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USING KELGIN TO INCREASE HEAT RESISTANCE AND STRENGTH OF THE SHEET OF PAPER AGAINST PROLONGED HEATING

by

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A Thesis submitted to the Faculty of the Department of Paper Technology in partial fulfillment

of the

Degree of Bachelor of Science

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ABSTRACT

The purpose of this paper has been to examine what properties of sheet strength may be stabilized by the use of kelgin in a size press application and after a subsequent heating period.

A softwood kraft pulp was used because of the strength properties which it possesses. Alkaline sizing with Aquapel and Kymene was felt to be the best because of the alkaline p.H. range which was desired. The sheets were sized with solutions of Kelgin (M.V.) of varying concentrations and treated at 150 and 175°C. The effects were studied using the M.I.T. Fold Test, the Instron Tensile (kg to rupture), and the Instron Elongation.

The results showed that as the concentrations of the kelgin size solutions increased, some protection was afforded the fiber against prolonged heating. The strength loss was appreciable in the samples which were treated at the different temperatures. The room temperature samples showed higher strength properties than either of the samples heated at 150 or 175°C. which was as expected.

It was shown that the high viscosity of the kelgin solutions at the concentrations of 1.00 and 1.25 percent tended to give a tapering off of the protection which was expected. A viscosity reducer (Azite 900 Liquifier) was used in conjunction with the kelgin solutions to study the effects of a reduced viscosity on strength maintenance. It was shown that the liquefier did indeed increase the penetration of the kelgin into the sheet and provide a better protection.

The experimental findings demonstrate that the use of kelgin in the form of a sizing solution can give beneficial results against loss of strength upon prolonged heating. It was also shown that the magnitude of this effect was not as great as had been expected from previous findings.

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INTRODUCTION

The degradation of the cellulose fiber upon exposure to heating has long been a big problem for the manufacturers of paper and paperboard products. As more and more papers are being used in processes involving a stage of heating, it is becoming an ever increasing problem to develop papers which will not loose their strength upon heating. Heating the cellulose fiber in the mat to temperatures over 100°C. causes several things to happen. The fiber has an equilibrium condition with its surroundings, in which it is strong and fairly flexible. Upon heating, the moisture content of the sheet is decreased because the water molecules present are provided with sufficient kinetic energy to break hydrogen bonds which have been formed between the fiber and the available water (moisture in the sheet is from 4 to 7 percent normally). The removal of this water makes the fiber less flexible and thus fold strength is reduced. As shown in the work done by A. H. H. Van Rayen (13), the folding endurance decreased because cellulose chains are scissored by high oxidative temperatures. The amount of fresh air seems to have little effect on the fiber, but the duration of and the temperature of heating are absolutely vital to the folding endurance of the fiber in the sheet as shown by P. F. Wehmer (19). The types of pulps tested also seem to have little effect upon the decrease in strength due to heating, again only temperature and length of heating period are

characterized as important. It is published that the moisture content in the sheet below 4 percent is classed as loosely bound water, and that the water below .5 percent (tightly bound water) cannot be removed without causing degradation of the fiber. In this range of .5 to 4 percent moisture content in the sheet, the strength of the sheet is being seriously reduced when the sheet is heated and the moisture content reduced into this area of moisture content. The aim of substances which have been used for flameproofing and heat resistance has been to develop a sheet which does not loose its water and is resistant to the oxidative scissoring which occurs at temperatures around and over 100°C. Some insulating papers are being tested at very high temperatures, while tire yarn has been manufactured to withstand temperatures up to 175°C.

Some work has been done, mostly in the last ten years, to protect the sheet at elevated temperatures and also to increase the aging qualities of papers. Almost all the methods which were researched deal with a phase of preparation of a solution with which the sheet is impregnated to give it the properties in the discussion previous, namely flame resistance and heat stability.

These impregnating solutions seem to be similar in certain respects. A long chain polymeric molecule or an amino linkage chain is used in combination with a smaller molecule sometimes organic and often times inorganic, and the resulting product

used to impregnate the sheet. In a paper by George E. Brown (1) a layer of reduced amino groups of peptide structure from a protein were placed on the sheet and then a layer of oxidized nitro groups of peptide structure was superimposed over this first layer. A neutralization reaction on and at the sheet surface giving the desired results of resistance to fire, water, and chemicals.

Alton A. Cook (3) received a patent for his process of using an aequous solution of an alkali borate and a carboxylic acid containing at least one hydroxyl group for impregnating the sheet. A cellulosic and porous sheet are the principal requirements for the success and use of this method. Water repellancy can be acquired in addition by adding a layer of paraffin wax over the alkali borate carboxylic acid coat. Harold P. Knight (8) was able to prevent embrittlement by adding urea to the alkali metal borate.

By mixing a finely divided metal oxide and a halogenated long chain thermoplastic polymer Joseph W. Creely (4) achieved a fire retardant character on his sheet. This method is relatively expensive and is generally used for regenerated fibers of cellulose structure. Along this line, John E. Dereich (6) used a composition consisting of a transparent, organic high polymeric thermoplastic material selected from the group consisting of polyester resins, polyethylene, cellulose acetate butyrate, and polystyrene and reacting this group with a

chlorinated aliphatic hydrocarbon (shrt chain 18 to 36 carbon atoms), and adding a potassium, barium, or antimonyl tartrate, or using ethylenediamine to get the flame resistant property onto the sheet.

John Truhlar (18) achieved fire proofing by using a chloronated napthalene together with a chlorinated paraffin wax and a fireproofing agent. The degree of corrosion and reactivity to chemicals was also reduced with these chlorenated compounds.

It has been shown by Walter F. Reynolds (14) that in the manufacture of asbestos papers, using a water soluble linear amino carbon polymer of molecular weight over 5 x 10^6 we get a substantial increase in drainage and also an increase in tensile strength with heating.

The use of compounds containing phosphorous atoms has been on the increase in recent years. Samuel Tewin (17) reacted a phosphonium halide with an olifinic aldehyde to acquire flame resistance. Roger C. Steinhauer (15-16) has developed the use of an aequous impregnating solution of an aziridinyl phosphoric oxide and a nitrogen compound. Also he has used ethylenediamine in combination with phosphorous pentoxide of phosphorous oxytrichloride to give a flame-proofedness to the cellulose fiber.

The work of Raymonde G. Madeline and Jean F. Pathus-Labour (9) is probably the offering which most closely relates to the thesis topic which I am proposing. In a process of fireproofing textiles and like substances an aequous impregnating solution

containing at least one water soluble compound selected from the group consisting of salts of esters of alginic acids was applied followed by a second bath. The first impregnating coating was followed by a treatment with hygroscopic calcium chloride so as to precipitate the calcium salt of alginic acid onto the fiber which is being treated. A gas permeable coating is thus formed on the fiber which prevents burning. Water is bound by the layer of coating material on the fiber and thus moisture removal also becomes much more difficult. Thus we get an impregnated material which is substantially gas impermeable and highly resistant to burning and which remains elastic and non-brittle due to the before mentioned bound water.

In a thesis presented last year, work was done using kraft pulp and making handsheets for subsequent testing. The experimenter used kymene resin and aquapel size solutions on the sheets, but added to two of the four sets tested a .5 and a l percent solution of kelgin by size press application. An increase in folding strength was shown. In this thesis an attempt will be made to reproduce these results and to conduct the appropriately associated tests in an endeavour to discover why this increase is fold strength was obtained, and to see if this strength increase carries over into the other physical tests on the paper treated with kelgin.

EXPERIMENTAL PROCEDURE

- 1. Pulp-bleached Kraft (celgar)
- 2. Beaten to C. S. F. 575 ml-
- 3. Handsheets on British Sheet Mold
- Used Aquapel followed by Kymene at 4 percent level on 0. D. fiber.
- Set A unheated Humidity Room conditioned (50 percent R. H. 70°F.).

Sized with solutions of Kelgin (M. V.) (in percent)

0**A** 1A 2A 3A 4A 5A

0.0% .25% .50% .75% 1.00% 1.25%

 Set B - heated 150°C. for 24 hours and conditioned humidity room.

Sets OB, 1B, 2B, 3B, 4B, and 5B treated with solutions as in part number 5.

 Set C - heated 175°C. for 24 hours and conditioned in humidity room.
Sets OC, 1C, 2C, 3C, 4C, and 5C treated with solutions as in part number 5.

 Additional sheets treated using Azite900 liquifier in powdered form at 50% level of dry solid weight of Kelgin in solutions of concentration . 25, .75, and 1.25 percent. Samples 1A, 1B, 1C - .25 percent Kelgin plus Azite 900 Samples 3A, 3B, 3C - .75 percent Kelgin plus Azite 900 Samples 5A, 5B, 5C - 1.25 percent Kelgin plus Azite 900

- 9. Samples in number 8 treated in accordance to sets A, B, and C in steps number 5 for set A, number 6 for set B, number 7 for set C.
- Kelgin applied by size press application using old mother hubbard's ringer, from an old style ringer washing machine.
- 11. Testing performed:

A. M.I.T. Double Folds (1 kg.)

B. Instron Tensile and Elongation

DATA TABLE

Sample	M.I.T. Double	Instron	Instron (%)				
	Folds	Tensile (Kg.)	Elongation				
0A	1279	5.94	2.55				
1A	1335	6.08	2.51				
2A	1351	6.11	2.61				
3A	1235	6.38	2.89				
4A	10 77	6.70	3.06				
5A	1215	6.59	2.73				
0B	193	4.72	1.37				
1B	174	4.06	.89				
2B	167	4.82	1.30				
3B	413	5.55	1.64				
4B	198	4.90	1.39				
5B	151	4.90	1.26				
0C	.25	3.17	.58				
1C	.12	2.42	.44				
2C	.25	3.16	.60				
3C	.45	3.16	.60				
4C	.45	3.63	.69				
5C	.25	2.83	.58				
	AZITE	E 900 LIQUEFIER					
1A	1627	4.53	1.66				
3A	1512	6.49	2.26				
5A	1538	5.81	1.99				
1B	367	5.30					
3B	1313	5.75					
5B	1080	5.70					
1C	2.5	4.46	.99				
3C	.5	3.75	.63				
5C	0	4.31	.76				







DISCUSSION OF DATA

The folding strength of the samples which were sized and treated at room temperature remained fairly constant, with a slight decrease showing itself in the samples sized with the 1.00 and 1.25 percent concentration of Kelgin. The folding strength of the samples treated at 150°C. also remained fairly uniform, at a much lower level than the unheated sheets however, with a slight rise at .75 percent and again a tappering off at 1.00 and 1.25 percent level of concentration of the kelgin size. The samples heated at 175°C. gave no register of folding strength at either 1 Kg. or 1/2 Kg. on the M. I. T. Double Fold Tester.

In explanation of the preceeding paragraph, clarification of the following points is in order. At room termperature it is not felt that the Kelgin used to impregnate the sheets received the proper curing to acquire the proper strength characteristics which are being explored. Also, the tapering off of the curve near the end of the concentrations of 1.00 and 1.25 percent may possibly be due to the fact that the kelgin solutions at these levels was very viscous and therefore an improper impregnation was acquired, and perhaps more of an outside the sheet layering effect was achieved rather than impregnation. This layering effect also showed itself in the 150°C. samples.

The Instron Tensile (Kg. to Rupture) showed the results of a gradual increase at room temperature. An initial decrease and then an increase and subsequent tapering off of strength at the 150°C. level. The 175°C. samples showed the same pattern as the 150°C. samples but at approximately a 1.5 Kg. less rupture strength.

The gradual increase and subsequent tapering off is once again due to the film forming character of the very viscous Kelgin solutions, as explained in the earlier paragraph.

It was shown that a gradual increase followed by a tapering off was again experienced in the percent elongation. These initial increases and subsequent decreases have already been discussed previously.

It was reasoned that if the improper impregnation was being achieved with the Kelgin solutions that the addition of a liquefier might reduce the viscosity at constant dry weight solids to a more effective form. Azite 900 liquefier was used in the appropriate Kelgin solution to test this hypothesis.

This hypothesis was indeed shown to be probable by the results obtained. In folding strength, the room temperature samples and the 150°C. samples showed a good increase, while the 175°C. samples were again untestable (showed no fold endurance).

In the samples tested for tensile, an increase was shown at 150°C. and 175°C. , which was as expected.

Elongation remained at a constant level for the 150°C. sample, and an increase and leveling off was shown at 175°C.

As hypothesized, it was indeed shown that there could have been some relation between the viscosity of the Kelgin and the results which were obtained. The reduced viscosity Kelgin solutions did give a better impregnation and thus better strength properties to the sheet.

CONCLUSION

The purpose of this thesis has been to experiment and attempt to determine what properties may be imparted to the sheet using Kelgin in a sizing solution application. The results are fairly gratifying in that it has been shown that some degree of protection to strength loss during heating may be acquired, but whether this gain is to be economical and justifiable on a commercial level would be doubtful until more extensive investigations have been made on the use of Kelgin in this manner.

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