Construction											Thre	e-La	yer]	Parti	cle	Boar	ď										
Species										Ja	ck F	lne	Face	s and	l Asp	en C	ore										
Particle Type													E.	lakes													
Average Density													.7.	l gm/	cm3							ļ					
Average Thickness													1/1(6" ur	Isand	led											
Adhesive Type											Ê	teri	or-Ph	nenol	1c F	lesin											
Treatment												Oven	Hea	t Tre	atme	nt									Conti	101	
Temperature					3	350	о F										9	25°F			}	{					
Time		30 1	Min				L.			4 2	<u>د</u>			30 Mj	r.			l hr			2	hr	}				
Replications			7			~	-			17				4				7				म				=	
Tests	MOE	IB	ω	TS	MOE	IB	ω	TS	MOE	IB	S	TS	MOE	IB	s S	M S	OE	IB	S S	S MO	н ш	n v	TS	MOE	IB	S	TS
Replications	32	24	7	7	32	24	7	7	32	24	7	7	32	24	7	7	32	24	7	4 3	5	7 7	7	32	24	7	7
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MOE = Modulus of Elasticity Test (Dynamic) IB = Internal Bond Test S = Two and Twenty-four Hour Soak Test TS = Thickness Swelling Test

Flake material was obtained from a commercial manufacturer using jack pine face flakes and aspen core flakes. The face material was a fine thin flake and the core flakes were larger, thicker, and rough in general. The adhesive used was a phenol formaldehyde resin developed for use in particle boards without a catalyst. A wax emulsion was applied to limit water absorption.

A ratio of 40% face material to 60% core material was used in the construction of boards. A glue content of 12½% resin solids in the faces and 7% resin solids in the core was applied by spraying fine droplets on the flakes as they were tumbled through the air in a converted cement mixer. A $\frac{1}{2}$ % wax emulsion was applied with the adhesive. Both the adhesive and the wax emulsion were 50% solids which could be sprayed satisfactorily without diluting. The moisture content of the face flakes after spraying was 17% and 9% for the core flakes. The actual amount of material used to obtain an average board density of .71 gms/cm³ was 588 gms of face flakes and 924 gms of core flakes.

After spraying, the face flakes were weighed and divided in two equal parts; one-half was randomly deposited on the bottom caul and the other half on top of the deposited core flakes. The mat formation was accomplished by hand with care being taken to deposit the flakes evenly. The cauls were stainless steel with the bottom caul having 7/16"

stops to control the board thickness. Actual board thickness was slightly less than 7/16" due to shrinkage while pressing. A silicone solution was applied to the cauls to prevent boards from sticking to the cauls. Following formation of the mat, the boards were pre-pressed by hand, the outside frame removed, and the top caul placed on the mat.

The mat was then placed in the press, which was preheated to 370°F, and the press was closed. The average closing time ranged from 8 to 10 seconds. The pressing time was 8 minutes at 400 psi. After 6 minutes had elapsed, pressure was gradually released to allow any trapped steam to escape. The press was then opened and the board removed from the cauls and allowed to cool. Cauls were cooled in a water bath to prevent pre-curing of the next mat. Boards were allowed to condition to an equilibrium moisture content of approximately 5% after manufacturing.

Heat Treatment

Prior to heat treating the boards, the loose edges were trimmed approximately 1" in from each side. One trimming from each board was used to determine the moisture content. Moisture content before heat treatment was required to allow differentiation between weight loss due to drying and weight loss due to thermal decomposition.

Since the outside edges were used to determine the average moisture content of the boards, the estimate of actual wood substance loss was probably low.

Four boards for each treatment were placed in an oven, heated to 350°F and 425°F, in wood racks where the boards would stand vertically. A set of four boards, heated to 350°F, were removed at 30 minutes, 1 hour, and 2 hours, then the oven was pre-heated to the next temperature and the procedure repeated. The oven was a large ceramics oven with circulating air and electronic temperature controls. Immediately after removing from the oven, the boards were placed in polyethylene bags to prevent pick up of moisture until they could be weighed. Following determination of moisture content and dry substance loss, boards were allowed to condition to an equilibrium moisture content of approximately 5%.

Samples

After being trimmed, boards were 16" x 16" in size. Following heat treating and conditioning, samples were sawn from the boards for the various tests. The location of the samples for each test was designed to give a cross section from the edge of the board to the center and back to the opposite edge. Each board was given a letter for identification and each sample from within that board bore that letter and a number. Samples for the modulus of

elasticity test were 1" x 16" and 8 samples were sawn from each board. This gave a total of 32 samples for each treatment and a grand total of 224 for all treatments. Internal bond samples were 2" x 2" and 6 were sawn from each board. This yielded 24 samples per treatment and a total of 168 for all treatments. Thickness swelling and soak test samples were 4" x 4" and 1 for each test was sawn from the board. An additional 4" x 4" sample sawn was used in tests to determine surface smoothness. The position of samples within the board is indicated in Figure 1.

Measurements and Tests

Modulus of elasticity samples were tested at room conditions, at equilibrium with 90°F and 90% relative humidity, and after redrying to room conditions. A small steel staple was inserted near both ends of each sample in order that a dynamic test could be performed. Samples were then weighed to the nearest 0.1 gram, measured for width and thickness in the middle to the nearest .001", and the length recorded as a constant 16".

The dynamic test was used because it is a nondestructive test and in this instance required only 1/3 the samples necessary for a static test. It is also faster and eliminates the creep involved in a static test. The sample was placed on fine taut wire supports at the nodes near the ends of the sample. The nodes are positions of zero amplitude and damping will be at a minimum when a vibration is set up in the sample. For a

Fig. 1.--Position from which samples were sawn from boards.



formula to determine the position of the nodes refer to Appendix I. Magnetic coils were positioned directly under the steel staples, one a drive coil and the other a pickup or receiving coil. A frequency was then generated by an oscillator and amplified so the sample would vibrate at that given frequency. Every material has a resonant or fundamental frequency at which vibration will be at a The frequency was varied until the maximum maximum. vibration was found. The point of maximum vibration was determined by the receiving coil picking up the vibrations and sending a signal to an oscilloscope. The signal was displayed on the screen of the oscilloscope as a sine wave and when the resonant frequency was reached, the sine wave was of greatest amplitude. The signal may also be fed into a frequency counter from which the frequency can be read in cycles per second. This resonant frequency was then recorded and the procedure repeated for each sample. Samples were transported and kept in polyethylene bags until tested to prevent pickup or loss of moisture. The test apparatus is shown in Figure 2 and the equation for determining the modulus of elasticity from the resonant frequency is given in Appendix I.

Internal bond test specimens were tested at room conditions and at equilibrium with 90°F and 90% relative humidity. Samples were chosen alternately (1, 3, 5 or 2, 4, 6) for the test at room conditions and the remaining

Fig. 2.--Equipment used for testing dynamic modulus of elasticity with sample in place on wire supports.

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samples were used for the high humidity test. Steel 2" x 2" blocks were heated on a hot plate and specimens glued between two blocks with De Khotinsky Cement, which comes in stick form and melts when heat is applied. This is the standard test described in ASTM D1037-64. Samples glued between blocks were then cooled and tested in tension, perpendicular to the plane of the sample. till failure. Samples were weighed to the nearest 0.1 gram, measured in the middle for thickness to the nearest .001", and the length and width recorded as a constant 2". The load in pounds at failure was recorded and the internal bond strength computed in pounds per square inch. The location of the line of failure was also recorded. Density was computed for both modulus of elasticity and internal bond specimens. The Instron Testing Machine on which internal bond tests were run and the test apparatus are shown in Figure 3.

The soak test performed was the standard test listed in ASTM D1037-64. The samples were chosen alternately as in the internal bond test, and prior to the test, were measured for thickness to the nearest .001" by averaging four measurements 1" in from each corner. They were also weighed to the nearest 0.1 gram and a constant length and width of 4" recorded. Samples were placed horizontally under 1" of water and remeasured after 2 and 24 hours of soaking. They were then oven dried and weighed. The moisture content, water absorption as per cent weight and

Fig. 3.--Instron testing machine and sample at failure in test apparatus.

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per cent volume, and the thickness swelling as per cent original thickness was then calculated for 2 and 24 hours.

Thickness swelling tests were run in an Aminco Air Climate Chamber in which samples were placed at conditions of 90°F and 90% relative humidity and allowed to come to equilibrium. After reaching that point, the chamber was reset for room conditions and samples were brought back down. Before placing in the chamber and periodically during the test samples were measured for thickness and weighed the same as in the internal bond test. Samples were selected alternately as before. The samples were placed in racks vertically so air could circulate between them. Density was calculated for both soak test and thickness swelling specimens. When out of the chamber for measurement, samples were kept in polyethylene bags as before. The jig for measuring thickness in the internal bond and thickness swelling tests is shown in Figure 4.

Surface profiles of treated and untreated samples were obtained using a Proficorder. A visual comparison of smoothness or roughness was then made from graphs of the profiles. A stylus with a 3/32" diameter sapphire ball on the tip was moved across the sample for approximately 1" at a rate of .005" per second and the total profile was graphed.

Fig. 4.--Measuring jig for measuring thickness to the nearest .001". Samples were measured approximately 1" in from each corner.



CHAPTER IV

RESULTS AND DISCUSSION

Effect on Color of Samples

Exterior particle board that has been heat treated darkens appreciably with increasing severity of treatment. This is probably due to some chemical change in the lignin under flow conditions that exist at high temperatures (8). It may partially be due to a darkening of the adhesive as well. Which ever the case, it is not seen as being detrimental to possible uses of exterior particle board for reasons already stated. Samples arranged in increasing order of darkness or severity of treatment are also in the order of increasing dimensional stability, with the exception of samples heated to 350°F for 2 hours and samples heated to 425°F for 30 minutes which are very nearly the same. Figure 5 gives some idea of the discoloration that takes place. The control sample is relatively light with a purplish cast due to the adhesive used. As severity of treatment is increased, samples gradually darken and take on a brown tone. The two most severe treatments result in a dark chocolate brown colored board. The interior of the board was slightly lighter in color.

Fig. 5.--Discoloration of particle board due to heat treatments. Samples are arranged in order of increasing severity of treatment and darkening.

Control (untreated)
(anor carea)

Treated at 350°F

425°F

for



l hour

l hour

30 minutes

2 hours

1

2 hours



Strength Properties

The data from the modulus of elasticity test were analyzed and several relationships plotted and examined. In Figure 6 the average modulus of elasticity, hereafter referred to as M.O.E., for the various conditions is plotted over the length of treatment. Each point is an average of 32 samples. The effect of a heat treatment on M.O.E. at 350°F for all treating periods and at all three exposure conditions tested is very slight and of no significance. However, the effect of heat treating on M.O.E. at 425°F when tested at room conditions is significant. There is a considerable reduction in M.O.E. following heating for 1 and 2 hours. This is undoubtedly due to thermal deterioration at these severe conditions. The average dry substance loss for boards heated to 425°F for 1 and 2 hours was approximately 3% and 5% respectively.

When exposed to high humidity and tested at equilibrium conditions, the effect of heat treating on M.O.E. is very nearly the same for all samples treated and untreated. An explanation for the M.O.E. of samples heated to 350°F and samples heated to 425°F moving closer together, when exposed to high humidity, is that the less stabilized samples heated to 350°F swelled more causing a greater change in cross sectional area. With this increased cross section and resulting reduction in density, the reduction in M.O.E. was greater relative to the samples heated to

Fig. 6.--The effect of heat treatments on the modulus of elasticity of exterior particle board.

Treatment Temperature	Conditions Tested At
350°F	Room conditions
425°F	Room conditions
425°F	Redried after high R.H. exposure
350°F	Redried after high R.H. exposure
425°F	Equilibrium with 90°F and 90% R.H.
350	Equilibrium with 90°F and 90% R.H.
	Treatment Temperature 350°F 425°F 425°F 350°F 425°F 350°F 350



425°F with greater stability. Samples heated to 350°F probably have a greater capacity for bond water also, although it may be slight.

When redried to room conditions after exposure to high relative humidity, recovery of the M.O.E. of samples heated to 425°F for 1 and 2 hours is very satisfactory compared to samples heated to 350°F and untreated samples. The M.O.E. value of samples treated at 425°F for 2 hours comes very near to the original value before exposing to high humidity. This indicates that most of the reduction in M.O.E. of samples heated to 425°F for 2 hours, when exposed to high humidity, was due to the natural weakening effect of moisture on wood and springback was appreciably reduced. In samples treated at 350°F and untreated samples, more of the reduction in M.O.E. was due to reduced density with perhaps failures in areas of low density and unrecoverable springback.

Figure 7 is the regression of the average M.O.E. of untreated samples and samples treated at 425°F for 30 minutes, 1 hour, and 2 hours plotted over density. All samples were exposed to room conditions. Samples heated to 350°F were not plotted since their regression lines were nearly the same as the control. Room conditions only are shown since all regression lines retained the same slope and the effect of cycling through high humidity has already been illustrated. The regression formula

Fig. 7.--The effect of heat treatments on the regressions of M.O.E. plotted over density.

No.	Treatment Temperature	Treating Period	Conditions at Testing
1	Untreated	Untreated	Room Conditions
2	425°F	30 minutes	Room Conditions
3	425°F	l hour	Room Conditions
4	425°F	2 hours	Room Conditions
5	Line connecti	ng average densitie	es
6	Vector forces	effecting movement	t of avg. density



used can be found in Appendix II. There is no significant difference between the regression lines of untreated samples, samples treated at 425°F for 30 minutes, and samples treated at 425°F for 1 hour. However, the regression line of samples treated at 425°F for 2 hours is different enough to assume it is significant. The effect of heat treating for all treatment levels up to treating at 425°F for 2 hours appears to have the same effect as a reduction in density. That is to say that up to that point, the slope of the regression lines remains the same and with increasing severity of treatment, the regression lines and their average density slide down the same slope. The vector force shown parallel to the regression lines in Figure 7 illustrates the movement down the same M.O.E.-density line. A reduction in density will bring about a proportional reduction in M.O.E. when this vector is dominant, as is the case for the untreated samples through samples treated at 425°F for 1 hour. This being the case, boards with higher than desired density could be made to make up for density losses when heat treated. When samples are treated at 425°F for 2 hours, the regression line and average density not only slides down but drops as well. The reduction in M.O.E. is not proportional to the reduction in density but greater as the magnitude of the vector causing a reduction in M.O.E. at the same density increases with severity of treatment.

The line connecting average densities begins to slope downward at this point and could be expected to follow a path indicated by the dotted line if higher temperatures or longer heating periods were used.

Figure 8 is a graph of internal bond strength over length of treatment at room conditions and at equilibrium with 90°F and 90% relative humidity. Each point is an average of 24 specimens. The effect of a heat treatment on the internal bond strength is most significant for samples heated to 425°F for 30 minutes. However, a substantial increase is also noted going from the untreated samples to samples treated at 350°F for 30 minutes. At room conditions little change is noted after heating for 30 minutes to 350°F, but an increase becomes more magnified after exposing to high humidity. After heating for 30 minutes to 425°F, there is a marked reduction in internal bond strength which levels off after 1 hour of heating, but is magnified as a gradual increase when exposed to high humidity. The gradual increase for samples heated to $350^{\circ}F$ and the marked increase for samples heated to $425^{\circ}F$ for 30 minutes is a time-temperature relationship involving additional curing of the adhesive. After heating for 30 minutes to 425°F, all curing has probably taken place and thermal deterioration begins to offset increased strength as a result of this additional curing. In all treatments at each exposure the strength values were higher than the

Fig. 8.--The effect of heat treatments on internal bond strength when plotted over length of treatment.

No.	Treating Temperature	Conditions at Test
1	350°F	Room Conditions
2	425°F	Room Conditions
3	350°F	Equilibrium with 90°F and 90% R.H.
4	425°F	Equilibrium with 90°F and 90% R.H.



untreated value at that exposure. Even after exposure to high humidity, samples treated at 425°F for 30 minutes and samples treated at 350°F for 2 hours had a higher internal bond strength than the untreated control samples at room conditions.

An attempt was made to relate internal bond strength to average board density, but little correlation existed. This is because the average board density varies a great deal from the actual core density to which internal bond is more closely related. The failure in testing always occurred in the center of the core although the line of failure was sometimes irregular. Figure 9 shows a specimen at failure.

Thickness Swelling and Hygroscopicity

Figures 10 and 11 are the moisture content and water absorption (per cent of original weight) respectively of samples soaked for 2 and 24 hours plotted over length of treatment. They are nearly identical since they were determined from the same data, but moisture content is based on the oven dry weight where water absorption is based on the original weight at room conditions. In Figure 10 the moisture content at room conditions is given before emersion in water, which averaged from 4 to 5%. Each point is an average of 4 samples. Samples treated at 350°F for 2 hours show a gradual decrease in moisture content and water absorption. However, this probable

Fig. 9.--Irregular centerline failure in the core of an internal bond test specimen.

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Fig. 10.--The effect of heat treatments on the moisture content of samples soaked for 2 and 24 hours when plotted over length of treatment.

No.	Treating Temperature	Conditions at Test
1	425°F	After 24 hours of soaking
2	350°F	After 24 hours of soaking
3	425°F	After 2 hours of soaking
4	350°F	After 2 hours of soaking
5	350°F	Room Conditions
6	425°F	Room Conditions



Fig. 11.--The effect of heat treatments on water absorption (per cent of original weight) after 2 and 24 hours of soaking when plotted over length of treatment.

No.	Treating Temperature	Conditions at Testing
1	425°F	After 24 hours of soaking
2	350°F	After 24 hours of soaking
3	425°F	After 2 hours of soaking
4	350°F	After 2 hours of soaking



reduction in hygroscopicity is very slight and can not be considered as significant. There is also a slight reduction for samples heated to 425°F for 30 minutes, but those samples heated to 425°F for 1 and 2 hours show a marked increase of about 5% higher than the untreated control samples. After soaking for 24 hours, the decrease in moisture content and water absorption for samples treated at 350°F is more significant as is the decrease for samples heated to 425°F for 30 minutes. However, samples heated for 1 and 2 hours to 425°F. again show an increase, although all samples treated have lower moisture contents and less water absorption than the untreated samples after soaking for 24 hours. A possible explanation of this relationship is that there is a slight reduction in hygroscopicity of boards heat treated, but when the severity of treatment becomes such that an appreciable amount of dry substance loss and resulting reduction in density occurs, such as in samples heated to 425°F for 1 and 2 hours, the capacity for free water is increased more than the reduction in capacity for bond water. Therefore, an increase in moisture content and water absorption is noted at more severe temperatures and long periods of treatment.

Figures 12 and 13 are the thickness swelling (per cent of original thickness) and water absorption (per cent of original volume) respectively over the length of treatment for samples soaked for 2 and 24 hours. These graphs

Fig. 12.--The effect of heat treatments on thickness swelling (per cent of original thickness) after soaking for 2 and 24 hours when plotted over length of treatment.

No.	Treating Temperature	Conditions at Testing
1	350°F	After soaking for 24 hours
2	425°F	After soaking for 24 hours
3	425°F	After soaking for 2 hours
4	350°F	After soaking for 2 hours



TIME (hours)

Fig. 13.--The effect of heat treatments on water absorption (per cent of original volume) after soaking for 2 and 24 hours when plotted over length of treatment.

No.	Treating Temperature	Conditions at Testing	
1	350°F	Soaked for 24 hours	
2	425°F	Soaked for 24 hours	
3	425°F	Soaked for 2 hours	
4	350°F	Soaked for 2 hours	



are similar since they were determined from the same data. Although there is no significant difference between the thickness swelling or water absorption of samples treated at 350°F and samples treated at 425°F after 2 hours of soaking, values for all samples treated are below the value for untreated samples. However, soaking for 24 hours does not result in the same amount of thickness swelling or water absorption (per cent of original volume) although all samples have roughly the same amount water absorption (per cent of original weight). Thickness swelling is reduced from 13% for the untreated samples to 7% for boards heated to 425°F for 2 hours. This is about a 46% reduction. This agrees with Stamm's (14) findings that the reduction in thickness swelling is always greater than the reduction in hygroscopicity. Indications are that the reduction in thickness swelling is due primarily to a relaxation of compressive stresses and only slightly to reduced hygroscopicity, as will be borne out further in tests exposing samples to high humidity.

Figure 14 is a graph of the weight increase (per cent of original weight at room conditions) over length of treatment when exposed to high humidity. Samples treated at 350°F and at 425°F, when tested at equilibrium conditions with 90°F and 90% relative humidity, show no significant reduction in weight increase or no marked difference from each other or the untreated control value. There is only about a 2% range in observations which are an average of 4

Fig. 14.--The effect of heat treatments on the weight increase (per cent of original weight) of samples exposed to high humidity and redried to within 1% of the original weight when plotted over length of treatment.

No.	Treating Temperature	Conditions at Testing
1	425°F	Equilibrium with 90°F and 90% R.H.
2	350°F	Equilibrium with 90°F and 90% R.H.
3	350°F	Redried to within 1% of original weight
4	425°F	Redried to within 1% of original weight



samples. This indicates again, as did the soak test, that there is no significant reduction in hygroscopicity at these temperatures or treating periods. There are also no significant differences between treatments when redried to within 1% of the original weight.

Figure 15 is the thickness swelling (per cent of original thickness) over the length of treatment when brought to equilibrium conditions with 90°F and 90% relative humidity and redried to within 1% of the original weight. The results obtained from exposing samples to high humidity are similar to those found in the soak test. Although the samples picked up roughly the same amount of moisture, it did not result in an equal amount of swelling. When brought to equilibrium with 90°F and 90% relative humidity the untreated samples swelled 10.4% as opposed to 6% for the samples treated at 425°F for 2 hours. This is a reduction in thickness swelling of 42% due to a relaxation of compressive stresses. When redried, the untreated samples had a residual swelling of 2.7% and the samples treated at 425° F for 2 hours only .25%. If the samples had been redried to exactly the original weight there probably would have been no residual swelling or springback in samples heated to 425°F for 2 hours. The reduction in springback for samples treated at 425°F for 2 hours is 91%.

The thickness swelling (per cent of original thickness at room conditions) over the weight increase (per cent of
Fig. 15.--The effect of heat treatments on the thickness swelling (per cent of original thickness) of samples exposed to high relative humidity and redried to within 1% of the original weight when plotted over length of treatment.

No.	Treating Temperature	Conditions at Testing
1	350°F	Equilibrium with 90°F and 90% R.H.
2	425°F	Equilibrium with 90°F and 90% R.H.
3	350° F	Redried to within 1% of original weight
4	425°F	Redried to within 1% of original weight



original weight at room conditions) is plotted in Figure 16. Samples are brought from room conditions up to equilibrium with 90° F and 90% relative humidity and back down to within 1% of the original weight at room conditions. In Figure 16 the thickness swelling--weight increase lines are extended to the axis by extrapolation as if samples had been redried to exactly the original weight at room conditions. Each line represents an average of 4 samples and starts from zero. Samples treated at 350°F for 30 minutes, 1 hour, and 2 hours as well as samples treated at 425°F for 30 minutes are indicated by long dotted lines because there are no significant differences between them and the untreated samples. However, as Figure 16 clearly shows, the most significant reductions in thickness swelling and weight increase combined come with heating samples to 425°F for periods of 1 and 2 hours. When samples are redried, the thickness swelling--weight increase lines do not follow the same path they took when being brought up to equilibrium conditions with 90°F and 90% relative humidity due to a moisture content gradient. When being brought up to equilibrium with 90°F and 90% relative humidity, most of the weight increase will take place in the less dense core of the samples as will most of thickness swelling. When equilibrium is reached, the moisture content should be the same in the faces and the core. Upon redrying, water will be lost more rapidly from the less dense core

Fig. 16.--The effect of heat treatments on thickness swelling (per cent of original thickness) and weight increase (per cent of original weight) combined when exposed to high humidity till equilibrium is reached and redried to within 1% of the original weight at room conditions.

No.	Treatment Temperature	Length of Treatment
1	Untreated	Untreated
2	350°F	30 minutes
3	350° F	l hour
4	425°F	30 minutes
5	350°F	2 hours
6	425°F	l hour
7	425°F	2 hours



resulting in a lower moisture content and more shrinkage there than in the faces. This moisture content gradient is responsible for the looping or hysteresis effect. Samples treated at 425°F for 2 hours come back to their original thickness for all practical purposes when redrying is extended to the exact original weight.

The moisture content of samples at room conditions was approximately 4% and increased to an average of 18-18.5% at equilibrium with 90°F and 90% relative humidity. They were redried to an average moisture content of approximately 5%. All samples were within approximately 1% of each other when at equilibrium conditions with 90°F and 90% relative humidity.

Surface Smoothness

Figure 17 shows graphs of surface profiles of untreated samples and samples treated at 425°F for 2 hours under room conditions, exposed to 90°F and 90% relative humidity till equilibrium then redried to room conditions, and soaked for 24 hours then oven dried. Only the two extremes are used so that comparison of any differences due to a heat treatment when tested under various exposures would be clearly brought out. The profiles taken are total profiles that consist of the wavy reference surface of the unsanded boards as well as any roughness caused by swelling. The graphs are compressed on a 1/50 scale (.001" for each vertical division and .050" for each

Fig. 17.--The effect of a heat treatment on the surface profiles of treated and untreated samples after exposure to various test conditions.

No.	Treating Temperature	Length of Treatment	Conditions Exposed to Before Test
1	Untreated	Untreated	Room Conditions
2	425°F	2 hours	Room Conditions
3	Untreated	Untreated	Equilibrium with 90°F and 90% R.H. then redried
4	425°F	2 hours	Equilibrium with 90°F and 90% R.H. then redried
5	Untreated	Untreated	Soaked for 24 hours then oven dried
6	425°F	2 hours	Soaked for 24 hours then oven dried



horizontal division) to bring out differences. The areas that have been shaded are the rough areas of concern. Waviness is not important. Most of the waviness would be eliminated by sanding, but roughness would result from exposure to humidity and moisture changes. There is no clear differences between the untreated sample and the sample heated to $425^{\circ}F$ for 2 hours when exposed to room conditions, but a marked difference is noted at the high humidity exposure and for the 24 hour soak treatment. The samples treated at 425°F for 2 hours are clearly smoother with fewer bumps and most of these in the magnitude of .001" to .002". The untreated control samples have considerably more bumps and most of those with magnitudes of .001"-.004". While this test is only a comparison of 2 treatment levels at 3 exposure conditions, it appears that there is an appreciable reduction in surface roughening after heat treating. This reduction is significant enough that a difference can be detected when running a hand over the untreated samples and samples treated at 425°F for 2 hours.

CHAPTER V

CONCLUSIONS

A number of favorable performance properties can be ascribed to exterior particle board with few adverse effects by heating it to temperatures high enough and for periods long enough to cause an appreciable amount of stabilization. This experiment has verified a temperature range that is effective in doing this and has revealed what some of the more important effects of a heat treatment are.

Heating exterior particle board to temperatures below approximately 400°F will not yield an appreciable amount of dimensional stabilization without extending treating periods beyond what would be practical and economical from the standpoint of a commercial operation. There is also some ceiling temperature above which exterior particle board could not be heated because of excessive thermal deterioration and diminishing strength properties. The additional stabilization would probably be diminishing also as this ceiling temperature is approached. This temperature is probably somewhere near 500°F. A temperature range that can be effectively used is around 400°F to 450°F. As temperatures increase in this range, treating periods would have to be shortened to keep thermal

deterioration and resulting strength losses within an acceptable range.

In this study, the most favorable amount of stabilization with an acceptable amount of adverse results is obtained when exterior particle board is heated to 425°F for 2 hours. Favorable results are also obtained by heating to 425°F for 1 hour but not to as great a degree. When exterior particle board is heated to a temperature sufficient to cause a flow condition of the lignin and a plastic yielding, such as heating to 425°F for 2 hours, there is a relaxation of compressive stresses that results in reduced thickness swelling and elimination of springback. For the samples heated to 425°F for 2 hours, there is a 40 to 50% reduction in thickness swelling with complete elimination of springback. While this heating is going on, there is also thermal decomposition with resulting strength losses taking place. In the treatment mentioned above there is approximately a 12% reduction in modulus of elasticity, but this does not appear to be critical for a board with a relatively high M.O.E. as that tested. Also, most exterior applications of particle board are relatively nonstructural requiring only moderate strength values. This may well be a small price to pay for the dimensional stability obtained.

Also during the heating process, there is an additional curing of the resin taking place that will increase the internal bond strength of boards to a value greater than

the value for untreated boards. This was true for all treatments in this experiment.

As boards are heated to temperatures around 425°F, the faces, which experience the greatest amount of thermal deterioration, become appreciably darkened. This is not an undesirable effect to any significant degree since most exterior applications of particle board are such that the unfinished surface doesn't show.

There is no or very little effect on the hygroscopicity of the boards treated at 425°F for 1 and 2 hours. However, although boards absorb roughly the same amount of water, it does not result in the same amount of swelling as has already been noted. If there is some reduction in hygroscopicity, it is possibly offset by an increase in capacity for free water due to a loss in density brought about by thermal deterioration.

There is as a result of heat treating a definite reduction in surface roughening of boards exposed to extreme environmental conditions. This makes finishing of boards in exterior applications feasible without telegraphing through finishes.

Boards dimensionally stabilized to the degree described above will perform very similarly in swelling to exterior plywood and other wood based products.

In light of the results obtained and the conclusions that can be drawn from them, a heat treatment of exterior particle board seems to hold promise as a method of

dimensional stabilization. However, the research reported here is still of a basic nature and additional investigations seem necessary before this method could be accepted as a commercial process. Some suggestions as to areas of further study are:

- Further investigation and measurement of actual dry substance loss and effect on hygroscopicity,
- Measurement of the effect of a heat treatment of exterior particle board on embrittlement, toughness, and abrasion resistance,
- 3. Methods of catalyzing the reaction to increase the rate of dimensional stabilization thereby reducing the length of treating periods.

Economically and technically it appears that the heat treating of exterior particle board, in perhaps a process similar to the tempering of hardboard, at temperatures in the area of 425°F shows much promise in making particle board a more competitive product in the exterior wood based products market. APPENDICES

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APPENDIX I

Equation for Positioning Nodes in Dynamic Testing

When working with the fundamental frequency a constant .2242 x L (length of beam in inches) will give the position of the nodes from each end of the beam.

Equation for Dynamic M.O.E. from the Resonant or Fundamental Frequency

$$E = \frac{fr^2 l^3 w}{h^3 b} 5.4 \times 10^{-6}$$

where	Ε	Ξ	dynamic M.O.E. (psi)
	fr	=	fundamental frequency (cycles/second)
	1	=	length of sample (inches)
	h	=	thickness of sample (inches)
	Ъ	=	width of sample (inches)
	w	=	weight of sample (grams)

APPENDIX II

Linear Regression Formulas

Let Y be the dependent variable and x be the independent variable. From the data obtain the following statistics:

$$\sum_{i}^{\sum X_{i}}$$

$$\sum_{i}^{\sum Y_{i}}$$

$$\sum_{i}^{\sum X_{i}}$$
N

From the statistics the following are obtained:

$$m = \frac{N\Sigma X_{i}Y_{i} - \Sigma X_{i}\Sigma Y_{i}}{N\Sigma X_{i}^{2} - (\Sigma X_{i})^{2}}$$
$$b = \frac{\Sigma Y_{i}\Sigma X_{i}^{2} - \Sigma X_{i}\Sigma X_{i}Y_{i}}{N\Sigma X_{i}^{2} - (\Sigma X_{i})^{2}}$$

The regression equation of Y on X is

$$Y = mx + b,$$

where (m) is the slope and (b) is the Y intercept.

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