CHARACTERISTICS OF EPOXY RESIN-KAOLINITE COMPOSITIONS

by

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Requirements for the Degree of

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May 20, 1957

Professor L. F. Hamilton
Secretary of the Faculty
Massachusetts Institute of Technology
Cambridge 39, Massachusetts

Dear Sir:

We herewith submit this thesis entitled "Characteristics of Epoxy Resin-Kaolinite Compositions" in partial fulfillment of the requirements for the degree of Bachelor of Science in Chemical Engineering from the Massachusetts Institute of Technology.

Respectfully submitted,

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The authors wish to express their sincere appreciation to Professor Alan S. Michaels for his guidance during the investigation. His criticisms and suggestions aided greatly in the experimental work.

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I. SUMMARY

The purpose of this thesis was to investigate the characteristics of highly filled epoxy resin systems. Varying amounts (twenty per cent or less the weight of the filler) of the resin were compounded with clay to yield a ceramic like material of good physical properties.

The resulting product was a hard, smooth material with a maximum tensile strength of 1320 psi. in the range of nine to twelve per cent resin. The material has a density of approximately 1.70 grams per cubic centimeter; this value is considered low. Immersion in water for 24 hours, at room temperature, decreased the tensile strength of the product on the order of fifty per cent.

An epoxy resin, Epon 828 (Shell Chemical Company) in varying percentages of the filler was the binder; kaolinite was the clay filler; dimethylaminomethylphenol, DMP-10 (Rohm and Haas) at twenty per cent of the resin was the curing agent; xylene, at fifty per cent of the resin was used to decrease the viscosity of the resin. The curing agent mixed with the cut resin was added to an agitated kaolinite-water slurry. The sludge was suction filtered and air dried; this yielded a dry powder which was compression molded at high temperature and pressure.

The product material has properties which would allow its use as a substitute for wood or ceramics. Using the powder as a base, a material can be formed that can be molded, sanded and machined. The resulting product may also

be nailed or cut to any desired shape.

Due to the relative costs of the materials the price of the product is essentially that of the resin used. The cost of the final product will be determined by the percentage of resin present; for a system containing ten per cent resin the cost of the product will be one-eleventh that of the resin.

It is recommended that further work be done to verify the results of this thesis along with obtaining the properties of systems above twenty per cent resin content. Experimentation should be done using the techniques detailed in this thesis. Further experimentation to develop methods of calendering, extruding, and molding the powder should be carried out.

II. INTRODUCTION

Inorganic materials, such as clays and carbon black, have long been used as fillers for rubbers and plastics. These fillers, used in large weight fractions of the final product do not markedly affect or decrease the physical properties; in some cases the fillers increase the final physical properties of the product and are called reinforcing fillers. As would be expected, these fillers decrease the cost of the finished material.

In the last ten years a series of epoxy resins have been developed which have found wide application in industry due to their ability to bond and produce very resistant surface coatings. Epoxy resins are strong, abrasion resistant, chemically inert and water repellant. Since they have these desirable characteristics, but are rather expensive, it would seem appropriate that research be done to investigate the possibility of blending epoxy resins with cheap fillers. In this thesis the experimental system used consisted of a clay filler, epoxy resin, and curing agent. Heat and pressure were applied in the curing step to increase the strength of the final product.

Epoxy Resin

Epoxy resin is the condensation polymer resulting from the reaction of epichlorohydrin and bisphenol in the presence of caustic soda (2),(9).

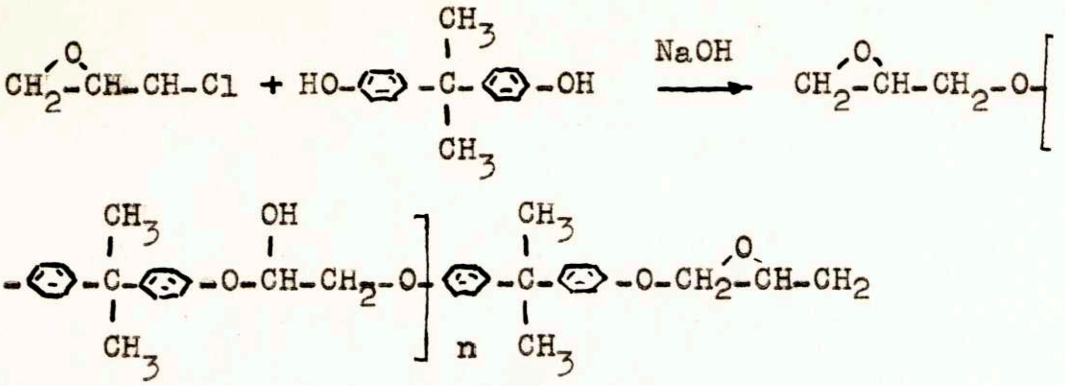


Figure 1. Epoxy Resin Synthesis

The polymer is a linear thermoplastic for which "n" varies from zero to ten giving a molecular weight of from 400 to 8000. The polymer contains only the carbon-oxygen and carbon-hydroxyl bonds besides the carbon-carbon bonds. The epoxide groups $(\overset{O}{C-C})$ and hydroxyl groups (OH) are evenly but widely separated on the linear polymer, as a result, when the polymer is cross-linked by a curing agent, the product has a flexibility not often found in thermosetting polymers.

The epoxy resin obtained from the above reaction, is an orange to yellow colored relatively useless viscous liquid. The epoxide and hydroxyl groups present in the molecular chain, however, are active and capable of further reaction. These groups are most reactive with compounds which contain active hydrogen, as acids or amines. When such a compound is added to the resin the resulting reaction product is a cross-linked thermosetting polymer with the properties stated above. This process is known as "curing".

The curing operation results in little shrinkage of the polymer leaving no internal strain in the final product, this factor and the excellant adhesive properties of the resin have been used to bond metals, wood, and plastics. A commercial resin-curing agent combination is available which produces a bond that can be machined and is superior to a good weld.

The literature refers to epoxy resins as epoxide resins, ethoxylenes, and epikote resins. The resins are produced under various trade names such as Shell's Epon Resin series.

Curing Agents

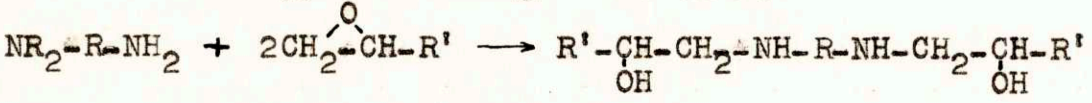
The commercial curing agents for epoxy resins, which contain active hydrogen are: polyfunctional amines, acid anhydrides, dibasic acids, pheno-, urea-, and melamine-formaldehyde resins, with secondary and tertiary amines being the most important. Although the exact curing mechanism is still not understood, it may be summarized by these steps:

I. (a). An amino group reacts with an epoxide group at room temperature.

(b). The polyamines produced from (a) couple to the epoxy resin.

II. (a). Coupling occurs at elevated temperatures with certain amines and both the hydroxyl and epoxide groups.

A typical curing reaction is:



$$NR_2-R-NH_2 + 2CH_2\overset{O}{-}CH-R' \longrightarrow R'-\underset{OH}{CH}-CH_2-NH-R-NH-CH_2-\underset{OH}{CH}-R'$$

(where R' signifies the remainder of the resin molecule)

Figure 2. Curing Reaction

The curing agent used in this investigation was DMP-10 (Rohm and Haas)- dimethylaminomethylphenol, a tertiary amine.

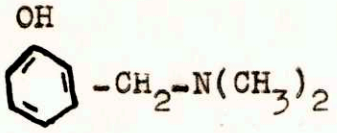


Figure 3. Curing Agent

The resulting product is a thermosetting, cross-linked polymer with such physical properties as high tensile strength, chemical, electric, water and abrasion resistance with strong adhesive power. The final product is determined by the nature and amount of curing agent, therefore by varying the amount of the curing agent a product with different properties can be produced. The curing agent was kept at a constant percentage of the resin in this investigation.

Kaolinite

Kaolinite is the name given to a family of clays having the general formula $Al_2O_3-2SiO_2-2H_2O$. It is an inorganic, cheap, crystalline substance of numerous uses.

Clay particles exist in water suspensions as small plate-like objects approximately one-tenth of a micron in width and one micron in length. Either due to adsorption or disturbances in the crystal lattice, the particle becomes negatively charged when placed in water. That is, in solution hydroxyl groups adhere to the particle and produce a resultant negative charge and polarity (3),(4). This surface effect tends to keep the particles apart, producing good dispersion in water solutions. If a sodium or magnesium salt was added to the solution the charge on the particles would become neutralized and flocculation would occur (4),(6).

Bonner (1) reported that compositions with one-tenth weight fraction of epoxy resin can be prepared and that these compositions have properties paralleling those of wood and ceramics.

The mechanism for the combination of epoxy resin and clay is as follows: assuming the clay particles to be uniformly distributed in solution the resin is added and also assumed to become uniformly distributed. The clay particles are then coated by the resin molecules, and the resin molecules are joined by curing. It is seen that an essential factor determining the strength of the final product is the bond between the resin and the clay. Water remaining in the product will adhere to both particles, water and resin, preventing them from approaching close enough to form a firm bond. Therefore, the maximum amount of water should be removed from the product before pressing.

Once the adhered water has been removed the next determining factor in the strength of the product will be the amounts of the reactants. In the case where there is either all resin or all kaolinite the product will have the properties of the material used. However, at some weight fraction between these two cases there should be a composition of maximum physical properties. At this point the amount of resin should be sufficient to cover the clay particles and form a strong network when cured.

A composition with too low a resin content will contain voids which will break the solid network and reduce the strength of the product.

Compositions with a resin content above the maximum will exhibit local spots of higher strength but will contain areas of less strength. Any force applied will follow these areas of low strength and therefore the added resin should not be greatly noticed.

Uniformity and density are critical factors in the final product, in as much as it is desired to have the particles evenly dispersed and as close as possible for maximum strength. Densities of 1.60-1.80 grams per cubic centimeter have been reported as compared to the theoretical value of 2.50 grams per cubic centimeter (1), (8). Varying the amount of curing agent should show a maximum point above which little affect is seen by adding more curing agent. In this thesis the resin was varied from twenty per cent the weight of the filler to four per cent.

The literature shows that the combination of epoxy resins with fillers is a new field and little work has been reported. Deery (2) tried using polystyrene as a binding resin with kaolinite but obtained a final product of poor strength and little practical application.

III. PROCEDURE

A general outline of the experimental method is given in this section; a detailed discussion of the experimental procedure is presented in Appendix I-A.

The experimental method outlined in this section is a direct result of the authors' work in the Soil Stabilization Laboratory, and consultation with Professor Alan S. Michaels.

A kaolinite-water slurry was produced by adding the kaolinite to a violently agitating water solution. A solution of the epoxy resin and curing agent, cut with xylene, was then slowly added to the agitating kaolinite slurry. The resulting mixture was allowed to settle, the liquid layer decanted off and the sludge suction filtered until a firm cake remained.

This cake, containing fifty per cent by weight of water, was left to air dry. After 24 to 48 hours the water content decreased to below one per cent, and the material suitable for compression molding.

The water content is determined and the powder pressed under pressure and temperature to a firm hard slab. A sample, in the shape of a dogbone, was cut from the center of each slab to be tested for tensile strength and density.

Additional samples were weighed, immersed in water for 24 hours, then reweighed in order to determine the amount of water absorbed. The wet tensile strength was also determined from these samples.

The values obtained in the experimental work are recorded in Appendix I-B. A condensation of these values also appears in the section entitled "Results."

IV. RESULTS

The results presented in this section are essentially the same as those in Appendix I-B. The average values are presented here in condensed form for easier analysis.

Table I.

The average dry tensile strength of the materials produced in the experiment:

Resin Content (% Weight Kaolinite)	Average Tensile Strength (psi.)
6	741
7	845
8	1010
9	1313
10	1304
12	1270
14	778
16	-
18	875
20	440

Table II.

The wet tensile strength is compared to the dry tensile strength:

Resin Content (% Weight Kaolinite)	Wet Tensile Strength (psi.)	Dry Tensile Strength (psi.)
4	-	-
5	200	-
6	595	741
7	596	845
8	705	1010
9	640	1313
10	625	1304
12	725	1270
14	-	778
16	-	-
18	460	875
20	-	440

Table III.

The water absorbed as a weight per cent of kaolinite for the different resin percentages and the density of the product before submersion in water:

Resin Content (% Weight Kaolinite)	Water Absorbed (% Weight Kaolinite)	Density (gms./cc.)
4	17.50	1.55
6	12.30	1.73
7	10.40	1.73
8	13.70	1.70
9	15.30	1.65
10	9.25	1.77
12	15.20	1.57
18	12.20	1.40

Figure 4

The graph is a plot of the dry tensile strength vs. resin content. It is seen to approach a peak at about 9-11% resin content.

Figure 5

Wet tensile strengths are plotted against resin content.

Figure 6

The water absorbed as a per cent of kaolinite in the sample is plotted against the resin content. There appears to be no correlation so no curve was drawn.

Figure 7

The water absorbed is plotted vs. the density of the final product. These points represent products of different resin content as taken from Table III.

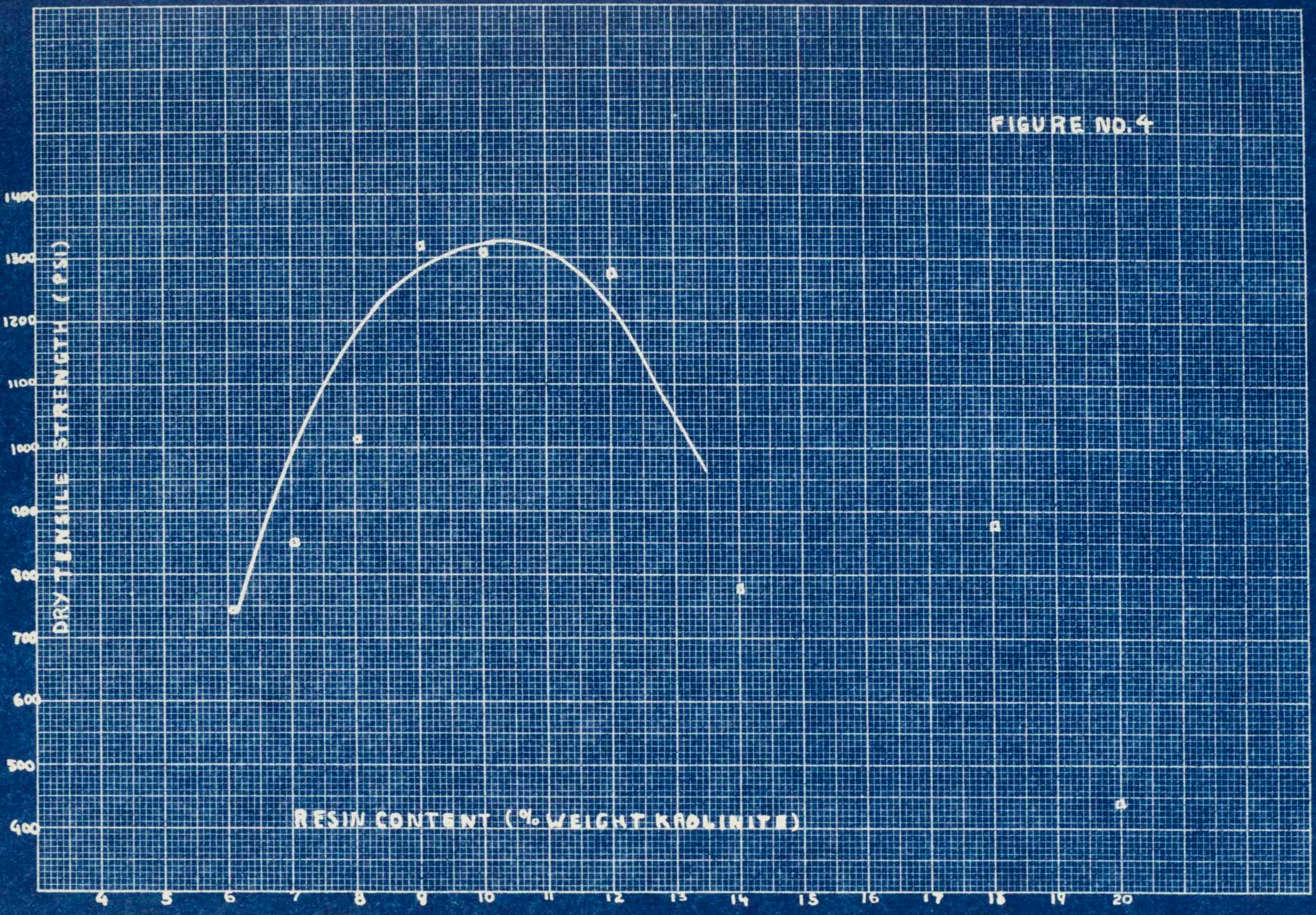


FIGURE NO. 4
DRY TENSILE STRENGTH (PSI)
1400
1300
1200
1100
1000
900
800
700
600
500
400
RESIN CONTENT (% WEIGHT KAOLINITE)
4
5
6
7
8
9
10
11
12
13
14
15
16
17
18
19
20

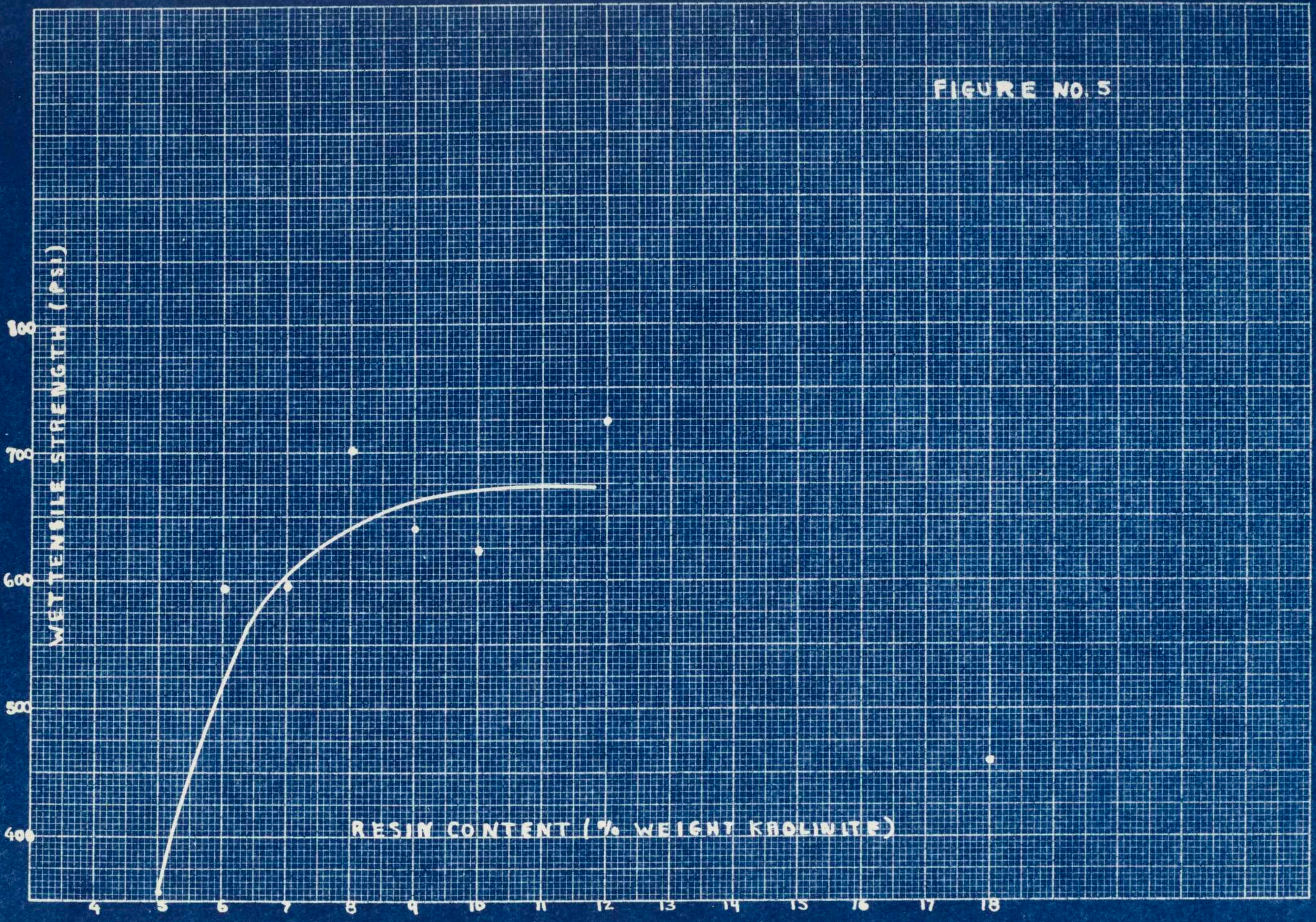
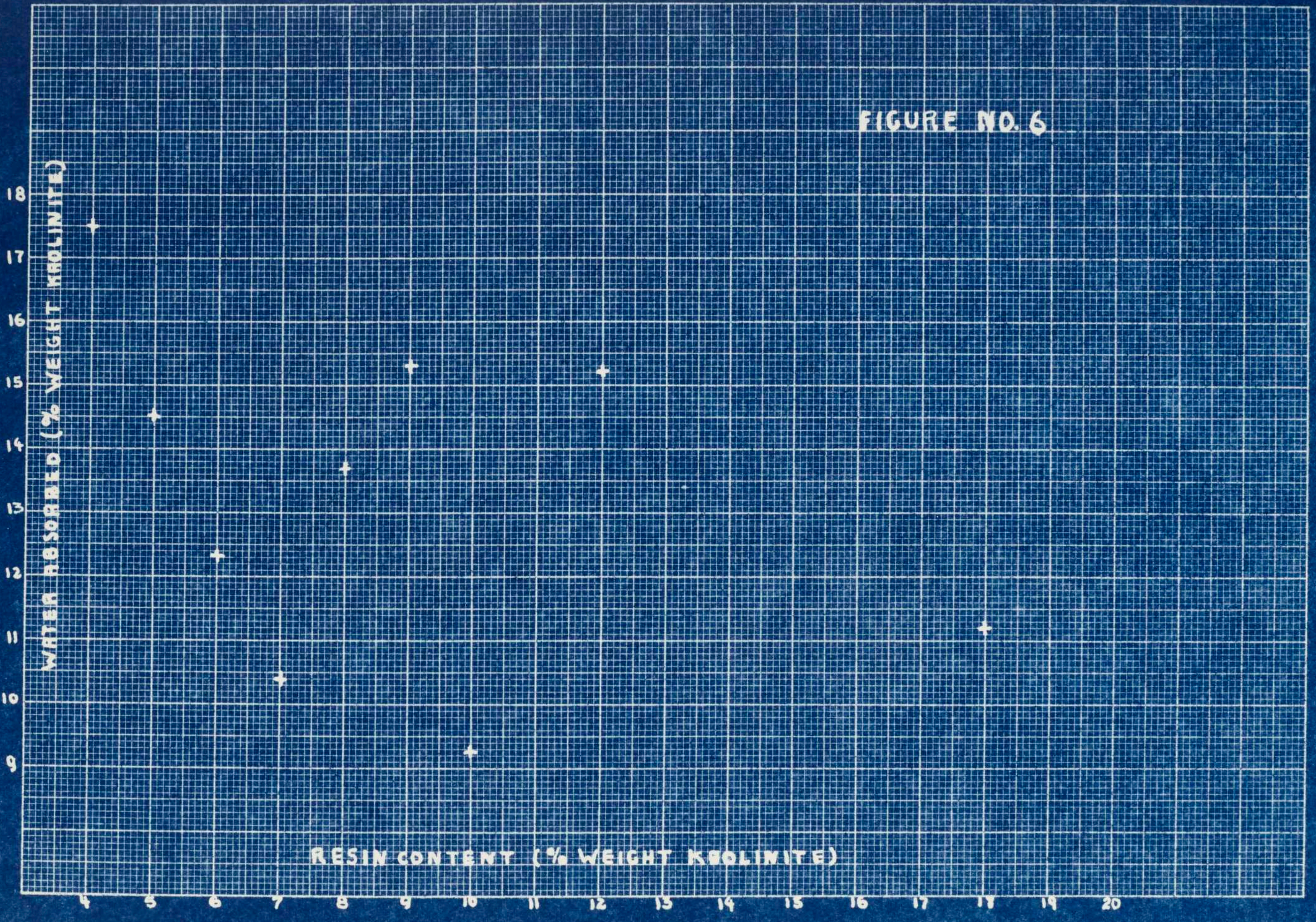

WET TENSILE STRENGTH (PSI)
800
700
600
500
400
RESIN CONTENT (% WEIGHT KAOLINITE)
4
5
6
7
8
9
10
11
12
13
14
15
16
17
18

FIGURE NO. 5

FIGURE NO. 6


WATER ABSORBED (% WEIGHT KAOLINITE)
18
17
16
15
14
13
12
11
10
9
RESIN CONTENT (% WEIGHT KAOLINITE)
4
5
6
7
8
9
10
11
12
13
14
15
16
17
18
19
20

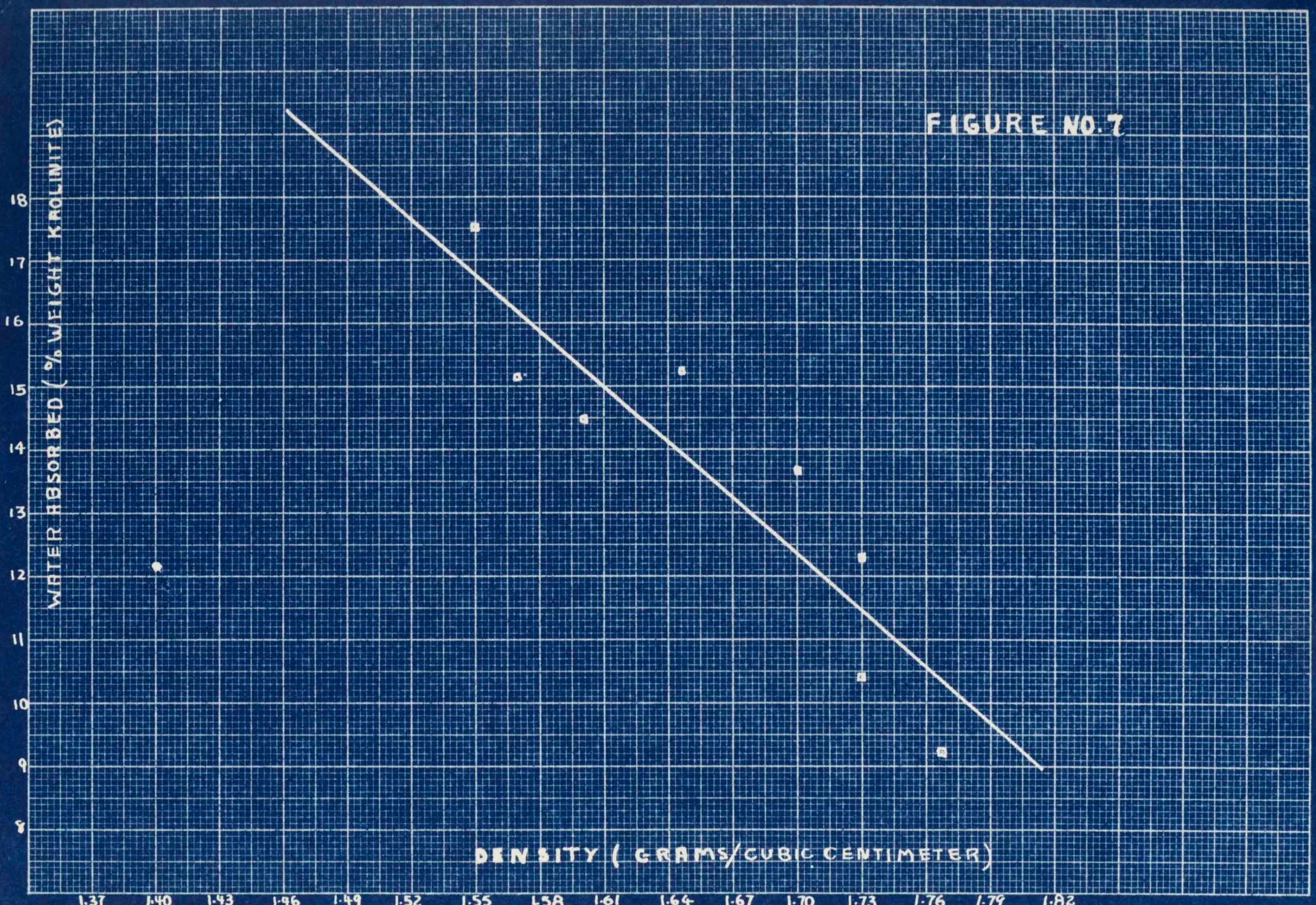


FIGURE NO. 7
WATER ABSORBED (% WEIGHT KAOLINITE)
18
17
16
15
14
13
12
11
10
9
8
DENSITY (GRAMS/CUBIC CENTIMETER)
1.37
1.40
1.43
1.46
1.49
1.52
1.55
1.58
1.61
1.64
1.67
1.70
1.73
1.76
1.79
1.82

V. DISCUSSION OF RESULTS

In the discussion of results all references will be to data as presented in the previous section entitled "Results," and in Appendix I-B.

From the results it is seen that no values are given for products below 6% resin. Below 6% resin content the samples were found to have a rougher surface than those of higher resin content, indicating that the material was not being compressed or densified as completely as the other samples. It is probable that at low resin content there is insufficient lubricant in the form of the resin and DMP-10 to allow complete flow and resulting densification. It was also noted that the lower the resin content the greater the pressure needed before no slippage occured in the hydraulic press. This fact also indicates that at lower resin content there is a resistance due to particle friction which is overcome by increasing force up to the point where the resistance force is greater than the hydraulic press force.

From Figure 4 it is noticed that as the resin content increases the curve reaches a peak at approximately 10% resin content. It is therefore concluded that in the range of 9 to 12 per cent resin there occurs a point of optimum resin content, that is, a resin content for which the tensile strength is a maximum in this particular range. At this point there is exactly enough resin to coat all the clay particles and form a complete network of bonds upon curing. While the data does show that in this particular resin content range of 9-12% there is a definite "plateau"; it is only supposition to say that this is the peak value. The data beyond this point is at best scanty, and while it does indicate a decrease it is not believed to be as reliable as the data below 12% resin content. It was seen, however, that the samples at 18 and 20 per cent resin did not break cleanly under a given force but tended to fracture. This fact gives rise to a theory that at the higher resin contents, where all of the clay particles are covered, the additional resin does not evenly disperse itself but tends to agglomerate with itself and the cured product contains areas of varying strength. When a force is applied to this material, the force will tend to follow the path of least resistance and instead of a clean break a fracture will occur.

The pertinent result of Figure 5 (wet tensile strength vs. resin content) is that the wet strength is lower than the dry strength. In the range of the investigation there is a drop in the tensile strength of approximately 50% is obtained by submersion in water for 24 hours. The submersion in water will allow the water molecules to enter between the kaolinite and resin molecules thereby reducing the attraction force and supply a lubricant for slippage. This would tend to, as the data indicates, reduce the tensile strength. Due to the reasons presented in the ensuing discussion the shape of the curve cannot be connected on, the important fact is the decrease of tensile strength due to water penetration.

Figure 6 is a plot of the water absorbed vs. the resin content. The random distribution of data on this graph indicates that the water absorbed in the system is not dependant on the resin content. In a preliminary analysis it might be thought that the water absorbed should be a function of the attractive force of the substance, but this is disproved by the data.

In Figure 7 the water absorbed is plotted as a function of the density. From this graph it is seen that the water absorbed is an inverse function of the density, that is, as the density is increased the absorbed water decreases. This observation is easily explained by discussing the mechanism of absorption; in the system at hand the water molecule must enter the material between two particles and then diffuse or be attracted inward away from the edge to allow additional water molecules to enter the system. Where the particles are closely packed, high density, there is a smaller probability of a water molecule "seeing" a space to enter the system, and once the particle has entered the system the close packing of the other molecules will offer a large resistance to movement. In a system of loose packing, low density, the water molecule will be able to enter and disperse more easily and at a greater rate; the result is a greater absorbtion of water at low densities, which is clearly shown by the data.

The data of Figure 7 is for various resin contents, this tends to verify the conclusion drawn from Figures 6 and 7; that the water absorbed is not a function of materials but of density.

A factor of importance in the experimental work which is not shown in the results is the affect of water. Before each sample was pressed the water content was determined to insure that it was below 1%, however there was always some variation in the water content as is seen in Appendix I-B. In the curing operation any water present will, due to the high temperature, vaporize and seek escape from the system, this additional internal pressure will force the individual particles apart and resultingly reduce the attractive force between them, and the strength of the product. One sample was pressed and upon being released "exploded", that is, when the hydraulic press was released, part of the material was forced out from the mold under a severe pressure. This is explained by water vapor being trapped in the system and when its pressure overcomes that of the system it forced its way out, the resulting product was simply a whitish, chunky powder of no visible strength.

It should be noted that all of the samples were of the slab type, and the dogbones were taken from the densest part of the slab- the center. The average product from the press was a circular slab about 6 to 7 inches in diameter; the specimen was cut from the center of the slab, from an area of about 2½ square inches.

The densities measured in the experiment are listed in Table III. At first it was attempted to obtain the density from the center of the spent dogbones, this however proved impossible, as the displacement apparatus, described in Appendix II-A, proved insensitive to these small samples. The densities were then taken on the whole dogbone and as a result included some material, on the edges, which was not as dense as that of the center. This fact and the estimation, that for the particular sample size the apparatus is only reliable to one part in ten, tends to indicate, in the author's judgment, that the densities are low.

A general examination of the product indicates that it can be nailed, sanded, and treated as wood. The substance is smooth, hard, ceramic like in appearance, and can be molded.

The dogbone samples were obtained by bandsawing the cured slabs to a desired shape. It was noted during the sawing that the smoothest sawing occured at samples which gave the maximum physical properties. The higher resin content products were brittle and sawed unevenly, an examination of these samples showed small layers of resin distributed parallel to the plane of compression.

VI. CONCLUSIONS

The results of the investigation of filling epoxy resins with kaolinite lead to the following conclusions:

Using the procedure and materials described in this thesis a workable product can be made in the range of six to twenty per cent resin content.

The product exhibits maximum physical properties in the range of nine to twelve per cent resin content. The maximum properties that were observed are: dry tensile strength, 1313 psi.; wet tensile strength, 725 psi.; and density, 1.77 gms. / cc. .

The maximum physical properties were observed at the maximum density.

Immersion in water will reduce the strength of the material. The reduction in strength observed in this investigation was of the order of fifty per cent, but in general it will be an inverse function of the density of the product.

The material as produced in this thesis presents possibilities of being used as a substitute for wood and ceramics.

The material can be sawed, nailed, and molded.

VII. RECOMMENDATIONS

It is recommended that additional research be carried out, using the procedure and materials of this thesis, to determine:

a. The variation in wet and dry tensile strength above twelve per cent resin content.

b. The variation in physical properties with constant density.

c. The relation between the water present during curing and the final properties.

d. Better methods of compression molding.

Experimentation should be carried out to determine the properties of resin-filler systems with the density as a variable.

It is also recommended that experimentation be carried out following the procedure of this thesis but using different resins, fillers, and curing agents.

The properties of the material indicate that research should be done to find other methods of manufacture, such as calendering and extruding.

APPENDIX I

APPENDIX I

A. DETAILS OF PROCEDURE

The procedure described in this section was used to prepare all the samples. A description of the equipment used is given in Appendix II-A.

A main problem in the investigation was to produce a uniform product; this was accomplished by approaching complete mixing. A three liter beaker containing two liters of water was the reaction vessel. A Premier Dispersator was inserted to agitate the water and 300 grams of kaolinite was slowly added to the churning water. After the kaolinite had been completely added the system was maintained in agitation to insure complete dispersion.

A resin mixture composed of the resin (varying weight percentages of the filler), the curing agent (twenty per cent of the resin), and xylene (fifty per cent of the resin) was mixed thoroughly. The curing agent, DMP-10, is valuable in such a system due to its high density and insolubility in water. The xylene reduces the viscosity of the mixture allowing it to be handled easier.

The resin mixture was slowly added, dropwise, to the agitating water-kaolinite solution, this is a tedious operation but necessary to insure complete dispersion of the resin. Adding the resin mixture to fast causes foaming which if not checked, will overflow the beaker.

Agitation was continued for ten minutes after the resin had been added, then the slurry was allowed to settle. The settled mixture was composed of a clear liquid over a sludge, the clear liquid was decanted off and the sludge was vacuum filtered with a Buechner funnel. The material from the filtering step was a firm cake of fifty per cent water content. The cake was air dried for 24 to 48 hours, a smooth powder, less than one per cent water resulted. One hundred grams of this powder was charged into the Vulcanization press where the material was cured for one hour at 30,000 psi. and 300°F.

The charge is placed between steel plates which are placed in the press. A silicate suspension was initially tried as a lubricant between the resin and the press plates, this proved unsuccessful as half the samples stuck to the plates. The problem was solved by using thin aluminum foils between the charge and the plates. In the pressing operation the press was maintained at the pressure at which no slippage ocurred in the sample.

The cured slab from the press contained a strong, dense center and a weak, powdery circumfrence. Samples were taken from the dense center by bandsawing and sanding, these samples were tested for wet and dry tensile strength and density.

The dry tensile strength was determined after the curing step. The wet strength was obtained from samples immersed in water for 24 hours, the water absorbed was also recorded in this step. The density was determined by weighing a sample and finding the volume of mercury it displaced in a Mercury Displacement device.

APPENDIX I

B. SUMMARY OF DATA AND CALCULATED VALUES

Table IV.

Dry Tensile Strength

% Resin	Force to Break Sample (lbs.)	Cross Sectional Area at Break (square inches)	Tensile Strength (psi.)
4	TOO WEAK		
5			
6	34	0.0528	642
6	45	0.0505	890
7	50	0.0592	845
8	64	0.0582	1105
8	52	0.0572	910
9	49.5	0.0376	1313
10	77.5	0.0562	1380
10	43	0.0342	1228
12	58	0.0397	1460
12	45	0.0418	1080
14	49	0.0650	753
14	35.5	0.0440	805
18	50	0.0578	875
20	30	0.0637	441

Table V.

Water Content Before Curing Operation

% Resin	% Water Present
4	0.70
5	0.50
6	0.40
7	0.21
8	0.22
9	0.18
10	0.45
12	0.60
14	2.10*
16	1.90*
18	1.60*
20	1.10*

*- Given additional drying time

Table VI.

Wet Strength and Water Absorption

% Resin	Density (gms./cc.)	% Water Absorbed	Tensile Strength (psi.)
4	1.55	17.5	-
5	1.60	14.5	200
6	1.73	12.3	595
7	1.73	10.4	596
8	1.70	13.7	705
9	1.65	15.3	640
10	1.77	9.3	625
12	1.57	15.2	725
18	1.40	12.2	460

APPENDIX I

C. LOCATION OF ORIGINAL DATA

The original data taken in this investigation may be obtained from Professor Alan S. Michaels of the Massachusetts Institute of Technology.

APPENDIX I

D. NOMENCLATURE

Throughout this thesis the following notation was used:

DMP-10 - dimethylaminomethylphenol

% curing agent - grams of curing agent per one-hundred grams of resin

% absorbed water - grams of water per one hundred grams of kaolinite

% resin - grams of resin per one hundred grams of kaolinite

resin content - same as % resin

% xylene - grams of xylene per one hundred grams of resin

dry strength - tensile strength after curing step

wet strength - tensile strength after immersion in water for 24 hours

filler - kaolinite clay

resin - epoxy resin (Epon 828- Shell Chemical Company)

APPENDIX II

APPENDIX II

A. EQUIPMENT

The equipment used in this investigation which is not usually found in a laboratory will be described in this section.

Agitator

A Premier Dispersator (Premier Mill Corp.) was used to agitate the water solutions. The mixer consisted of a motor mounted on a movable stand which was connected to a long shaft. At the end of this shaft was a hexagonal shaped cylinder with slits cut in the sides. Two blades from the shaft were connected to the inner part of the cylinder. The speed of the agitator was controlled by an external variat.

Press

The press used in this investigation was a Preco Hydraulic Press (Preco Mfg. Co.) . The press was capable of exerting a pressure of 40,000 psi., the press faces contained thermostats which could maintain the temperature at a fixed point between 100°F. and 600°F. A set of flat steel plates served as the molds.

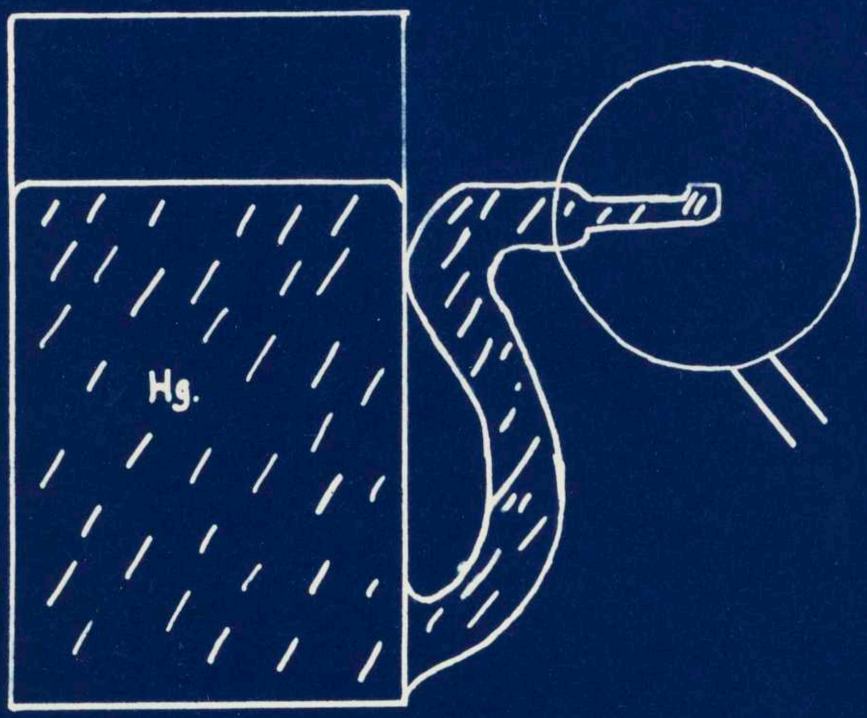
Density

Figure 8 is a reproduction of the Mercury Displacement apparatus used in this experiment. A sample is weighed then submerged in mercury, the displaced mercury is weighed, its volume determined, and the density of the sample calculated.

Tensile Strength

The tensile strength machine used in this investigation was of standard form. A motor was geared to raise or lower a stress ring which contained an indicator calibrated to the applied force. The sample is placed taunt between a fixed bottom ring and the movable top ring, the motor is set in motion and the gauge is read when the sample breaks. The cross sectional area is measured and the tensile strength calculated.

FIGURE NO. 8



MERCURY DISPLACEMENT APPARATUS

APPENDIX II

B. LITERATURE CITATIONS

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