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Report Summarizing Work Done With

"SYNTHETIC RESINS"

F 407

Oregon State College

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by

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References

1. "Synthetic Resins and Their Plastics" by Carleton Ellis
   The Chemical Catalog Company Inc., N.Y.
2. "The Chemistry of the Natural and Synthetic Resins" by
   T. Hedley Barry, Alan A. Drummond, and R. S. Morrell
   D. Van Nostrand Company, N.Y.
3. "Chemical Utilization of Wood" by Henry K. Benson
   U.S. Department of Commerce, U. S. Government Printing Office.
4. "Synthetic Adhesives" by Paul I. Smith
   Chemical Publishing Co., Inc. Brooklyn, N.Y.
5. "Modern Wood Adhesives" by Tomas D. Perry
   Pitman Publishing Corporation, New York and Chicago.
6. "Synthetic Resins Glues", Forest Products Laboratory
   Bulletin No. 1336
7. "Commercial Timbers of the United States" by H. P. Brown
   and A. J. Panshin, Mc Graw-Hill Book Co. Inc., NewyYork
   and London.
8. "Synthetic Resins" by the Resinous Products and Chemicals
   Co. of Philadelphia Pa.
9. Plastics Catalog----The 1944 Encyclopedia of Plastics,
   Plastics Catolog Corporation, New York, N.Y.

Objective:

The objective of this course was to give the student a basic working knowledge of synthetic resins. It was to be accomplished by the student's working on the problem of impregnating wood fibres with synthetic resins so as to make possible the manufacture of a good hardboard product using a one-pressing technique.

Procedure:

The student was to first read all the available material dealing with synthetic resins in order to gather the background material necessary to working out the problem. A list of these references accompanies this report.

Then, having gathered the necessary background material, the student was to work with the available synthetic resins using his own method of attack of the problem at hand. This work was done in the Forest Products Laboratory on the Oregon State College campus in Corvallis, Ore.

Results:

In view of the fact that the field of synthetic resins is so broad it was thought advisable to narrow the scope of the problem by working almost entirely with the phenol-formaldehyde synthetic resins. In the same vein, and since it was most readily available, the fibre used was for the greater part Allis Chalmers shredded Douglas-fir fibre.

By way of background material a synthetic resin may be defined as a complex amorphous organic semi-solid or

solid material, usually a mixture of substances; built up by chemical reaction and approximating the natural resins in various physical properties; namely, lustre, fracture, comparative brittleness at ordinary temperatures, insolubility in water and fusibility or plasticity when heated or exposed to heat and pressure but commonly deviating widely from natural resins in chemical consitution and behavior with reagents.(1) While it is without the scope of this report to describe the chemical formulation of the phenol-formaldehyde synthetic resins, a glimpse at the process for manufacturing these resins is helpful in understanding the subsequent behavior of the resins. One method of forming a phenol-aldehyde resin consists essentially in mixing about equal volumes of commercial formaldehyde solution with phenol. On heating, with or without the addition of other chemicals that may modify the product, a reaction takes place and a heavy viscous layer separates from an aqueous solution. The aqueous layer is discarded and the syrupy layer, which constitutes the resin, is soluble in alcohol or acetone---Further action of heat and pressure will convert the mass into a hard, insoluble substance. The specific properties of the resin are subject to control by modifications in the process. For example, by suitable modifications an intermediate product may be obtained that can be powdered and suspended in water. (6) It is apparent that the condensation and polymerization of resins come mainly through the influence of heat. These terms refer to the cure or hardening of the resin that is essential to making a bond between two pieces

of wood.(5)

The basic problem at hand was the impregnation of the wood fibre with the synthetic resin. It seemed logical that if the fibre could be impregnated with the resin, the application of heat and pressure to the mass of impregnated fibres would set up the resin within and without the fibres thus making a hard, insoluble, water resistant, strong homogenous product. Thus the manufacture of the hard-board could be accomplished in a one-pressing operation. It was thought that this would be much superior from many stand points to the two-press procedure of first pressing the fibre preform and then again pressing it after soaking the preform in the desired resin.

The actual impregnating of the wood fibre was a matter of getting the resin through the various pits in the cell wall into the cell cavity. This is probably done by straight conduction and by osmosis. It seemed logical in this connection that those resins with low molecular weights could be most expediently used. It was decided to try both water and alcohol soluble resins although special emphases was to placed on water soluble resins. The cost of alcohol nearly prohibits the use of alcohol resins in actual production.

The matter of removing the water and volatiles was another thing to be considered. It was decided that this might be done either prior to the pressing of the preform or in conjunction with the pressing.

The matter of preforming was open to question. Using the liquid resins it seemed to be clearly a matter of wet preforming. Variations in wet preforming methodology were considered and attempted. It was thought too that some time could profitably be spent regarding the matter of dry preforming using powdered resins.

With the desired results clearly in mind the student set to work to attain these results. There follows a discussion of the experimental work done and the results attained.

First we shall consider the work done with the possibility of dry preforming. While this was really outside the scope of the original problem it was thought that some effort could be spent here to look into the possibilities if nothing else. The dry preforming setup used consisted essentially of a recirculating blower type fan. The system was made up of stove pipes leading from the fan around several corners and into a preform box with a screen at the bottom, made easily removable by a built in drawer. The pipe then lead out from the preform box back to the blower. Between the box and the blower an opening was left for feeding the fibre into the circuit. Essentially the setup worked fairly well, the big problem arising being that of getting an even distribution of the fibre over the screen to make a preform of regular thickness. Accomplishing this was clearly a matter of adjusting the air currents through the system so that an equal air pressure was exerted on the entire surface of the screen. Several types of wooden air deflector fins

were made and installed in the circuit. These showed that with the proper kind of a fin, the air currents could be controlled to give the even pressure desired. The fins made of wood however offered too much resistance to the air flow and caused the circulating fibre to pile up. The necessary materials for building the right type of metal fins were not available so this problem was discontinued. The work done did indicate that such an arrangement could be made which would work very satisfactorily. It would be highly desirable to perfect the dry preform method in order to eliminate the problem of removing the water incurred in wet preforming methods.

The problem of impregnating the wood fibre with various types of synthetic resins could not be approached in the most desirable way. The ideal approach would have been to decide upon the desired specifications of the synthetic resin to be used and secure a resin with those specifications. Due to the shortage of liquid synthetic resins, however, the approach had to be changed to a matter of using the available resins in a hit-or-miss manner. The resins used were samples secured from the various synthetic resin manufacturers. Very little information regarding the make-up of the resin was supplied.

It was decided that the most pertinent information to be obtained about the use of the various resins in this hard-board manufacture would be the resulting swelling and water absorption of the hard-board samples made with the

various resins. The strength figures would be of interest but since the proper testing machines were not available, it was decided to eliminate any tests of that type.

Following the completion of each individual hard-board sample, the general appearance was noted, carefully watching for any blisters, noting the apparent resin penetration etc. The dry sample was then weighed and the thickness measured at a spot which was picked as being of average thickness. The samples were then soaked in tap water for twenty four hours. Following this the samples were again inspected visually, weighed, and measured. The percent swelling figure was arrived at as shown in the formula:

$$\% \text{ Swelling} = \frac{\text{wet thickness - dry thickness}}{\text{dry thickness}} \times 100$$

The percent water absorption was calculated from the formula:

$$\% \text{ Water absorption} = \frac{\text{wet weight - dry weight}}{\text{dry weight}} \times 100$$

In the way of background information several samples of hard-board were made using no resin whatever. The wet preform was made merely by pouring a mixture of water and fibre into the wet preform box. The water was drained off allowing the fibre to collect on the screen. This method, a tried and true method of wet preforming, proved very satisfactory as regards an even preform. The preform so made was then pressed for twenty minutes under 500 psi. (pounds per square inch) at 135° C. The samples were then evaluated. The finish was very good but naturally the swelling

and absorption values were very high showing about 112% swelling and 132% water absorption.

Resinox 468

Resinox 468 is manufactured by the Monsanto Chemical Company, plastics Division, Springfield, Mass. It is a clear, reddish-brown liquid with a solids content of 70 to 74% in water and a pH of 7.6 to 7.8.

Several hard-board samples were made using this resin.

1) A quantity of Douglas-fir fibre was soaked in a solution of about 50% Resinox 468 for 24 hours in laboratory air. The sample was wet preformed with water. It was then pressed for 15 minutes at 150° C. under 350 psi. The resulting sample had an excellent finish and showed absorption of 35.8% and swelling of 10%. The sample was .110 inches thick when dry.

2) The fibres were soaked in a solution of about 25% Resinox for 24 hours. The sample was preformed wet and pressed at 146° C. for 15 minutes under 300 psi. The finish was excellent, absorption 59.6% and swelling 20.9%

3) The fibres were soaked in a 25% solution of Resinox 468 for 24 hours. The sample was preformed wet and pressed for 15 minutes at 140° C. under 500 psi. The resulting finish was excellent, absorption 27.3% and swelling 17%.

4) The fibres were soaked in a 25% solution of Resinox 468. The sample was preformed wet. A layer of

wood flour was then added to the surface of the preform. This was done merely to see what kind of a finish would result. The sample was handled exactly as was sample #(3) pressing it for 15 minutes at 140° C. under 500 psi. The resulting finish was very smooth and nice but the absorption figures were higher since the wood flour was not resin impregnated. The absorption was 37% and the swelling was 24.8%.

From this work done with Resinox 468 it was apparent that a good product could be obtained. The absorption and swelling figures were a little high. This was probably due mainly to the fact that the impregnation was not complete and also to the fact that void spaced probably existed between the fibres. This latter assumption was born out by noticing the lower swelling and absorption values obtained under greater pressure. This would indicate that the greater pressure eliminated many of the void spaces. The resin was easily handled and proved quite satisfactory.

Lauxite Impreg Resin 442

This resin is manufactured by the I. F. Laucks Co. Inc., of Seattle, Wn. The resin is a reactive phenol-formaldehyde suitable for impregnating of veneers without dilution. It is 60% solids in alcohol. A very small quantity of this resin was available so it was possible to make only one sample.

1) The fibres were soaked in 100% resin for 24 hours. The fibre was then washed in alcohol to remove the surplus resin. The sample was pressed for 15 minutes at 150° C. under 350 psi. The resulting sample had a good

finish. The water absorption was 34.9% and the swelling was 4.62%.

This resin showed good possibilities but little could be concluded from the one trial. The resin however was alcohol soluble raising the cost of its use considerably.

Cascophen BM-306A

This resin is manufactured by the Casein Co. of America, Seattle, Wn. The resin is a thermosetting phenol-formaldehyde. It is water soluble and comes in a solution of 50% solids. The resin was submitted as a low cost, recently developed resin about which little was known.

1) The fibre was soaked for 5 days in a solution of about 25% Cascophen BM-306A. The sample was preformed wet and pressed for 15 minutes at 140° C. under 500 psi. The resulting product was not good at all. The finish was poor, the absorption was 151% and the swelling was 142%.

2) The fibre was soaked as above for 5 days in the 25% resin solution. The sample was preformed wet and then baked in an oven for 75 minutes at 95° C. in order to attempt to remove the volatiles. The preform was then pressed for 15 minutes at 150° C. under 500 psi. The resulting product was little better than #(1). The finish was slightly better, the absorption was 118% and the swelling 24.4%.

3) As above the fibre was soaked for 5 days in the resin solution. The sample was preformed wet and the preform was baked under an infra-red lamp for 120 minutes. The preform was then pressed for 15 minutes at 155° C. under 500 psi.

The resulting hard-board sample was better than the two previous samples but was still far from good. The finish was fair while the absorption was 74% and the swelling was 18%.

The work with this resin showed rather conclusively that the resin cannot be used very successfully in this type of work.

The work done in conjunction with this resin however did prove of some importance. It was shown that by baking the preform under a lamp or in an oven, the volatiles can be removed, thus decreasing the possibility of void space in the board and decreasing the absorption and swelling figures.

In conjunction with this resin too, some work was done regarding the moisture content of the fibre used and its possible influence on the degree of impregnation attained. The moisture content of the fibre being used was found to be 16%. A duplicate of sample #(1) was made using the oven dry fibre to see if it would have any affect on the results obtained. The results were just the same as with the fibre of 16% moisture content however so it was concluded that within this range at least, the moisture content of the fibre is not critical.

Cascophen IV-80

This resin is also manufactured by the Casein Co. of America. Cascophen IV-80 is a phenolic impregnating varnish in alcohol solution. It is characterized by high

solids content (79% to 81%) and exceptional penetrating power. It may be diluted with either alcohol or water. Its specific gravity is 1.175 and the pH is from 7.5 to 8.0.

Again in the case of this resin only a small quantity was available, so it was impossible to soak any fibre in the resin.

1) A preform was made of unimpregnated fibre. This preform was then coated with wood flour and pressed under 500 psi at 150°C. for 15 minutes. The board was then soaked for 20 minutes in 100% Cascophen IV-80 and pressed at 138° C. under 600 psi for 20 minutes after baking the preform in the oven for 20 minutes at 95° C. The volatiles were evidently not removed and the sample blistered quite badly. The absorption value of 14.8% and swelling of 7.4% were fairly good however.

This resin looked as if it had definite possibilities but they could not be explored because of the insufficient quantity of the resin available.

Compregnite

This is another product of the Casein Co. of America. It is a water solution phenol-formaldehyde resin of approximately 52% solids and made especially for impregnating purposes. Several trials were made using this resin.

1) The fibre was soaked in 100% Compregnite for 5 days. A wet preform was then made after which the preform was baked under an infra-red lamp for three and

one-half hours. During this baking the preform was placed over the blower fan which drew the warm air through the preform. The preform was pressed for 30 minutes under 500 psi. with the temperature varying between 165 and 180° C. The resulting sample was slightly blistered but the absorption and swelling values were excellent being 6% and 0% respectively.

2) The fibre as above was soaked in 100% Compregnite for 5 days. The preform was made wet using the same resin wash water as was drained off of #(1). The preform was then allowed to stand in the laboratory air for 120 hours to allow the volatiles to escape. The preform was then pressed at 180° C. under 500 psi. with bleeding each 30 seconds for the first 5 minutes to allow the volatiles to escape. After they had escaped the sample was pressed for 50 minutes at the same temperature and pressure. The bleeding was merely a matter of releasing the pressure each 30 seconds momentarily to allow the volatiles to rush out. This resulting sample seemed excellent. The finish was perfectly smooth and nice looking. The absorption was 7.6% while the swelling was 0.0%.

3) In this case the fibre was soaked for 3 days in a 50% solution of Compregnite resin and water. The sample was preformed wet using resin water. It was then pressed for 50 minutes under 500 psi. at temperatures ranging from 180° to 200° C. with bleeding for the first few minutes until the volatiles had escaped. The resulting sample had an excellent finish. The absorption value was 14% while the swelling was 5%.

4) In this sample fibre was used which had soaked for 7 days in 50% Compregnite resin and water. It was preformed wet with resin water. An unusually thick sample was made (0.203 inches dry) to provide a maximum of difficulty for the escaping volatiles. The preform was pressed as before under 500 psi. at 160° C. for 50 minutes with bleeding for the first few minutes. The resulting finish was again excellent. The swelling was 11.3% while the absorption was 17.6%.

5) The above procedure was followed to provide a check on the results. In this case the finish was excellent as before, the absorption was 16.9% and the swelling 12%.

From the work done with this resin, it was found that excellent results could be obtained from its use. It seemed to do a better job of impregnating the fibre. The results were really very good. Further work along this line could be done profitably with this resin to bear out the results found here.

Melmac Resin

This resin is manufactured by the American Cyanamid Co., Plastics Division, New York. This particular sample was of type S-72 from batch B-8148. Instead of being a phenol-formaldehyde as were the other resins used, this resin is a melamine as indicated by its name. Prof. Voorhies stated that this resin worked very well in coating unimpregnated preforms so it was decided to try to impregnate the fibre with the resin.

1) Fibre was soaked for 2 days in 100% Melmac Resin. A wet preform was then made using water. The preform was then put into the press and baked under no pressure while the press heated from 138° to 180° C. The preform was then pressed under 500 psi. for 45 minutes with the temperature varying from 160° to 180° C. The preform was bled for the first few minutes to remove any volatiles not previously removed by the baking. The finish was good while the absorption value was 36% and the swelling 7.1%.

2) For this sample the fibre was soaked 9 days in a 50% solution of melmac resin and water. A wet preform was made using resin water. The preform was pressed at 500 psi. at 140° C. for 20 minutes with continual bleeding. After all of the volatiles had escaped the sample was pressed for 40 additional minutes with the pressure remaining at 500 psi. and the temperature ranging from 140° to 180° C. The finish of the sample was good but it didn't look as if the resin was getting into the fibre very well. The absorption was 29% and the swelling 16%.

3) The fibre was soaked 7 days in a 50% solution of resin and water. A wet preform was made with melmac resin water. The preform was then pressed for 45 minutes under 500 psi. at temperatures ranging from 160 to 200° C. Bleeding was allowed for the first fifteen minutes. Again the finish was good but it didn't appear that the resin had thoroughly impregnated the fibre. The absorption figure was 21.8% while the swelling was 7.33%

This resin as shown by the work of Professor Voorhies is a good resin but it does not seem adaptable to the work of impregnating wood fibre.

CONCLUSIONS:

From the work done it can be concluded that wood fibre can be impregnated with resin thus making the manufacture of hard-board possible using a one-press operation. This work showed rather conclusively that it can be done. Of course the proper resin must be used. It is impossible from this work to state which is the best resin for the purpose since so few were tried. Of those tried however, the Compregnite resin looked very good.

If the hard-board is to be manufactured using a one-pressing technique, some allowance must be made for the escape of volatiles. This can be done either prior to pressing or in conjunction with the pressing by bleeding.

Much work needs to be done before any definite conclusions can be arrived at but on the basis of this work, it can be stated that hard-board can be manufactured using impregnated wood fibre and a one-press operation.

SUGGESTIONS:

Naturally it is suggested that additional work be done on this project. Additional resins may be used to seek out their merits if any. In addition to further work along the lines of the work done here, it is suggested

that the thing be investigated from other angles.

With the equipment available for this work, it was impossible to attempt impregnating wood fibre under pressure. This definitely should be attempted. It might improve or speed up the impregnation considerably.

Preforming methods need further investigation. The wet preforming used in this work produced satisfactory preforms but introduced the problem of getting rid of the excess water. The fibres might possibly be impregnated, dried to some extent, and then the preform made dry thus eliminating the problem of water removal.

If the use of wet preforming is deemed necessary, the methods for removal of the water as well as the volatiles from the resin might be improved considerably. It was shown in this work that the infra red lamps did the work quite well. However when one looks to the mass production of this project the expense of a battery of these lamps is something to consider. The work might be done in ovens if the circulation is sufficient and the temperature low enough to prevent the polymerization of the resin. The drying in ovens would however be time consuming and expensive. From this work it appears that an efficient method of bleeding during the actual pressing operation would work out quite well. This matter needs further investigation however. It does work but the proper use of the bleeding method certainly was not determined from this work.

In conclusion it might be said that the results of

this work were gratifying if to no one but the student. The student believes that the work did produce information of some value. The problem of impregnating wood fibre to bring about the manufacture of hard-board with a one-pressing operation is not solved. Still much work leading to the solution of that problem has been done. The student had neither the available time nor materials to follow the thing to its conclusion. As a result he cannot say "In this report will be found a description of the method for manufacturing hard-board with a one-press operation using resin impregnated wood fibres." He can say however " In this report will be found preliminary work on the problem of impregnating wood fibres to bring about a one-press operation in the manufacture of hard-board, which work should when enlarged upon under more favorable conditions yield the solution to the problem."