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Optimization of the Bleached Optical Properties for Southern Pine Sulphonated Thermomechanical Pulp

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OPTIMAZATION OF THE BLEACHED OPTICAL PROPERTIES

FOR SOUTHERN PINE SULPHONATED THERMOMECHANICAL PULP

A THESIS SUBMITTED
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INTRODUCTION

The object of this thesis was to determine if the addition of Anthraquinone to sulphonated CTMP process would increase the optical properties without decreasing the strength properties. Along with the AQ addition, the percent refining done on the fibers was increased to see what effects it would produce on the optical properties.

The pulp used was southern pine and was obtained from American Fibrit Company, Battle Creek, Michigan.

The distinction that sulphonated thermomechanical pulps produce a outstanding material for inexpensive paper such as newsprint has been known for a long time. With the introduction of high brightness newspaper, like USA Today, has come the need for brightness levels of around 60. To reach these higher levels the pulp companies have increased the bleaching process, which produced a lower yield. To correct this problem bleached kraft was added, typically levels around 20-25% have been added. With these high levels of kraft needed an alternative pulp which costs less is needed.

The results showed that with the addition of 1% Na_2SO_3 and 0.2% AQ the optimum optical properties could be attained. This combination produced a brightness increase of 1.5 points and only a slight decrease in opacity.

LITERATURE SEARCH

The properties of thermomechanical pulps in the production of inexpensive paper is well known. It has been used for many years in the production of tissue, newsprint, and board. The main reasons for the large use of TMP has been its high yield, excellent opacity, lower capital cost and lower environmental impact. Along with these advantages, are the production of a greater percentage of long fibers and less shives.[1] Also associated with TMP are the disadvantages of low brightness stability and poor bleachability.

With the current trends in the industry going towards high yield pulps with final high optical properties the process of post-treatment chemical addition was chosen. Sulphonation of thermomechanical pulp using sodium sulfite was chosen for this process.

SULPHONATION TREATMENT:

It was shown in a thesis done by Alfonso Bellos that the sulphonation treatment of CTMP increases bonding strength significantly without a large yield loss. Mr. Bellos concluded that the optimum temperature and cooking time was achieved at 120° C and 40 minutes respectively.

In another study done on sulphonation, Mr. Gummers concluded that sulfonation was shown to increase pulp strength by chemical modification in contrast to the purely physical changes brought about by alkaline swelling agents.[18] He also determined the optimum range to run the cooking pH, it was shown to be between 7-8 pH. Increasing the pH beyond this point produced increased fiber strength but lowered the optical properties significantly. Another negative effect of increasing pH was that the cohesiveness of the pulp decreased mainly due to hemicellulose degradation. Also, the sulphonation of CTMP lignin offered improved fiber flexibility and bonding potential with a minimum yield loss.[4,8]

The strength properties of sulphonated CTMP fibers are promoted mainly by the sulphonation of lignin, because of this sulphonation is quite extensive in the CTMP process. After sulphonation, the fibers are more hydrophilic, softer and more swollen.[8] Which directly correlates to the fact that the fibers are of high conformability and relatively good cohesiveness.

Concerning the area of consistency, it was shown by Skiney that a consistency of 10% produced a high degree of sulphonation in a short amount of time.[1]

Along with the sulphonation of the CTMP fibers it was decided to use soluble Anthraquinone (SAQ) in the pulping process. The reason being that with the addition of SAQ to the sulphonated CTMP process the cooking time and yield

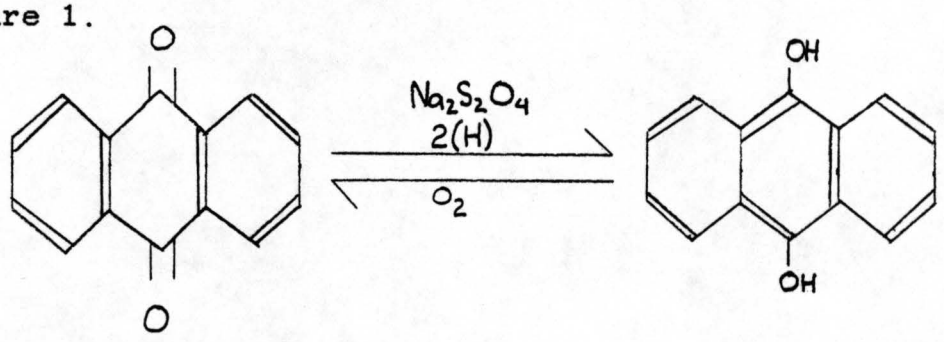
could be increased. The effect of the addition of SAQ to the optical properties will be examined in this

THEORY OF ANTHRAQUINONE PULPING:

Anthraquinone pulping has been greatly researched and the following is a description of the processes involved in AQ pulping and the generalized results produced by these studies. Anthraquinone has been known for a long time for its ability as a catalysis is such processes as removal of oxygen traces from nitrogen gas. The use of AQ for the pulping process has been tested quite extensively by numerous professionals but, Lowendahl and Samuelson have produced the most extensive testing and results.[5]
In their study of chemical behavior of AQ it was shown that with the addition of 0.1% and 1% AQ, as compared to the blank run, the pulping of Scotch pine produced two types of acid end groups: (1) the peeling-aggravating acids, which accelerate the peeling reaction in the pulping process, and (2) the formation of aldonic acids, which are relatively stable under pulping conditions. The results of this study can be seen in Table I.

Based on their study, Lowendahl and Samuelson determined that the oxidation of carbohydrate end groups in cellulose and hemicellulose by AQ to aldonic acids renders them more stable toward alkiline degradation.[8] In turn, the AQ becomes reduced to anthrahydroquinone.

The anthrahydroquinone then reacts with the lignin to reduce the lignin to soluble fragments, which then oxidizes back to AQ. The AQ is then free to react in the pulping process again. The reaction of AQ in the pulping process is shown in Figure 1.



ANTHRAQUINONE ANTHRAHYDROQUINONE

Lowendahl and Samuelson concluded that, the stabilization of the carbohydrates against alkaline peeling by oxidation of reducing end groups to aldonic end groups and the catalyzed delignification of cellulose are greatly affected by the addition of AQ. This was shown in the lower required cooking temperature, lower chemical demand, lower energy demand and improved pulp quality.[9] Also noted was that the use of AQ needed no special equipment and is not corrosive.

With the use of anthraquinone in the sulphonated CTMP pulping process a high yield, good bleachability grade of pulp will result. The strength properties of this type of pulp will also be quite comparable with kraft.

PRESENTATION OF PROBLEM

With the ever increasing demand for better fiber quality the industry has looked towards increasing existing plant pulping facilities. In the high yield area of pulping the present trend is to include a chemical addition to the pulping process. Current literature reveals that the addition of AQ to the high yield process has not been completely researched and would possibly produce pulp of higher optical and strength properties.

This brings up the question, if AQ was added to a high yield process like sulphonated CTMP would the optical properties increase without decreasing the strength properties? Also, if the CSF conditions were varied on the sulphonated CTMP (SCTMP) would the optical properties increase?

In a thesis presented by Alfonso Bello, he determined that the optical strength properties of southern pine CTMP occurred at the following pulping conditions: 120°C for 30 minutes at a pH of 8. This thesis will continue the work of Alfonso Bello at the optimum strength conditions by adding SAQ to the pulping process and varying the degree of CSF on the pulp. By changing these variables the optimum optical conditions will be determined.

EXPERIMENTAL PROCEDURE

In order to do this thesis southern pine (spruce) chips were obtained from the American Fibrit Company, Battle Creek, Michigan. The chips were stored at Western Michigan University Pilot Plant.

PRECOOK PROCEDURE:

The chips were first prepared for testing by removing a large portion from the barrel and mixing the chips to produce an even moisture content. The reason for this was due to the long length of time which the chips had sat in the barrel.

Once mixing was complete a representative sample was taken and a moisture content determination made. This was done by placing a 100 gram sample into the oven at 100°F for 24 hours. After the moisture content was determined 18 different 100 gram O.D. samples were weighted out.

Next, the Amco Oil Bath Digester was prepared for pulping. The oil bath digester consists of six individual steel cylinders which are filled with chips and cooking liquor. These individual cylinders are placed into a holding rack and lowered into hot oil. The hot oil is the medium by which the cooking takes place. The rack will rotate for the entire pulping time. The digester was turned on and set to 120°C. Once the

temperature was reached the digester was set to auto. While the digester was being heated up the cooking liquor was made.

LIQUOR MAKEUP:

The cooking liquor for each bomb was made up at a 4:1 ratio, liquor to wood. Each bomb consisted of sodium sulfite (Na_2SO_3) alone or sodium sulfite plus AQ, in varing percentages. Water was also be added to the cooking solution to achieve the concentration. Each runs cooking liquor was corrected for pH. A pH of 8 was used. The liquor charge calculations for each bomb were calculated and run according to Table II.

TABLE II

Cook #1	1% Na_2SO_3 + 0% AQ	(control)
	2% Na_2SO_3 + 0% AQ	(control)
Cook #2	1% Na_2SO_3 + 0.1% AQ	
	1% Na_2SO_3 + 0.2% AQ	
Cook #3	2% Na_2SO_3 + 0.1% AQ	
	2% Na_2SO_3 + 0.2% AQ	

Each charge is based on the 100 gram O.D. chip charge per bomb.The cook is held at 120°C for 45 min. at a pH of 8. The pH of the cooking liquor was corrected by using NaOH and H_2SO_4. The consistency was held at a constant 10%.

COOKING PROCEDURE:

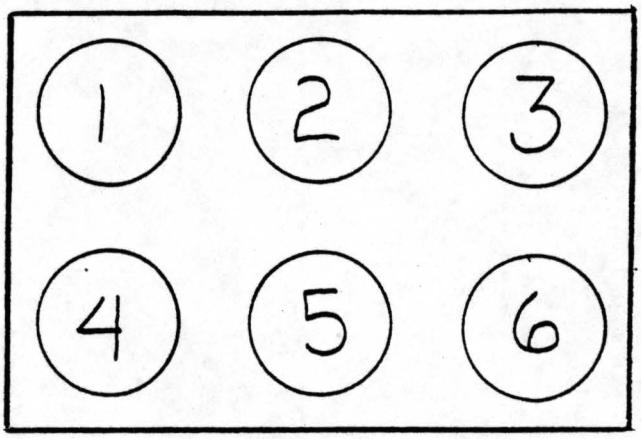
Once the bombs were properly made according to Table II and properly seated into the oil bath rack, see figure 2, the rack was lowered into the digester. At this point the timer was set and the rack rotation turned on. The temperature of the oil was checked every 15 minutes and corrections made if necessary.

After 45 minutes the oil bath was turned off and the rack lifted out by the use of a crane. The rack was then placed into the water cooling tank for 10 minutes. Once the rack had cooled the individual bombs were removed and opened. A sample of the cooking liquor was saved for pH testing later. The chips were then drained on a funnel with hot water to remove black liquor. The chips were placed into 1000 ml beakers with hot water and set into a heated water bath to keep the temperature of the chips as high as possible. The temperature of the water bath was between 130 to 140°F. This was done for all six bombs.

FIGURE 2

DIGESTER BOMB PLACEMENT

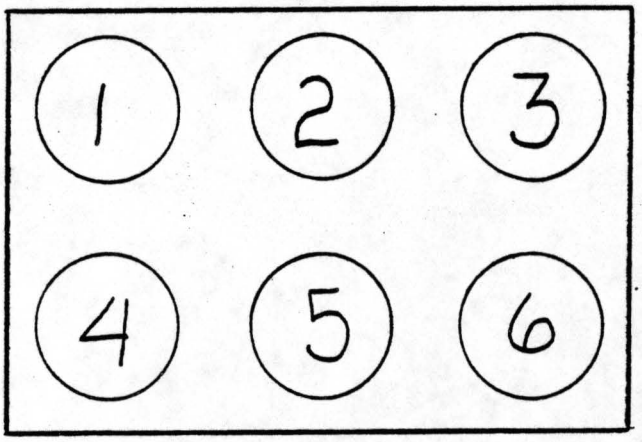
OOK #1



(1-3) 1% Na_2SO_3 + 0% AQ

(4-6) 2% Na_2SO_3 + 0% AQ

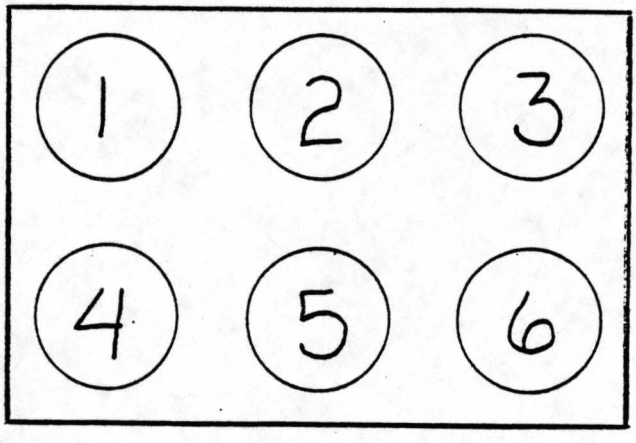
OOK #2



(1-3) 1% Na_2SO_3 + 0.1% AQ

(4-6) 1% Na_2SO_3 + 0.2% AQ

OOK #3



(1-3) 2% Na_2SO_3 + 0.1% AQ

(4-6) 2% Na_2SO_3 + 0.2% AQ

REFINING PROCEDURE:

After the bombs were all emptied the three 100g O.D. samples which had the same cooking liquor were combined into one 300g O.D. sample. Each cook produced two 300g O.D. chip sample. The chips were then taken to the Valley Beater where the consistency was increased to 3 - 4% with hot tap water. The Valley Beater was run for 10 minutes with no weight. This step was used to break down the chips without doing much fiber damage. Once the pulp was done defiberizing it was transferred to the Mead Refiner, which had been heated up with hot water prior to the run. Refining was done for approximately 30, 50, 70, and 90 seconds. This correlates to Canadian Standard Freeness readings of 120, 110, 100, and 90 respectively. The refining time for each pulp varied, so freeness reading were taken frequently. Two pulp samples were collected at each freeness level. Two quart jars were used for pulp sampling, which contained a sufficient amount of fiber to allow for bleaching and handsheet making. A total of 24 samples were taken, four from each cooking condition.

At each freeness level a 500ml pulp sample was removed and unbleached brightness determined. This was achieved by producing a filter pad on the buchner funnel which was used for brightness testing. Also, a Kappa Number test was run on each 120 CSF sample. This was used as a representation of the lignin content removed before and after bleachin. This

test was used to show if the bleaching process produced any fiber or lignin degradation.

BLEACHING PROCEDURE:

The bleaching procedure used on each pulp sample was a low density Sodium Hydrosulfite one step process, as outlined by Virginia Chemicals. The bleaching was run at 120°F for one hour at a consistency of 3%. The pulp was placed into 1000ml beakers in a water bath for the bleaching process. The sodium hydrosulfite addition was equivalent to 15 lb/ton, as used by industry.

The following write-up was provided by Virginia Chemical for the bleaching of softwood pulp using the low density Sodium Hydrosulfite one-step process.

The procedure, as described, results in brightness values which are about 0.7 point (%MgO) lower than the values obtained when the pulp samples are bleached in an oxygen-free systems. Since the masses of pulp treated in mill practice are large in comparison with the surfaces exposed, the effects of air oxidation are reduced to a minimum. Mill applications, in most cases, result in brightness values higher than those produced in laboratory tests.

To insure adequate stability, one liter of bleaching solution was prepared and used within one day. A one percent solution was recommended and prepared as follows:

1. Dissolve exactly 10.0 grams of V-Brite in 990ml of deaerated, distilled water contained in a one-liter small-necked Erlemeyer flask. Beating air into the solution was avoided. Cover the exposed surface with a thin layer of mineral oil to reduce exposure to air and stopper the flask.

The actual bleaching procedure was run as follows:

1. Weight accurately about 800 grams of pulp of known consistency (range 3 to 5%) into a 1000ml beaker.

2. Bring the temperature of the pulp to 5°F above the desired bleaching temperature in a water bath.

3. Mix the required amount of bleaching solution, added from the 50ml burette into the pulp. An egg beater-type mixer, equipped with twin beaters, is recommended for blending the chemical with the pulp. The speed should be set so the V-Brite solution is rapidly folded into the pulp and thoughly mixed without beating in air. After the solution has been added, mix the pulp an additional 15 seconds while rotating the beaker slowly around the agitator blades.

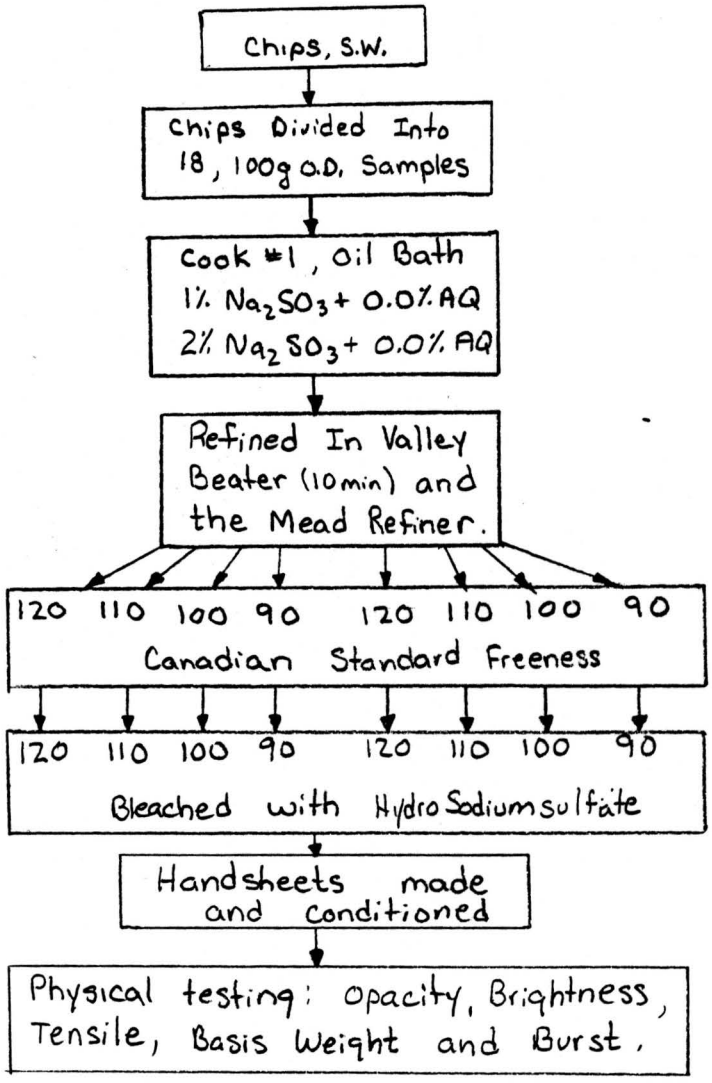
4. Cover the beaker with a watch glass and place in a constant temperature bath at the desired bleaching temperature for the reaction period.

5. After the bleaching period, remove the sample from the water bath. Discard the top 1.5 inches of pulp, which does not bleach to the full extent because of cooling and surface oxidation. Sample the remainder of the pulp for pH measurement, if desired, and for sheetmaking.

Once the bleaching was complete the pulp sample was removed from the water bath and drained on the buchner funnel. A sample from the 120 CSF pulp was used for the Kappa Number testing. The pulp was placed into tared plastic bags and percent moisture determined on each sample. The samples were stored in cold storage overnight while the moisture was determined. This moisture calculation was used for handsheet slurry determinations.

FIGURE 3

Experimental Work



This was repeated for each cook (1, 2, and 3)

EQUIPMENT DIRECTORY

1. AMCO OIL BATH
2. PULPING LAB WATER BATH
3. BUCHNER FUNNEL AND FILTER PAPER
4. ANALYTICAL BALANCE
5. OVEN AT A CONSTANT TEMPERATURE OF 105°C
6. THIRTY PLASTIC BAGS AND LABLES
7. TWO 50ml BURETS
8. 2ml PIPETS
9. FOUR BUCKETS
10. pH METER
11. MIXER WITH HEATER
12. 150 AND 10ml BEAKER
13. 1500ml GRADUATED CYLINDERS
14. EYE DROPERS (2)
15. ALUMINUM TRAYS (4)
16. 1000ml BEAKERS WITH LIDS (4)
17. GLASS STIRRING RODS
18. VALLEY BEATER
19. MEAD REFINER
20. NOBLE AND WOOD SHEET MOLD
21. CSF TESTER
22. TENSILE TESTER
23. BRIGHTNESS AND OPACITY TESTERS
24. BURST TESTER
25. IBM COMPUTER

CHEMICAL LISTING

1. Anhydrous Sodium Sulfite, Na_2SO_3
2. Deionized Water
3. Soluble Anthraquinone
4. Sulfuric Acid, H_2SO_4
5. Sodium Hydroxide, NaOH
6. Iodine Solution of Known Normality (.1N)
7. Sodium Thio Sulfate, $Na_2S_2O_3$
8. Starch Indicator
9. Sodium Hydrosulfite

DESIGN PARAMETERS

The sequence of cooks which were run on southern pine CTMP are sepcified in Figure 4. A code list is specified in Table III.

The different conditions under which the pulp was produced is as follows:

1. A pH level of.....8
2. Level of Sodium Sulfite.....1 and 2%
3. Level of Antraquinone.....0.0, 0.1 and 0.2%
4. Oil Bath temperature.....120°C
5. Oil Bath time.....45 minutes

* Step 2 and 3 are basd on O.D. wood.

The following is a list of conditions under which the pulp was tested:

1. Defibered in Valley Beater...10 minutes
2. Refined in Mead Refiner to...120, 110, 100, and 90 Canadian Standard Freeness.
3. Bleached at 3% consistency with Sodium Hydrosulfite for one hour.
4. Bleaching temperature.....120°F

The main purpose is to see the interaction between %AQ and %NA_2SO_3 on the optical properties of sulphonated CTMP handsheets.

Also, the interaction between increased refining and the addition of %AQ and %Na_2SO_3, as it relates to the optical properties of sulphonated CTMP handsheets.

DISCUSSION

PERCENTAGE YIELD

From the Graph 1A it can be observed that with the addition of Anthraquinone the percent yield slightly increased as percent AQ addition was increased. This trend can also be seen at the 1 and 2 percent Na_2SO_3. The is due to the stabilization of the carbohydrates against alkaline peeling by oxidation of reducing end groups to aldonic end groups and the catalyzed delignification of the cellulose fibers.

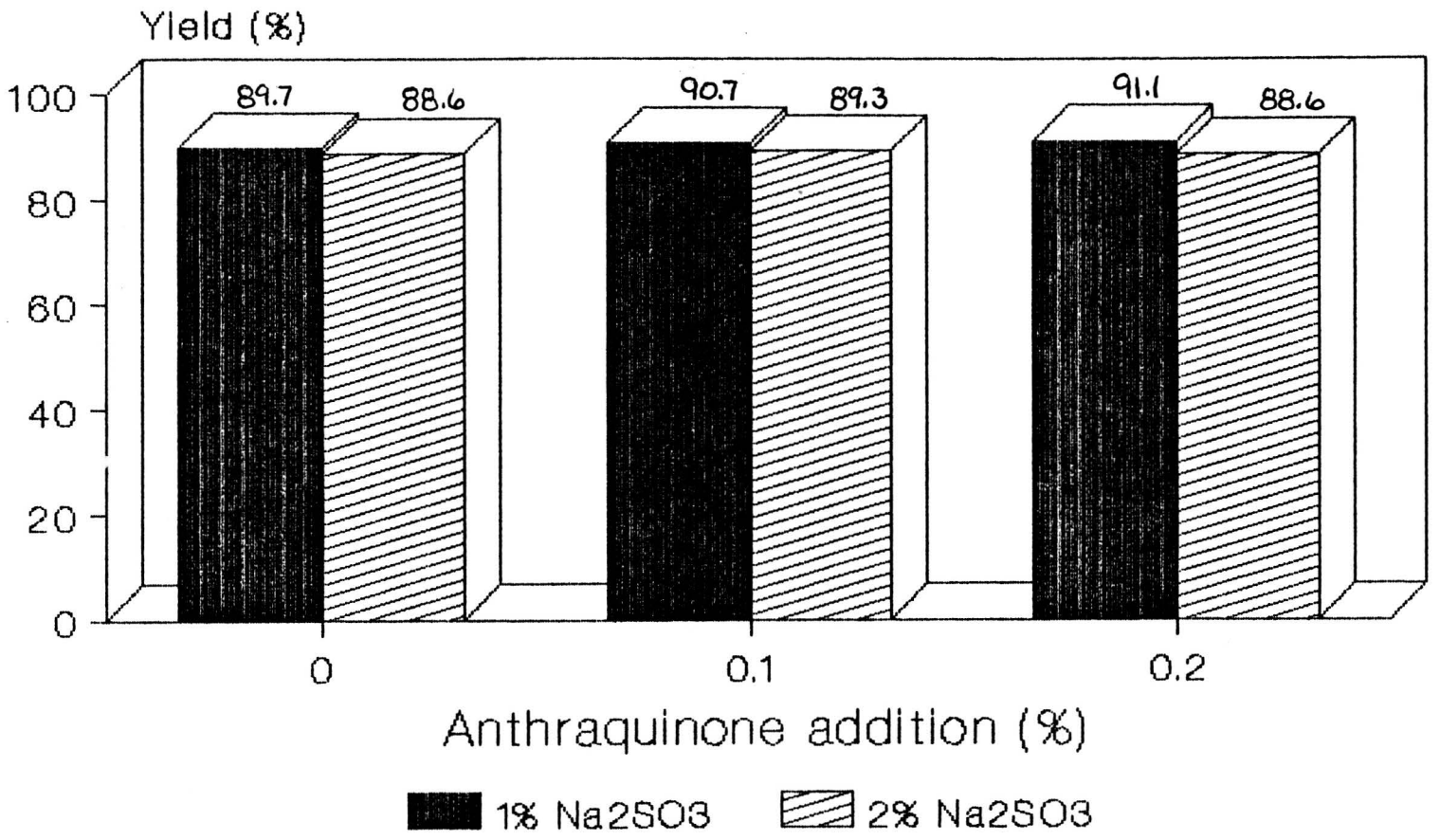
In the comparison between 1 and 2 percent Na_2SO_3 it was shown that as the percent of Na_2So_3 was increased the percent yield decreased by about 1.2%. This is the result of the stronger cooking liquor degrading the cellulose to a greater extent. This can also be seen in Graph 1A.

These trends have been well documented in the past and the results for the percent yield are what would be expected in the industry. This was not the main purpose of the thesis, but was included to confirm the known literature.

BRIGHTNESS

From Graphs 2A - 5A it can be seen that as the degree of refining was incresed the percentage of brightness also increased. This can be seen in the unbleached and bleached sheets. This trend can also be observed as the level of AQ increased in the unbleached ghaphs 2A and 3A. This increase

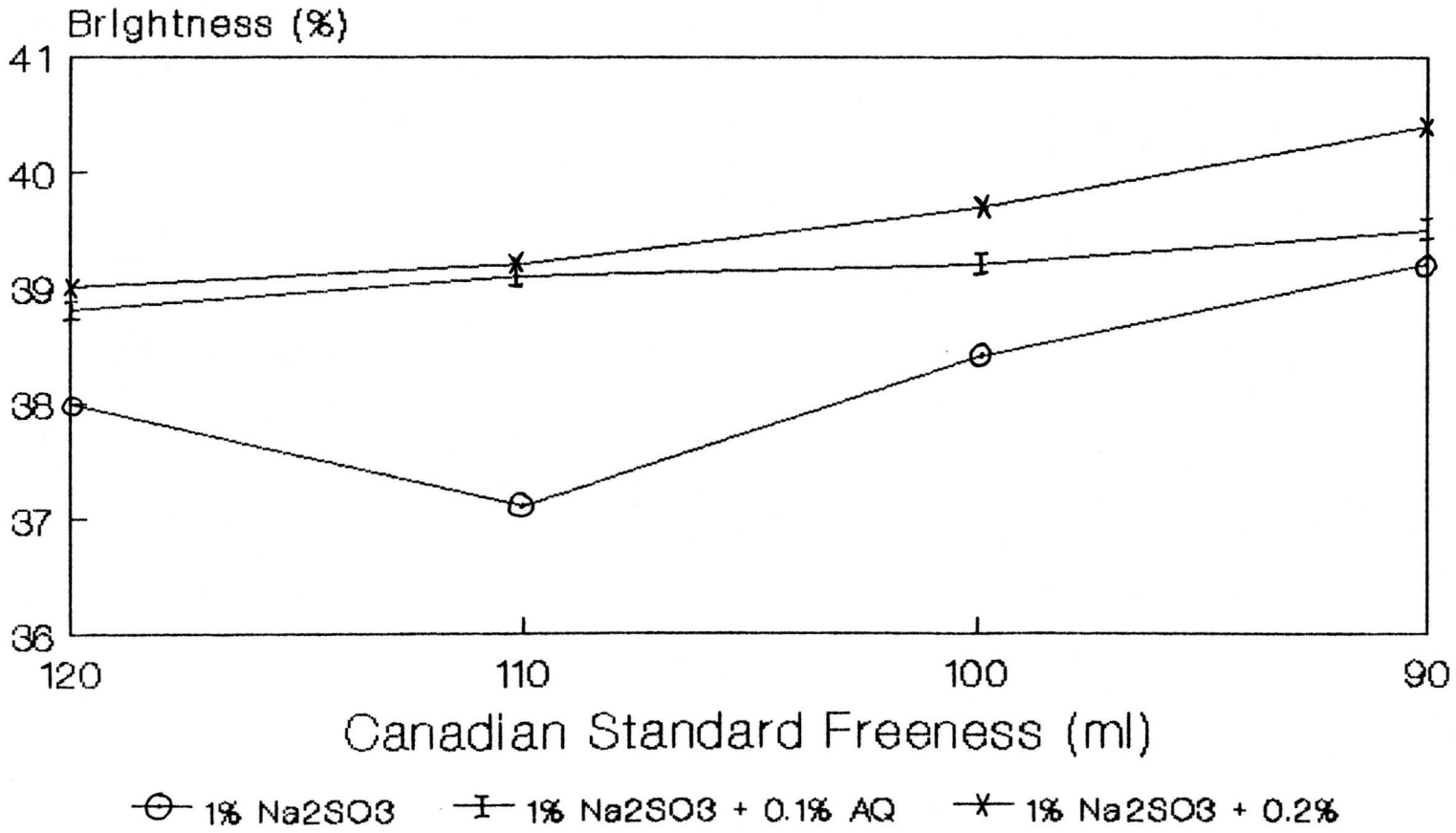
Yield vs. % AQ
unbleached pulp



GRAPH 1A

Brightness vs. CSF

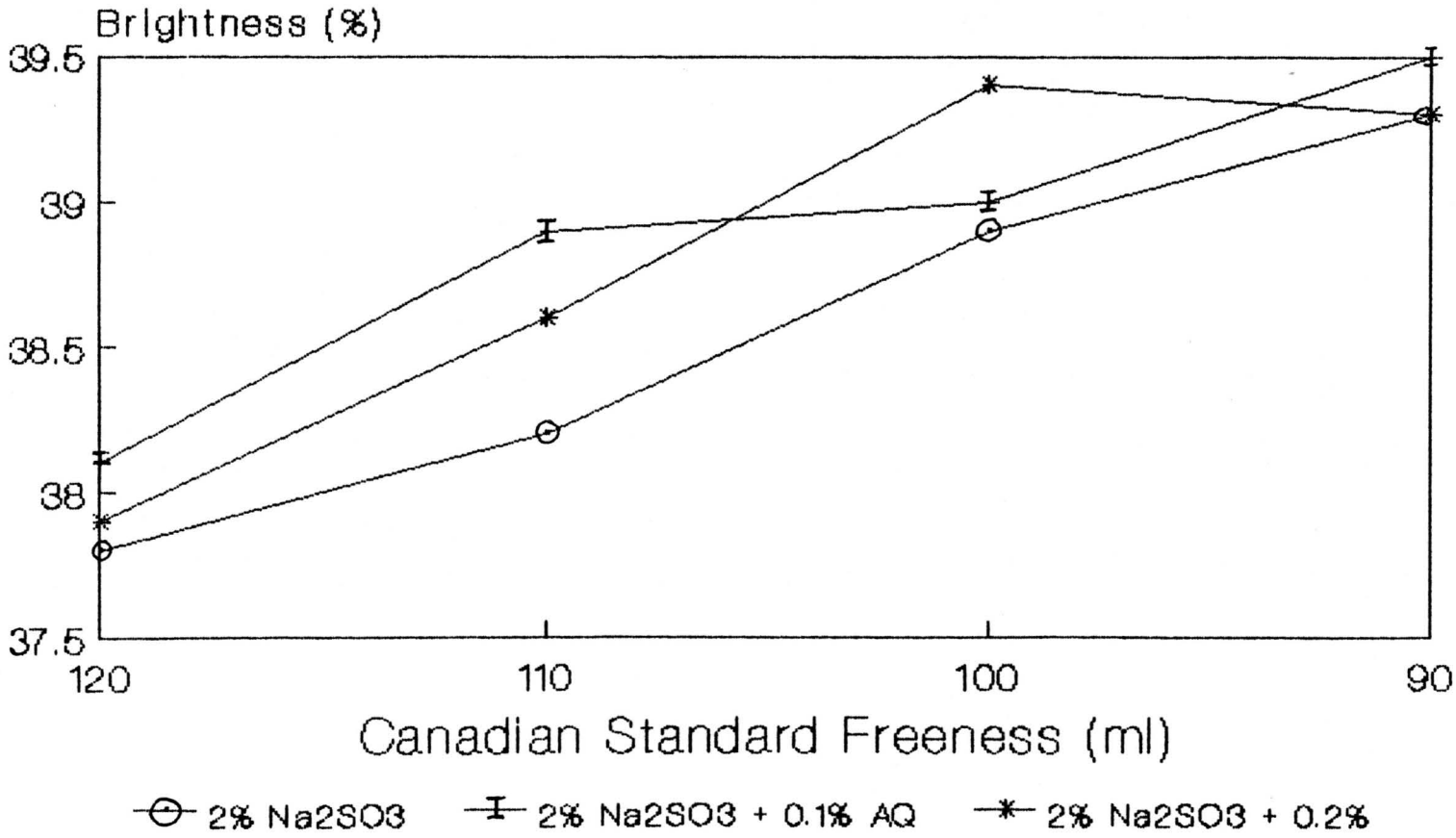
Unbleached Brightness



GRAPH 2A

Brightness vs. CSF

Unbleached Brightness



GRAPH 3A

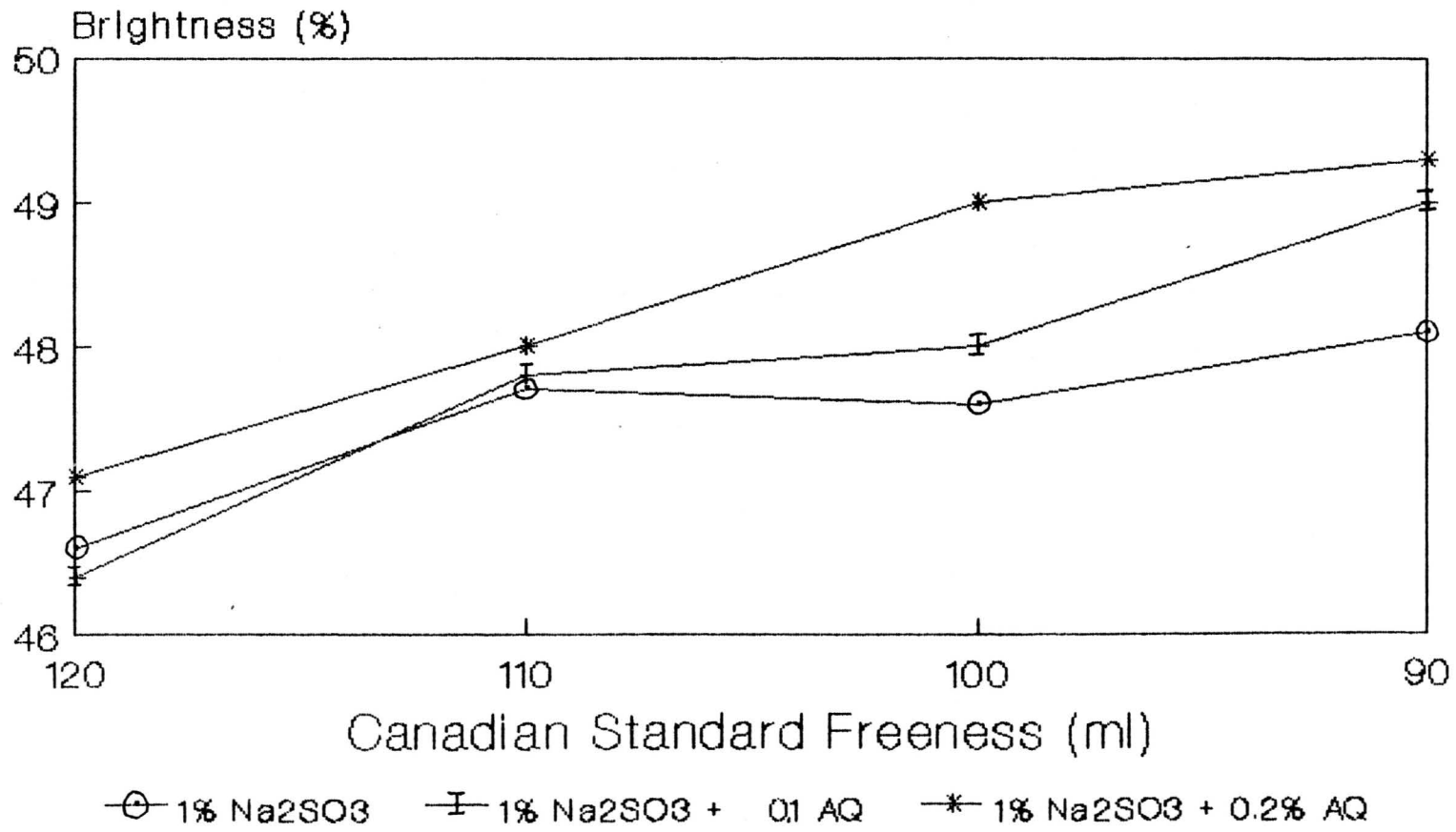
in the unbleached sheets is due to the fact that as the percent AQ was added the degree of delignification also increased. Thus, with the decreased amount of lignin present in the fibers the fibers brightness increased.

The bleached brightness which can be seen in Ghaphs 4A and 5A also increases with increasing percent AQ and increasing refining. The brightness increase which is seen is due to the unbleached brightness increase. The typical average brightness increase with Sodium Hydrosulfite is approximently 10-12 points. The results for the bleached sheets showed an increase of 9 to 10 points. This lower brightness level is due to the bleaching process not being run in an oxygen free enviroment.

The most significant result came from the fact that as the refining increased so did the percent brightness. From the literature on refining it was determined that this trend was due to the high yield pulp becoming more fibralated, which produced a increased surface area from which light could reflect. This increase in light reflectance produced a greater increase in brightness of approximently 1 point. In the bleached sheets a brightness increase of 1.5 points can be seen, again due to the increased surface area producing increased light reflection.

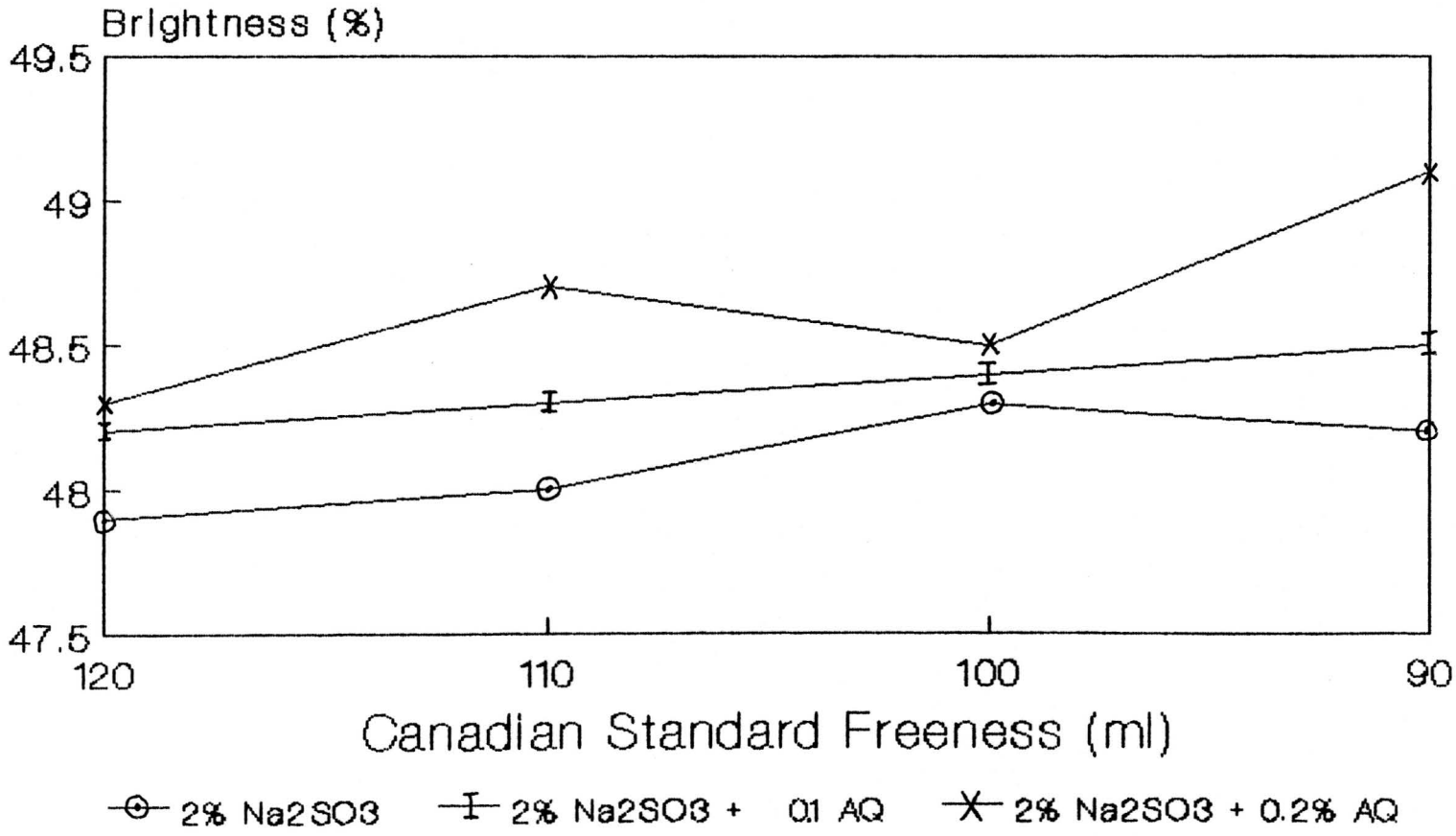
The results of a 1 to 1.5 point increase in brightness is quite significant in the industry and could be used if a lower CSF reading would work on the mills grade of paper.

Brightness vs. CSF
bleached Brightness



GRAPH 4A

Brightness vs. CSF
bleached Brightness



GRAPH 5A

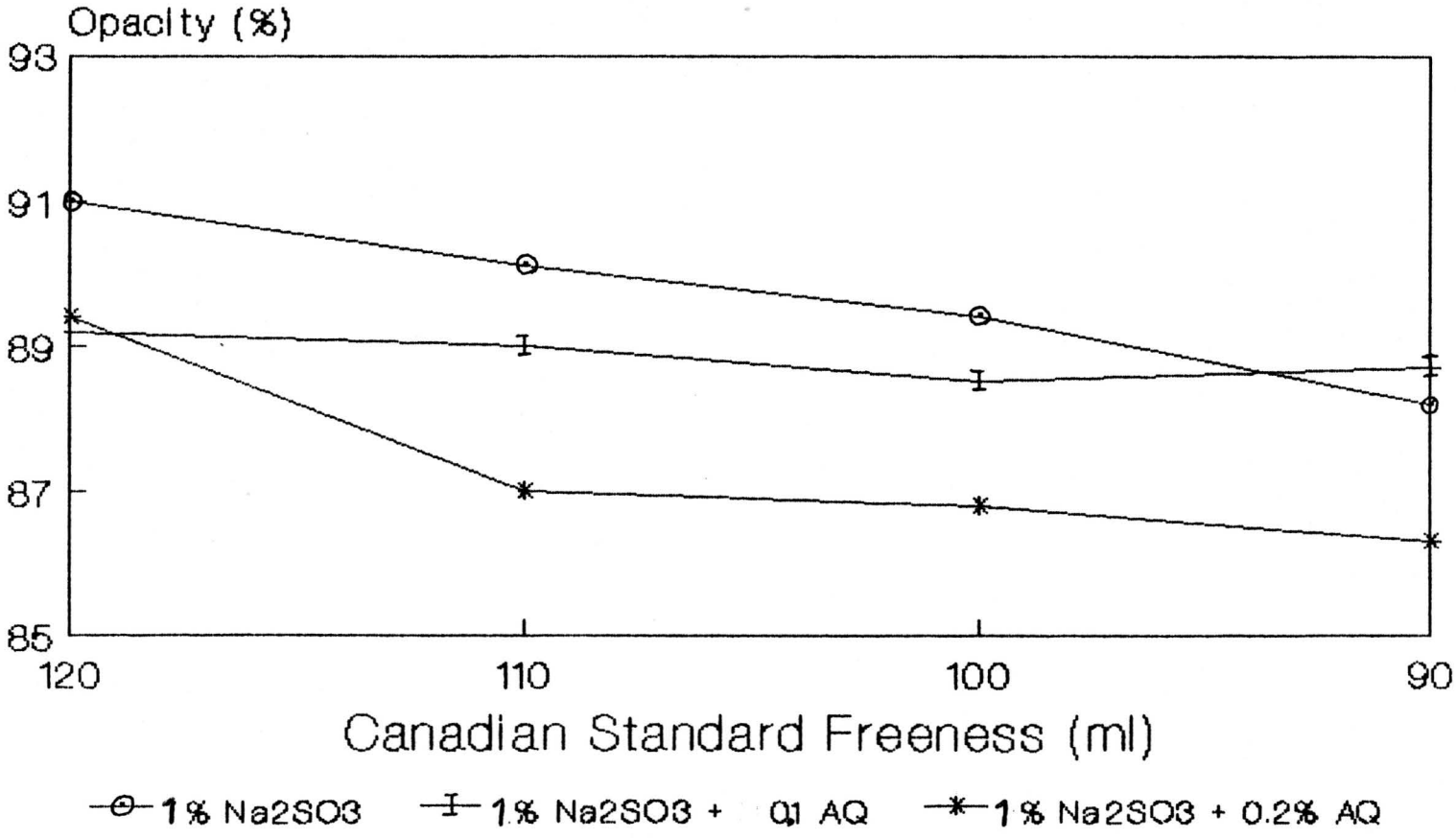
The opacity trends can be seen in Graphs 6A and 7A. The trends for the bleached sheets showed that as the percent AQ increased the percent opacity decreased. The graphs also show that as the refining is increased the percent opacity decreases. This is seen at all three levels of AQ and percent Na_2SO_3. This trend is due to the increase in density and relative bonding area due to the sulphonation of the fibers and the increased refining levels.

STRENGTH TESTS

The tensile and stretch results showed that as the percent AQ incresed, the percent tensile and stretch increased. These results are what was proven in the thesis by Alfonso Bellos, so the results were as expected.

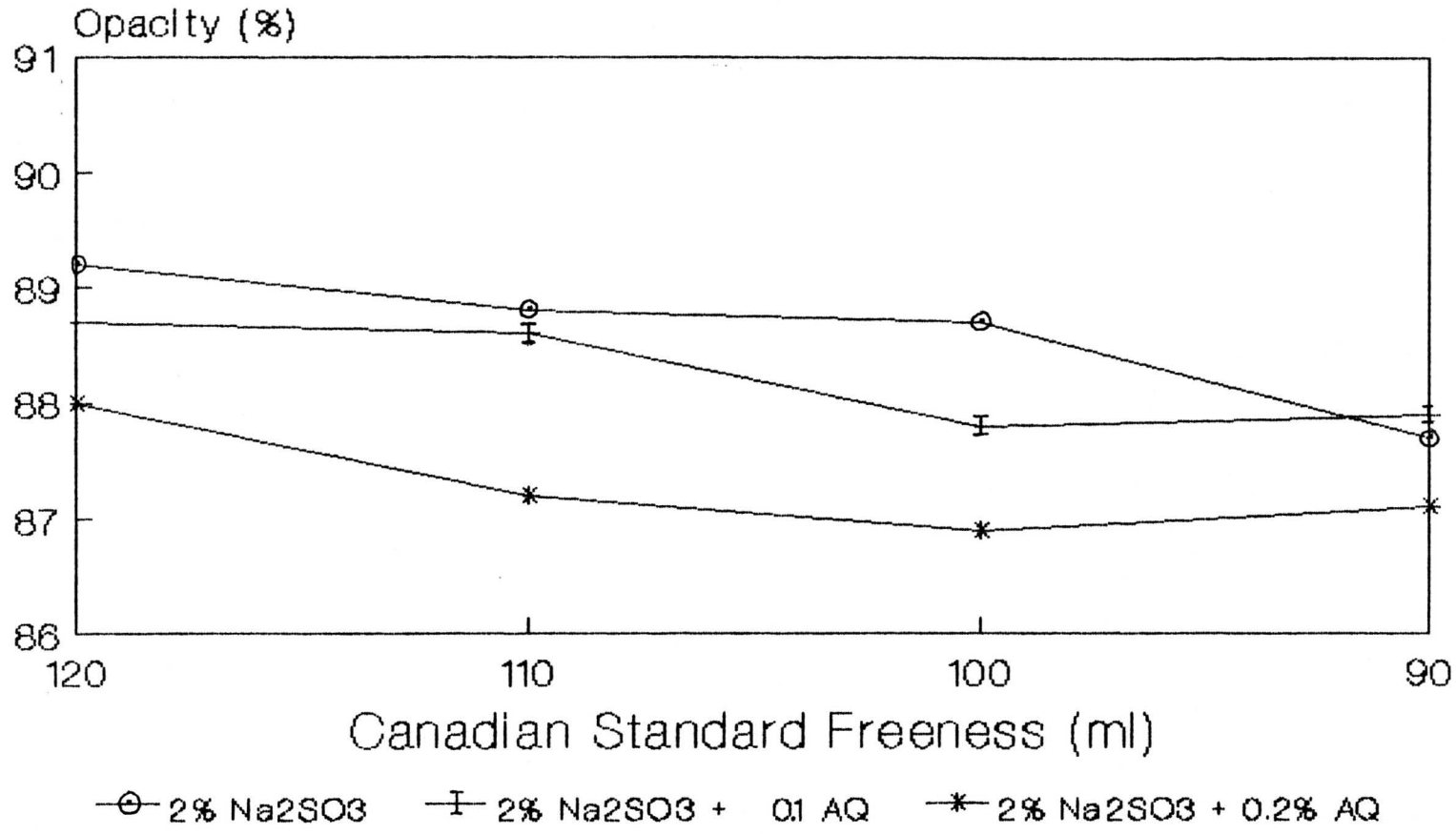
The reason for this increase is due to increased level of comformability and flexibility of the fibers. The main reason is that the fibers tend to swell in the alkaline conditions producing the above conditions.

Opacity vs. CSF
bleached Pulp



GRAPH 6A

Opacity vs. CSF
bleached Pulp



GRAPH 7A

SUMMARY AND CONCLUSIONS

The use of Anthraquinone (AQ) in the pulping process of high yield CTMP produced only a slight increase in percent yield. This slight increase in yield was not statistically analyzed and so no concrete conclusions can be drawn. In order to determine if AQ has any significant effects on CTMP multiple cooks must be produced, giving a more complete spread of data.

The addition of AQ did produce a increase in percent brightness and a slight decrease in percent opacity. This was achieved without a significant loss in strength.

The brightness increase was seen at each level of AQ addition, which was mainly due to the lower lignin content in the fibers allowing the fibers to bleach easier.

When looking at the results for each level of AQ the difference between the 1 and 2 percent Na_2SO_3 with the addition of 0.2 percent AQ was quite close. From these results it would be most likely that a mill would use the 1 percent Na_2SO_3 and 0.2% AQ to achieve the required brightness. The main reason being that lower cost would be achieved by using 1% as comparied to using the 2% Na_2SO_3 with the addition of 0.2% AQ.

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Appendix I (con't)

1% Na_2SO_3 + 0% AQ:

Unbleached brightness:

CSF		%
120	CSF	= 38
110	CSF	= 37.1
100	CSF	= 38.4
90	CSF	= 39.2

2% Na_2SO_3 + 0% AQ:

Unbleached brightness:

120	CSF	= 37.8
110	CSF	= 38.2
100	CSF	= 38.9
90	CSF	= 39.2

Appendix II

BLEACHED BRIGHTNESS

1% Na_2SO_3 + 0% AQ:

CSF	%
120	= 46.6
110	= 47.7
100	= 47.6
90	= 48.1

2% Na_2SO_3 + 0% AQ:

120	= 47.9
110	= 48.0
100	= 48.3
90	= 48.2

1% Na_2SO_3 + 0.1% AQ:

120	= 464
110	= 47.8
100	= 48.0
90	= 49.0

2% Na_2SO_3 + 0.1% AQ:

120	= 48.2
110	= 48.3
100	= 48.4
90	= 48.5

1% Na_2SO_3 + 0.2% AQ:

120	= 47.1
110	= 48.0
100	= 49.0
90	= 49.3

2% Na_2SO_3 + 0.2% AQ:

120	= 48.3
110	= 48.7
100	= 48.5
90	= 49.1

Appendix III

OPACITY %

1% Na_2SO_3 + 0.0% AQ:

CSF	=	%
120	=	91.0
110	=	90.1
100	=	89.4
90	=	88.2

2% Na_2SO_3 + 0% AQ:

120	=	89.2
110	=	88.8
100	=	89.4
90	=	88.7

1% Na_2SO_3 + 0.1% AQ:

120	=	89.2
110	=	87.0
100	=	88.5
90	=	88.7

2% Na_2SO_3 + 0.1% AQ:

120	=	88.7
110	=	88.6
100	=	87.8
90	=	87.9

1% Na_2SO_3 + 0.2% AQ:

120	=	89.4
110	=	87.0
100	=	86.8
90	=	86.3

2% Na_2SO_3 + 0.1% AQ:

120	=	86.5
110	=	87.2
100	=	86.9
90	=	87.1

Appendix IV

TENSILE STRENGTH (M)

1% Na_2SO_3 + 0% AQ:

CSF	(m)
120	2001
110	1981
100	2103
90	2141

2% Na_2SO_3 + 0% AQ:

120	2086
110	2107
100	2187
90	2270

1% Na_2SO_3 + 0.1% AQ:

120	2247
110	2301
100	2289
90	2345

2% Na_2SO_3 + 0.1% AQ:

120	2252
110	2159
100	2247
90	2362

1% Na_2SO_3 + 0.2% AQ:

120	2280
110	2313
100	2311
90	2340

2% Na_2SO_3 + 0.2% AQ:

120	2286
110	2264
100	2240
90	2380

Appendix V

% Stretch

1% Na_2SO_3 + 0% AQ:

CSF		%
120	=	2.48
110	=	2.60
100	=	2.58
90	=	2.51

2% Na_2SO_3 + 0% AQ:

120	=	2.51
110	=	2.6
100	=	2.52
90	=	2.59

1% Na_2SO_3 + 0.1% AQ:

120	=	2.52
110	=	2.4
100	=	2.6
90	=	2.54

2% Na_2SO_3 + 0.1% AQ:

120	=	2.61
110	=	2.35
100	=	2.55
90	=	2.67

1% Na_2SO_3 + 0.2% AQ:

120	=	2.6
110	=	2.78
100	=	2.6
90	=	2.63

2% Na_2SO_3 + 0.2% AQ:

120	=	2.81
110	=	2.61
100	=	2.51
90	=	2.68