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Kenneth C. Rasmussen
Western Michigan University

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Kenneth C. Rasmussen

THE BONDING OF FELTED GLASS FIBERS BY THE USE OF ORDINARY PAPERMAKERS' WET-STRENGTH RESINS

A Senior Thesis

Conducted under the guidance and supervision of Mr. R. T. Elias, Associate Professor of Pulp and Paper Technology, Western Michigan College, Kalamazoo, Michigan

Submitted June 6, 1955

Kenneth C. Rasmussen

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ABSTRACT

Papermakers' wet-strength resins have been added to felted glass fibers as to increase the strength of the fibers.

On the basis that a stronger sheet of paper made from glass fibers could be put to many uses, experiments were of two types. First, an excess of wet-strength resins were added to the fibers. Second, handsheets made of one hundred per-cent glass fibers were dipped into a two per-cent resin tub-sizing solution.

Favorable results were obtained in both cases.

Literature Survey of Glass Fibers and Wet Strength Resins

I. Glass Fibers

A Short History of Glass

In laboratories throughout the country, paper has been made from glass fibers with no additive. Much of this work was done by the National Bureau of Standards in co-operation with the Naval Research Laboratory. (1) Through this experimentation a relatively strong sheet of glass paper has been made compared to previous paper made from glass. However, the goal of the present work will be to make a stronger sheet of paper from felted glass fibers by the addition of papermakers wet strength resins.

The second edition of Webster's New International Dictionary defines glass as: "An amorphous substance, usually transparent or translucent, consisting ordinarily of a mixture of silicates, but in some cases of borates, phosphates, etc. Most glass is made by fusing together some form of silica, as sand, an alkali, as potash or soda, and some other base, as lime or lead oxide. It is blown, pressed, cast, and cut to a great variety of shapes."

Glass is one of the oldest and most useful minerals known to mankind. Glass has been used by man since the very earliest time. (2) The commonest use of natural glass by primitive people was for arrowheads and spear-heads. It is believed that glass was first made artificially early in the cultural history of mankind. It has been assumed that the discovery of glass came after, and was a consequence of the development of metallurgy. However it appears more probable that both metallurgy and glass-making were consequent upon the development of the potters art. The firing of crude pottery was the first step in the development of the arts based on fire, and from this the development of glaze and then of glass was a logical and probable step.

Possibly the production of glass owes its beginning to accident. An illustration of such a possibility is the glass formed by the burning of grain and the fusion of the ash, as the result of fire caused by lightning. In 1600 the art of cut glass was developed. In 1615 the use of coal instead of wood for fuel was discovered in England. Another English invention, flint glass, was made in 1675.

More recent developments have been enormous. The revolution in glass manufacturing methods which has taken place in the twentieth century, especially in the last three decades, has transformed the industry from one dependent on the skill of the individual worker, to one dominated by continuous machine production, precisely controlled by the application of scientific methods.[2]

In the last two decades glass has become even more important because of the many uses of its fibers. It is now used in making clothing and other things of the textile world, decorative articles, and paper making.

Microscopy and Identification of Glass Fibers

Glass fibers in general have the same characteristics as the mother substance, glass. The fibers will not dissolve in 90% phenol or concentrated HCl as will cellulose fibers. They will melt but will not burn.[3] The fibers however do not feel like glass. They can be bent like rubber, twisted like thread or squeexed like a sponge. Yet each tiny fiber still possesses all of the characteristics of the material. It has high temperature resistance and is non-hygroscopic; the individual fibers will not absorb moisture. The fibers are resistant to ordinary weathering and to acids, oils, and corrosive vapors. They are very strong and have high electrical resistance.[4]

Previous Experiments with Glass Fibers and their Advantages over other Synthetic Fibers

Glass fibers, used in the paper-making industry, began to come into their own about ten years ago. Most of the work on glass fibers for paper has been done by the National Bureau of Standards in co-operation with the Naval Research Laboratory.

The National Bureau of Standards in co-operation with the Naval Research Laboratory has developed a paper composed entirely of glass fibers with no additive.[1] The paper has several applications. It is very effective as an air filter for gas masks, respirators, etc. In gas mask tests in a smoke filled room, only one smoke particle in 100,000 passed through the glass paper filter. The paper has excellent electrical characteristics and should be useful as an insulator and dielectric. It can be used to make oil-impregnated paper compacitors which will withstand temperatures above 200 degrees Centigrade. Kraft paper, commonly used, will not, withstand temperatures much above 100 degrees Centigrade.(1)

The equipment used by the Research Laboratory was a 50-pound beater, 29-inch Fourdrinier papermaking machine with a wire 23 feet long, one press, nine 15-inch driers, and a reel. Hand sheets were made with a sheet mold, an hydraulic press, and a sheet drier. Four grades of "Fiberglas" were used. They were AAA- .5- .75 mu diameter, AA- .75- 1.5 mu diameter, A- 1.5- 2.5 mu, and B- 2.5- 3.75 mu. The fibers are clean and can be furnished directly to the beater.

The manufacturing procedures of glass fibers differ mainly in two ways from cellulosic fibers. First, the glass fibers are more brittle and easily broken, and second, the glass fibers cannot be fibrillated and gelatinized like vegetable fibers. Because of these properties prolonged beating is not necessary. The treatment of glass fibers in the beater is different from that of vegetable fibers. Vegetable fibers are beaten to increase their fibrillar nature and hydrate them so their gelatinous surfaces will adhere to the paper, increasing its strength. However, glass fibers apparently owe their strength to friction between fibers rather than to the sort of entaglement and adhesion that occurs in vegetable fibers. There should be as little breakage as possible when beating glass.

Heretofore, the most efficient commercial air-filter paper was made from asbestos fibers, mixed with cellulose fibers. Asbestos, however has many disadvantages. First, only 300-400 pounds of usable fibers can be obtained from a ton of pulp. Second, breaking down, dusting, and washing of these fibers requires much time. Third, the distance the asbestos has to be shipped also adds to the expense. The best asbestos mined is Bolivian "Blue". Fourth, asbestos - cellulose paper will support mildew growth whereas glass will not, andfifth, asbestos fibers are not as small in diameter as glass fiber. The smaller the fiber diameter, the more efficient the filter paper.(5) Because of these many disadvantages of asbestos fibers as compared to glass fibers, research and experimentation is being done with glass fibers in an attempt to increase its strength in papermaking. The Messr's O'Leary, Hobbs, Missimer, and Erving have been experimenting, trying to increase the strength of the final product.(6)

Although, through their experimentation, paper made from glass fibers now has reached a new high for strenght (a tensile strength of approximately 300 p.s.i. at a pH of 2.9.[6] I believe that even higher strength for paper made from glass is desirable. This will be the goal of the present work.

II. Wet Strength Resins

Cellulose paper, when wetted will lose most of its strength. This was a black spot in the paper industry but by the work done in chemical laboratories, wet strength resins were discovered. This brought about a great change in the paper industry bringing about the mass production of various paper products which still possess a high degree of strength even when wet.

Properties of Paper as Influenced by Wet Strength Resins

Wet strength resins affect a number of properties of paper. Some of the properties that tend to improve with resin treatment are: dry tensile and mullen, dry folding endurance, wax pick, anti-dusting and linting, sizing, dry stiffness, wet, rub resistance and wet tear. Properties which tend to decrease upon the addition of wet strength resins are: dry tear, softness, and water absorption.[7]

The Effect of pH on Wet Strength Resins

All three types of resins (melamine resin, anionic urea resin on unbleached kraft, and cationic urea resin) cure faster as the pH of the treated sheet is lowered.[7] However, this tends to promote hydrolysis of the resin during use or storage and also tends to degrade the base paper, so a compromise is necessary. The usual pH range is from 4.5-6.0. Melamine resins will cure at a higher pH however. Sometimes as high as 8.0. The pH of the stock though has only a slight effect on resin retention with somewhat higher results being obtained at the higher pH range. This is not the same as rate of cure.

Properties of Wet Strength Resins. (Parez)

The melamine resin is a triazine consisting of a six membered ring of alternating carbon and nitrogen atoms. Each carbon atom is also attached to an NH_2 group and it is these groups that react with formaldehyde. This resin has the following properties: It is a dry, white powder with an apparent density of 0.4 (approximately 25 pounds per C. F.). It disperses in water with agitation at

50 to 60% solids, but hydrophobes out on further dilution. It has a 3% maximum moisture content. The viscosity of the resin (60% solids at 25 degrees Centigrade) is 300-700 centipoises. The pH (60% solids dispersion) is approximately 9. The resin colloid carries a positive charge and is quickly adsorbed on the negatively charged cellulosic fibers.(8)

The use of wet strength resins on cellulosic fibers has become very important in many fields of papermaking. Experimentations in this work will deal with its effect upon the bonding of felted glass fibers in order to make a stronger sheet of paper from glass.

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Experimental Plan

The effect of wet strength resins on felted glass fibers is to be determined by using different concentrations of the substances at different pH levels. Because I have found no previous records on the combination of glass fibers and wet strength resins, the degree with which one will adhere to the other will first have to be determined. When working with glass fibers however, the pH may be varied to a large range. Lower and higher pH's than are mentioned in the outline, which will follow later, may be used. The pH will be controlled by the use of alum, sulfuric acid, caustic, and lime.

Retention of the Resin

The retention of the resin will be determined by running an ash test by the T 413 m-45 procedure. If the results are unsatisfactory a test to determine the amount of organic nitrogen in the sheet will be run according to the T418 m-50 procedure. This procedure is used to determine the amount of urea- formaldehyde resins, Melamine resin, and other nitrogenous organic materials in paper. A Kjeldahl digestion and distillation apparatus will be used.

The resin colloid carries a positive charge and is quickly adsorbed on the negatively charged cellulose fiber. However, this may not be the case with glass. Apparently retention is not dependent upon the use of coagulants such as alum or size. However, in the case of cellulose fibers, complete retention is not achieved, and the addition of more than five per cent of resin is considered beyond the range of maximum effectiveness. With three per cent of resin added, the retention is found to be from sixty to eighty per cent.

Treatment of Glass Fibers

Glass fibers will be treated in the beater differently than vegetable fibers are. Vegetable fibers are beaten to increase their fibrillar nature and hydrate them so their gelatinous surfaces will adhere to the paper increasing its strength. However, glass fibers apparently owe their strength to friction between fibers. Glass fibers are much more brittle and easily broken than cellulosic fibers. They also cannot be fibrillated and gelatinized like vegetable fibers. Because of these properties prolonged beating is not necessary.

Equipment to be Used

The glass fibers will be placed in a Valley Laboratory Beater for a period of thirty minutes. During this time the fibers will be mixed and not beaten. This is to break up the chunks of fibers but not the fibers themselves. Handsheets will then be made on a British Sheet Mold, pressed by an hydraulic press, and then dried on the Noble and Wood drier. The following strength tests will be run on the handsheets: tensile test on a Schopper-Riegler Tensile Tester, tear test on the Elmendorf Tear Tester, and bursting strength test on the Mullen Tester. A porosity test will also be run to determine the effectiveness of the sheet as a filter pad.

Outline of Experimental Work

GLASS FIBER SIZE	FIBER CONC. #/100# H_2O	RESIN CONC. #/100# H_2O	pH	
.5-.75 mu. diam.	1	.01	4	
.5-.75 mu. diam.	1	.01	5	
.5-.75 mu. diam.	1	.01	6	FIRST
.5-.75 mu. diam.	1	.02	4	
.5-.75 mu. diam .	1	.02	5	BEATER
.5-.75 mu. diam.	1	.02	6	
.5-.75 mu, diam.	1	.03	4	RUN
.5-.75 mu. diam.	1	.03	5	
.5-.75 mu. diam.	1	.03	6	
.75-1.5 mu. diam	1	.01	4	
.75-1.5 mu. diam.	1	.01	5	
.75-1.5 mu. diam.	1	.01	6	SECOND
.75-1.5 mu. diam.	1	.02	4	
.75-1.5 mu. diam.	1	.02	5	BEATER
.75-1.5 mu. diam.	1	.02	6	
.75-1.5 mu. diam.	1	.03	4	RUN
.75-1.5 mu. diam.	1	.03	5	
.75-1.5 mu. diam.	1	.03	6	

GLASS FIBER SIZE	FIBER CONC. #/100# H_2O	RESIN CONC. #/100# H_2O	pH	
fines	1	.01	4	
fines	1	.01	5	
fines	1	.01	6	THIRD
fines	1	.02	4	
fines	1	.02	5	BEATER
fines	1	.02	6	
fines	1	.03	4	RUN
fines	1	.03	5	
fines	1	.03	6	
mixture of sizes	1	.01	4	
mixture of sizes	1	.01	5	
mixture of sizes	1	.01	6	FOURTH
mixture of sizes	1	.02	4	
mixture of sizes	1	.02	5	BEATER
mixture of sizes	1	.02	6	
mixture of sizes	1	.03	4	RUN
mixture of sizes	1	.03	5	
mixture of sizes	1	.03	6	

These size fibers were used by the National Bureau of Standards in co-operation with the Naval Research Laboratory. They purchased them from the Owens-Corning Fiberglas Co. of Toledo, Ohio. In their experimentation they made paper from glass fibers with no additive. They received their strongest sheet of paper when they used a combination of long and short fibers free from fines. They also learned that concentrations of glass fibers above one per cent are undesirable becaause breakage of the fibers occurs.

DATA

The experiments in this work were not carried out exactly as was stated in the experimental outline (see page 12). Handsheets were made from glass fibers of one-half, one and three micron diameters. The wet-strength resins used were Uformite 700 and Kymene 234, cationic resins and Uformite 467 an anionic resin. Handsheets were made by the following method:

First, the fibers were mixed in a Waring Blender for five minutes. This was done to mix the fibers and break up any lumps which might occur. Beating was not necessary as glass fibers do not fibrillate or gelatinize as do vegatable fibers. Also, glass fibers are brittle and easily broken.

Second, the wet-strength resin was added and the sample was placed in a British Sheet Mold. The pH was adjusted to 4.7 by the use of alum.

Third, the sheets were pressed in a hydraulic press at fifty pounds pressure and then dried on a Noble and Wood Sheet Drier.

Fourth, the sheets were placed in the humidity room for a length of time and strength tests were run.

Strength tests run on the handsheets were, wet and dry mullen, wet and dry tensile, basis weight, and ash. The results of these tests are found on pages 18 and 19.

In these experiments, an excess of wet-strength resins were used and therefore this is not recommended for commercial use. However, a tub-sizing solution was prepared of a two per-cent Uformite 700 resin. Rsaults of these experiments were encouraging.

The first set of handsheets showed an increase in dry mullen as high as seven pounds in the handsheets containing the wet-strength resin over the sheet with no resin. An increase in dry tensile strength as high as four and two-tenths pounds was also abtained. The wet tensile and wet mullen tests ranged between fifty and sixty per-cent of the dry.

TABLE I

Tests Run on Handsheets of Glass Fibers Containing an Excess of Wet-Strength Resins

pH on all sheets------------4.7

Set	Resin	Fiber Size (diam.)	Basis Wt.	Mullen dry	Mullen wet	Tensile dry	Tensile wet	Ash (%)
1	U-700	1 u	48.8	2.5	1.5	2.0	.7	
2	None	1 u	47.9	0	0	.6	0	14.4
3	U-700	1 u	49.1	5.0	3.0	2.9	.6	30.3
4	None	½ u	48.3	0	0	.4	0	6.9
5	U-700	½ u	50.1	4.5	2.0	3.0	.4	
6	U-700	½ u	51.0	6.0	2.0	3.6	1.0	25.2
7	None	3 u	51.8	0	0	.3	0	
8	U-700	3 u	49.8	2.5	1.3	2.0	.6	
9	U-700	3 u	48.7	3.0	1.7	2.2	2.7	
10	U-467	3 u	47.9	1.5	0	..8	.2	
11	U-467	3 u	49.3	1.0	0	.9	.2	
12	U-700	½ u	50.4	3.5	2.0	2.3	.8	
13	U-700	½ u	51.0	6.5	4.0	3.4	1.1	
14	U-700	½ u	49.8	7.0	4.0	2.9	1.0	
15	U-467	1 u	50.4	5.5	3.0	2.8	.8	
16	U-467	1 u	50.5	6.5	4.0	3.1	1.0	26.2
17	K-234	½ u	50.0	3.5	1.5	2.1	.6	
18	K-234	½ u	48.9	4.0	2.0	2.4	1.2	36.4

Key
U------------- Uformite
K------------- Kymene
u------------- micron

TABLE II

Tests run on Handsheets of Glass Fibers Dipped in a 2%, Uformite 700 Tub-Sizing Solution

pH --------------------- 4.3

Size of fibers (diam.) -- ½ micron

Set	Basis Wt.	Mullen		Tensile	
		dry	wet	dry	wet
1	49.2	2.0	1.5	1.2	.7
2	50.8	2.5	1.5	1.7	1.1
3	51.7	2.5	1.5	1.5	1.0
4	49.3	2.0	1.0	1.4	1.0
5	50.2	2.5	1.5	1.7	1.2

Summary

The experiments were divided into two categories. First, a group of handsheets were made up using an excess of wet-strength resin. Second, a group of handsheets were made and dipped into a tub-sizing solution of two per-cent Uformite 700 resin.

The experiments showed that the one-half micron diameter fiber handsheets had the best strength after treatment with the resin. The cationic resins were the only ones retained in the glass fiber sheets. Ash tests showed that approximately thirty per-cent of the resin was retained.

The handsheets which were dipped into the tub-sizing showed promise. An increase of 2.5 pounds dry mullen and 1.7 pounds dry tensile were obtained.

Conclusions

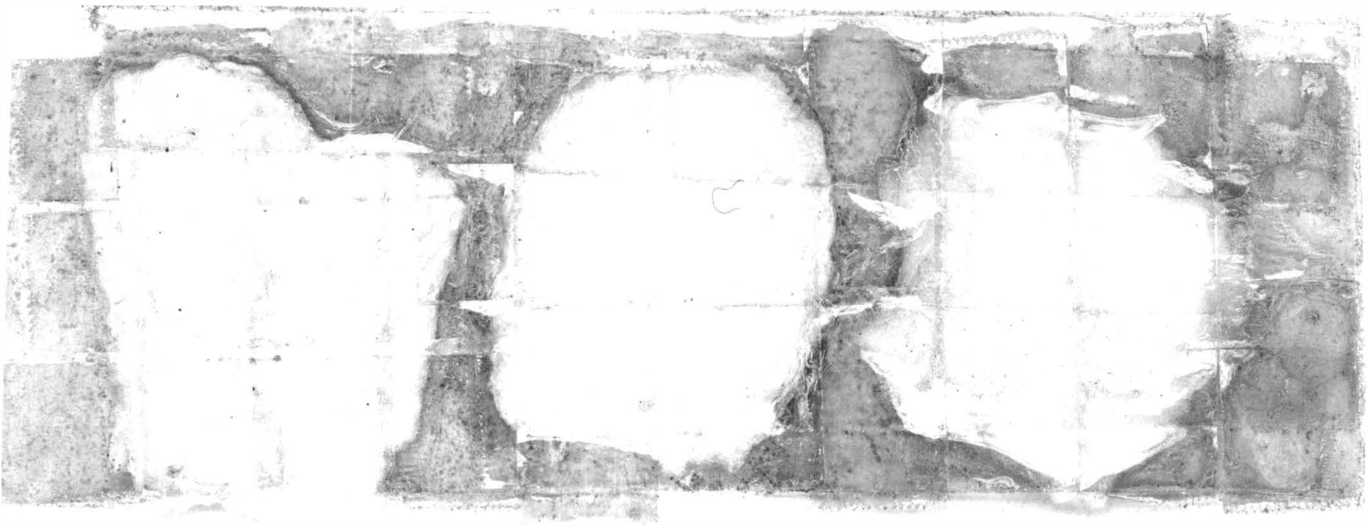
On the basis of the work done, it is believed that a definite amount of strength is obtained when cationic wet-strength resins are added to glass fibers.

Recommendations for Further Work

It is felt that a greater amount of strength can be obtained from glass fibers by the addition of ordinary papermakers' wet-strength resins. Further insight into the bonding of these fibers should be investigated.

SAMPLES

½ micron fibers   1 micron fibers   3 micron fibers



Glass Paper

The End