Practical experiences in additive screening using a torque-based flocculation analyzer

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ABSTRACT: This paper describes results from in-mill polymer screening experiments using a novel flocculation analysis technique. This method is useful for measuring the effects of typical papermaking additives on the degree of flocculation and the resulting floc's resistance to shear. As a sample is stirred at a constant speed, a retention aid is added and flocs begin to form. These flocs increase the load on the motor which is driving the impeller. Therefore, the degree of flocculation, which is proportional to the increase in load, can be measured in real time. As the impeller gradually destroys the flocs, a decrease in load is detected, which is related to the floc's strength or resistance to shear. Results from various retention aid chemistries and various papermaking furnishes are discussed. We observed sensitivity to product dosage and chemistry and a good correlation with Britt jar retention.

Application: This study describes a method to measure the impact of typical papermaking additives on the degree of flocculation and the resulting floc's resistance to shear. Mill personnel can use this technique to evaluate and monitor polymer reactivity with a given furnish.

Laboratory evaluation of a retention or drainage aid program should be the first step before considering any paper machine process change (e.g., product chemistry, dosage or addition point). Machine time is too valuable and trials are too expensive to be run without some laboratory data (performed with mill furnish) to support the process change. Papermaking process conditions change for many reasons (e.g., pulp mill operation, grade structure, degree of closure, etc.), so a periodic laboratory review of the retention and drainage chemistry should be a component of a technical service program.

Laleg and Collins [*1, 2*] recently described a technique for evaluating polymer performance in papermaking furnishes using a torque-based flocculation analyzer. We have modified the technique and used it successfully "in the field" to identify optimal polymer solutions with a wide variety of furnishes. This report describes the technique and highlights its advantages compared to conventional methods.

EXPERIMENTAL METHODOLOGY

Figure 1 shows the flocculation analyzer used to collect data for this paper. It consists of three modules: (1) a measurement unit, (2) a control unit, and (3) a personal computer. With a notebook computer the device is portable and can be set up on about 3 ft. of laboratory bench space.

Figure 2 shows the typical measurement cycle. An experiment begins by adding an aliquot of furnish (usually 250 mL) to the sample cup. The sample is placed in the measurement unit and the three vaned impeller is lowered into the sample cell. The impeller is then started using the Windows®-based software. As the impeller rotates in the slurry material, a load cell attached to the motor detects the resistance. The load detected is a function of sample volume, impeller speed, temperature, and the consistency of the sample. The experiments described in this work began with a 300 rpm mix for 15 s followed by a 200 rpm equilibration period (15 s) before injecting the first chemical. We mixed samples for 10 s between chemical additions and for 80 s after adding the final chemical. Since all of the experimental parameters are constant (e.g., sample volume, impeller speed, and temperature), the initial steady-state output detected by the instrument's load cell (expressed in Newtons) is a measurement of the sample consistency.

SENSITIVITY	OUTPUT SIGNAL (mV)	LOAD (gram-force)	LOAD (newtons)
S1	0 – 5000	0 – 43	0 – 0.422
S2	0 – 5000	0 – 86	0 – 0.843
S3	0 – 5000	0 – 177	0 – 1.736
S4	0 – 5000	0 – 444	0 – 4.354

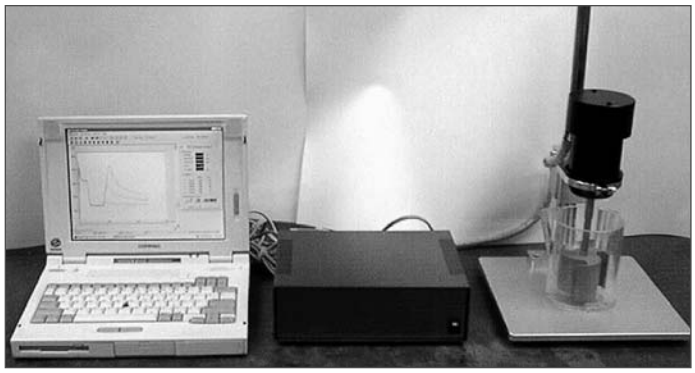
I. Flocculation analyzer output ranges.

The flocculation analyzer reports the force required to maintain the preset impeller speed in mV units. These units can be converted to more standard units of force (Newtons) with the conversion factors in **Table I**, depending on the sensitivity range selected.

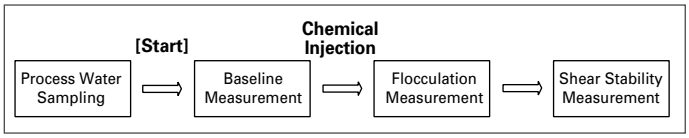
We collected the data described in case studies A and B below using the flocculation analyzer's maximum load/minimum sensitivity setting (S4). The remaining studies were all done using the S1 sensitivity range. **Figure 3** represents a typical flocculation response curve. After thoroughly mixing the sample and allowing it to return to a steady state condition, we applied the chemical treatment program as indicated by point A. The system's load cell immediately detects any flocculation that occurs, which is reported as a function of time. The maximum height of the curve, point B, is a measure of flocculation intensity. As the experiment continues, the impeller gradually destroys the formed flocs and the load cell detects decreasing resistance. So, the floc strength or the floc's "resistance to shear" is related to the area under the curve C. To facilitate the comparisons, all of the curves were normalized to a constant baseline of 1 Newton at 30 seconds. Since the flocculation of the sample is immediately detected and reported, chemicals can be added to the furnish on the same time scale as in the process, and the sequential impact of

CASE STUDY A: NORTHEASTERN PRINTING PAPER MILL BELT PRESS	
Furnish	88% primary sludge, 12% secondary sludge
Consistency	3.2%
pH	7.8
Current program	low-charge cationic polyacrylamide (AE2) @ 3 lbs/ton
CASE STUDY B: NORTHEASTERN RECYCLED TISSUE MILL BELT PRESS	
Furnish	100% primary sludge
Consistency	6.2%
Current program	medium-charge cationic polyacrylamide (BE1)
CASE STUDY G: MID-ATLANTIC VIRGIN PRINTING PAPER MACHINE	
Furnish	28% virgin softwood, 78% virgin hardwood, 13% PCC
Fines	42% fines (by Britt jar fractionation)
Cationic demand	18.5 µeq/L
Consistency	0.53%
Conductivity	700 µmho/cm
Current program	starch, cationic modified AKD, alum, anionic flocculant

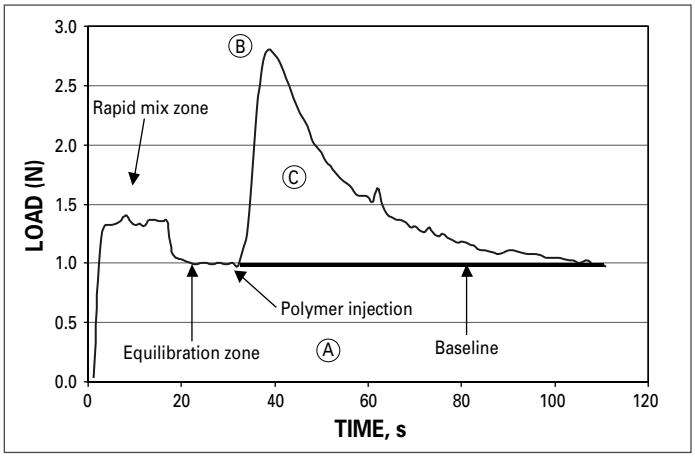
II. Summary of case studies presented in this paper.



1. A photo of the flocculation analyzer.



2. The typical measurement cycle.



3. A typical flocculation response curve.

each additive can be monitored. Although we did not utilize this method in our experiments, one could also vary the shear (impeller speed) as a function of time to simulate the stock moving through high and low shear environments.

Traditional methods of measuring retention and drainage were performed concurrently during some of the experiments for comparison. These methods included the Modified Schopper-Riegler (MSR), the Canadian Standard Freeness (CSF) and the Dynamic Drainage Jar (DDJ). The procedures for using these methods, briefly reviewed here, are described in [*3*]. An experiment with the MSR starts by pouring a 1 L sample of treated stock into the sample chamber with the plunger in the down position. The plunger is then released, and the time it takes to drain an aliquot of filtrate is recorded. Next, the consistency of the filtrate is determined. Canadian Standard Freeness tests were performed using the TAPPI Method [*4*]. Likewise, the procedure for evaluating retention using the DDJ is described in references [*5, 6*].

Traditional methods of measuring free drainage in belt press sludge samples were also performed for comparison with the flocculation analyzer. The free drainage experiments are performed by pouring 250 mL of treated sludge onto a piece of belt press fabric supported by a Buchner funnel with 0.25 in holes. The filtrate is collected underneath the funnel, and the amount of filtrate that drains through the fabric in a given amount of time is recorded as the free drainage. The free drainage time is selected based on the time the sludge is in the free drainage zone of the belt press.

All of the flocculants used in the following research are described generically (XX#), in which the first letter refers to the case study and the second refers to the physical form of the product (e.g., E=emulsion, S=solution, D=dispersion, P=powder). The coagulants are described generically (X#), in which the letter refers to the case study.

RESULTS AND DISCUSSION

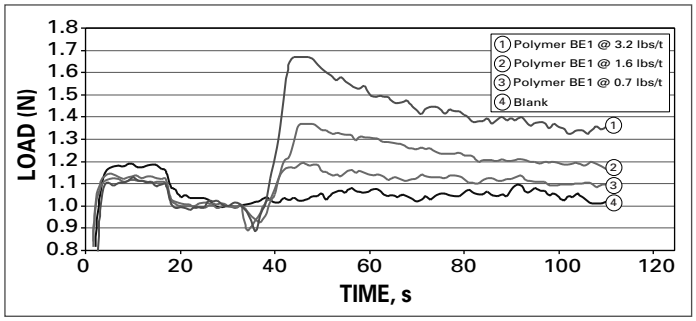
This section presents results from three case studies obtained using our flocculation analyzer in the field (see **Table II**). All seven case studies are presented in the 2001 TAPPI Papermakers Conference Proceedings [7]. The results demonstrate the unit's sensitivity to changes in polymer dosage and polymer chemistry. The results also demonstrate the wide range of consistencies the unit can analyze.

Case study A

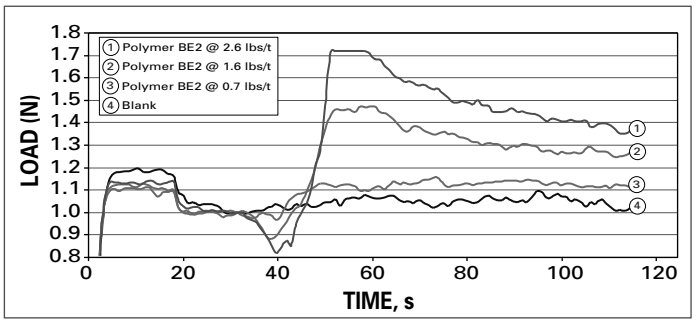
We performed the first set of polymer screening experiments on waste-treatment sludge from a northeastern printing paper mill. The sludge is composed of 88% primary clarifier solids and 12% secondary clarifier solids (Table II). The solids are concentrated in a thickener and fed to a belt press at approximately 3% solids. The current chemical program consists of a low-charge cationic polyacrylamide (AE2) fed at 3 lbs/ton. The flocculation experiments were run in the manner described above. Portions of the results appear in [7]. These data gave the mill confidence to run two month extended trials with each of the chemicals.

Case study B

We performed the second set of polymer screening experiments on primary clarifier sludge from a northeastern recycled tissue mill (**Table II**). The sludge feeds to a belt press at approx-



4. Case study B – Current polymer treatment at three dosages. The graph shows the reproducibility of the data prior to chemical injection and the stronger response at higher dosages.



5. Case study B – Recommended polymer treatment at three dosages. The graph shows the maximum measurable response at the 2.6 lb/ton dosage in which the peak is "clipped."

imately 6% solids. The current chemical program consists of a low-charge cationic polyacrylamide (BE1) fed at 3 lbs/ton. The experiments were run in the manner described above. **Table III** lists a portion of the results. **Figure 4** shows the dosage response curve for the existing chemical treatment program. **Figure 5** shows the improved results with the recommended polymer BE2, which is also a medium charge cationic polyacrylamide. This case demonstrates the effective maximum consistency for the instrument, as can be seen by the clipped peak for the high dosage response in Fig. 5. Experiments 4 and 5 are replicates and their responses are within 5% for both peak height and area under the curve.

The 35 experiments described in Table III cover a broad range of polymer chemistries. The cationic charge and degree of branching is crudely described in the two right-most columns of Table I. Multiple linear regression analysis was performed on the data, and equations to predict peak height and area were generated using three variables (e.g., dosage, cationic charge and degree of branching). The equations have correlation coefficients, R = 89% and 90% respectively. In other words, the three independent variables account for 80% of the variability in the flocculation analyzer data. The other 20% results from experimental error and polymer features not described by the three variables in Table III.

RUN NO.	POLYMER	DOSE (lbs/t)	PK HEIGHT (N)	AREA (N-s)	RELATIVE CHARGE	RELATIVE BRANCHING
1	Blank		0.09	3.55	–	–
2	Polymer BE1	0.6	0.19	8.84	–	–
3	Polymer BE1	1.6	0.37	17.18	–	–
4*	**Polymer BE1**	**3.2**	**0.67**	**30.69**	**–**	**–**
5*	**Polymer BE1**	**3.2**	**0.67**	**31.73**	**–**	**–**
6	Polymer BE2	0.6	0.15	8.29	40	1
7	Polymer BE2	1.6	0.46	21.07	40	1
8	Polymer BE2	2.6	0.70	31.04	40	1
9	Polymer BE3	0.6	0.28	13.64	20	1
10	Polymer BE3	1.6	0.54	25.51	20	1
11	Polymer BE3	3.2	0.72	35.26	20	1
12	Polymer BE4	0.6	0.13	7.44	25	1
13	Polymer BE4	1.6	0.26	14.15	25	1
14	Polymer BE4	3.2	0.56	27.31	25	1
15	Polymer BE5	0.6	0.09	5.30	30	4
16	Polymer BE5	1.6	0.18	9.65	30	4
17	Polymer BE5	3.2	0.22	11.57	30	4
18	Polymer BE6	0.6	0.14	6.50	45	4
19	Polymer BE6	1.6	0.37	17.71	45	4
20	Polymer BE6	3.2	0.60	27.79	45	4
21	Polymer BE7	0.6	0.08	4.52	10	3
22	Polymer BE7	1.6	0.13	6.47	10	3
23	Polymer BE7	3.2	0.30	15.86	10	3
24	Polymer BE8	0.6	0.12	4.81	10	2
25	Polymer BE8	1.6	0.34	16.58	10	2
26	Polymer BE8	3.2	0.45	21.84	10	2
27	Polymer BE9	0.6	0.09	3.74	20	3
28	Polymer BE9	1.6	0.15	7.46	20	3
29	Polymer BE9	3.2	0.30	16.55	20	3
30	Polymer BE10	0.6	0.22	11.30	20	2
31	Polymer BE10	1.6	0.37	20.12	20	2
32	Polymer BE10	3.2	0.65	30.26	20	2
33	Polymer BE11	0.6	0.20	9.58	10	1
34	Polymer BE11	1.6	0.48	19.99	10	1
35	Polymer BE11	3.2	0.71	30.20	10	1

*The current polymer program at the current dosage

III. Case study B data showing the flocculation analyzer responses for 11 polymers at three dosages.

Case study G

Case Study G contains data collected during a retention and drainage program audit at a mid-Atlantic mill that makes printing paper (Table II). The set of experiments listed in **Table IV** is a portion of the work that was performed. Polymers GE1-GE5 are anionic polyacrylamide polymers with increasing charge density. Polymer GE6 is the product that is currently being used, and GE7 and GE8 are high molecular weight/low charge cationic polyacrylamide polymers.

The current program consists of 15 lbs/ton of cationic starch and 3 lbs/ton of cationic modified alkyl ketene dimer (AKD) added to the thick stock. Next, 2 lbs/ton of alum is added followed by 1.2 lbs/ton of anionic polyacrylamide, GE6, just before the headbox. We simulated this by: (1) a 15 s rapid mix phase, (2) a 15 s equilibrium phase, (3) starch/AKD addition followed by a 10 s mix, (4) alum addition followed by another 10 s mix, and (5) flocculant addition.

Figure 6 shows the flocculation analyzer response curves for four different

RUN NO.	ALUM DOSE (lbs/t)	FLOCCULANT POLYMER	FLOCCULANT DOSE (lbs/t)	BRITT JAR FINES RET	PEAK HEIGHT	AREA
1	0	blank	0.0	–	0.002	0.025
2	2	blank	0.0	71%	0.002	0.064
3	2	GE1	0.6	86%	0.004	0.175
4	2	GE1	1.3	87%	0.009	0.267
5	2	GE1	1.9	79%	0.013	0.488
6	2	GE1	2.5		0.017	0.611
7	0	GE2	1.9		0.033	1.396
8	2	GE2	0.6	92%	0.016	0.656
9	2	GE2	1.3	90%	0.018	0.888
10	2	GE2	1.9	92%	0.040	1.672
11	2	GE2	2.5	–	0.043	1.863
12	4	GE2	1.9	–	0.024	0.949
13	6	GE2	1.9	–	0.026	1.179
14	2	GE3	0.6	88%	0.007	0.199
15	2	GE3	1.3	88%	0.017	0.623
16	2	GE3	1.9	88%	0.024	0.910
17	2	GE3	2.5	–	0.024	0.994
18	2	GE4	0.6	86%	0.005	0.187
19	2	GE4	1.3	91%	0.021	0.912
20	2	GE4	1.9	94%	0.019	1.013
21	2	GE4	2.5	–	0.039	1.783
22	0	GE5	1.9	–	0.043	1.791
23	2	GE5	0.6	92%	0.006	0.311
24	2	GE5	1.3	89%	0.020	0.890
25	2	GE5	1.9	81%	0.030	1.492
26	2	GE5	2.5	–	0.042	1.989
27	4	GE5	1.9	–	0.025	1.301
28	6	GE5	1.9	–	0.019	1.141
29	0	GE6	1.3	–	0.011	0.453
30	2	GE6	0.6	88%	0.004	0.148
31*	2	GE6	1.3	84%	0.015	0.515
32	2	GE6	1.9	70%	0.019	0.674
33	2	GE6	2.5	–	0.023	1.007
34	4	GE6	1.3	–	0.014	0.480
35	6	GE6	1.3	–	0.009	0.361
36	2	GE7	0.6	70%	0.006	0.160
37	2	GE7	1.3	70%	0.008	0.255
38	2	GE7	1.9	70%	0.014	0.537
39	2	GE7	2.5	–	0.017	0.665
40	2	GE8	0.3	87%	0.004	0.120
41	2	GE8	0.6	92%	0.011	0.398
42	2	GE8	1.3	98%	0.021	0.891
43	2	GE8	1.9	–	0.023	1.014
44	2	GE8	2.5	–	0.025	1.154

*** The current polymer program at the current dosage.**

IV. Case study G data showing the flocculation analyzer and Britt jar fines retention responses for eight flocculants and alum. The correlation between height and retention is 84% for the anionic polymers and 65% for all results combined.

dosages of the current retention polymer, GE6. The peak height is 0.015 N at the current dosage of 1.3 lbs/ton. **Figure 7** shows the results with polymer GE1, a low charge anionic polyacrylamide, in which there is a consistent trend between dosage and peak height; however the response is not as strong as the current product, GE6. **Figure 8** shows the results for polymer GE5, a medium charge anionic polyacrylamide, which gave the best response of the products tested. Again, there is a consistent trend between peak height and flocculant dosage. **Figure 9** shows the results for five dosages of a low-charge cationic polyacrylamide. Note the different shape of the curves compared to the previous three graphs. Also, unlike with the anionic polymers, there appears to be no additional benefit to increasing the dosage beyond 1.25 lbs/ton.

The cationic demand of this system is very low, 18.5 μeq/L. So, we investigated the impact of the thin stock alum on flocculation and discovered an interesting result. Adding alum actually hurt performance in this system. Adding a modest dose thin stock alum has many benefits and may be appropriate for this system; however from a purely flocculation perspective, it degrades performance. **Figure 10,** for example, shows the results with four different alum dosages and 1.9 lbs/ton of polymer GE5.

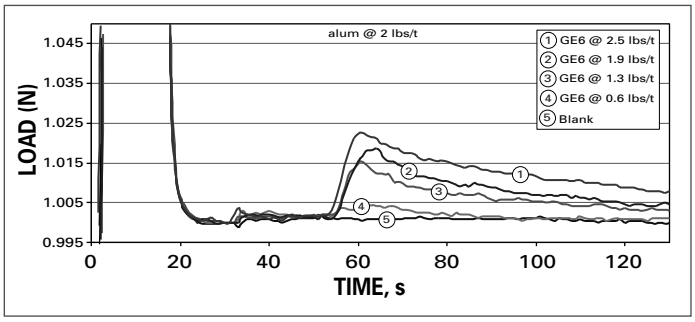
Dynamic drainage jar experiments were run in parallel with 25 of the 44 flocculation analyzer experiments, and the results are listed in Table IV. The correlation coefficient between fines retention and peak height was R = 84.4% for the six anionic polymers, but only R = 65.4% when all eight polymers are included. Similar results were observed for the fines retention correlation with peak area (R = 86% and 62.6% respectively). **Figure 11** shows the drainage jar results of the alum addition study for three polymers at three alum dosages. These results also show a decrease in performance as alum addition increases, which is consistent with the flocculation analyzer observations.

Additional five studies

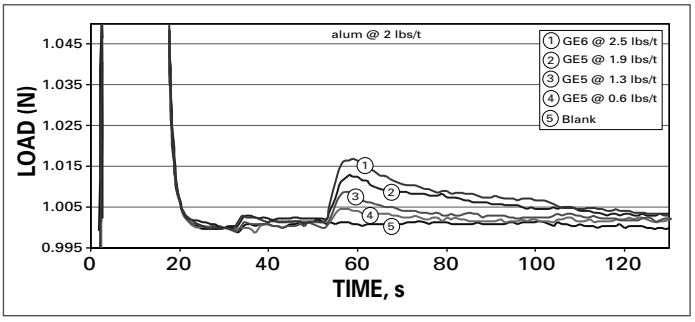
We also evaluated alternative chemistries to find a more cost effective drainage and retention program for a mid-Atlantic cylinder gypsum board machine, which was using a dispersion polymer (DD1). All of the polymer programs were cationic and all included a pretreatment with the current polyamine coagulant at the current dosage of 5 lbs/ton. We obtained moderate responses with an alternative dispersion consistent with the dosage, but we observed no significant flocculation. We also tried two high molecular weight cationic emulsion polymers, two cationic solution polymers with very low solids, and two powdered products, which have a much higher actives content than the other products tested.

From this study, we concluded that we could improve performance with emulsion, powder, and solution polymers and that molecular weight has a significant impact on polymer performance. MSR testing was performed concurrently, and similar polymer performance trends were observed, though not presented here. Subsequent trials on the paper machine confirmed the results and led to a 65% savings for the mill.

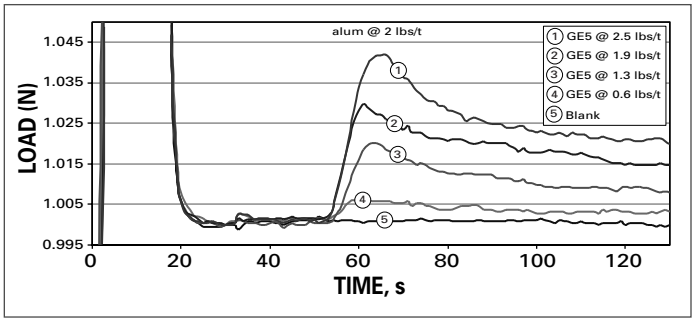
In a study of a Midwestern medium machine, we evaluated the wet end system and proposed a chemical program to increase speed through improved retention and drainage. We ran Britt jar experiments in conjunction with the floccula-



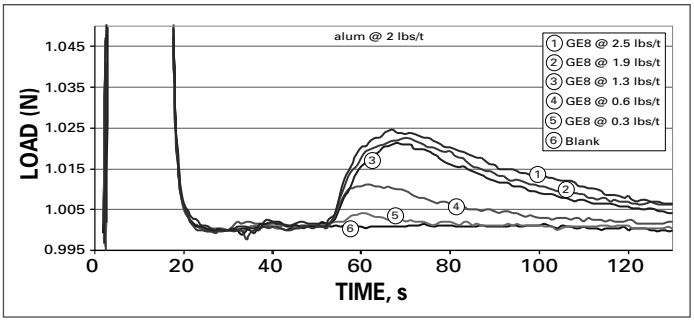
6. Case study G – Current polymer treatment at four dosages.



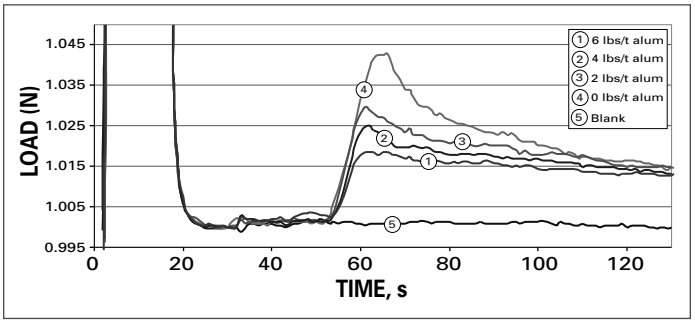
7. Case study G – Polymer GE1, a low-charge anionic polyacrylamide, at four dosages. The graph illustrates the consistent trend between dosage and peak height; however the response is not as strong as the current product, GE6.



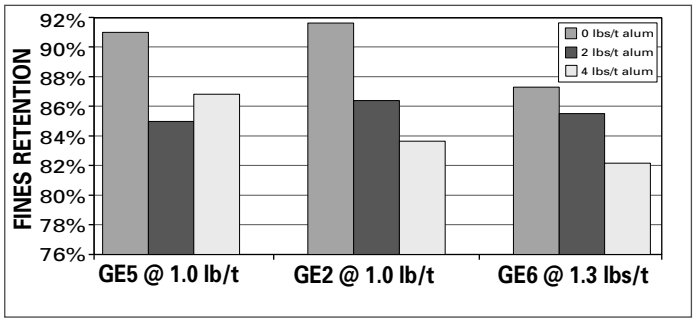
8. Case study G – Polymer GE5, a medium charge anionic polyacrylamide, at four dosages. The graph illustrates the consistent trend between dosage and peak height. This polymer gave the best response of the products tested.



9. Case study G – Polymer GE8, a low-charge cationic polyacrylamide, at five dosages. Note the different shape of the curves compared to the previous three graphs. Also, there appears to be no additional benefit to increasing the dosage beyond 1.25 lbs/ton.



10. Case study G – Polymer GE5 at 1.9 lbs/ton with four different alum dosages. Adding alum actually hurt performance in this system.



11. Case study G – The drainage jar results of the alum addition study for three polymers at three alum dosages.

tion tests. More details of those results, and results from studies of a Northeastern 100% recycled bag machine and a Midwestern printing paper machine, appear in our conference paper [7].

CONCLUSIONS

We have demonstrated that this flocculation analysis technique can be used to screen polymeric treatments of paper mill furnishes and also to optimize chemical programs. The device's applicability is based on the wide range of samples that it can analyze as well as its sensitivity to small changes in polymer chemistry and dosage. This work further supports and extends the conclusions drawn by Laleg and Collins [*1-2*]. We have shown that the torque-based flocculation analysis technique described above can measure the degree of flocculation and the formed floc's resistance to shear. The data generated by the analyzer are dependent on (1) the type of chemical applied (e.g., molecular weight, degree of branching and charge density), (2) its dosage, and (3) the sample being studied. For example, in case study E (reported in the conference paper), adding a coagulant increased the degree of flocculation, whereas in case study G, the addition of alum actually reduced the degree of flocculation. The technique can be used on a wide range of samples from a belt press sludge at 6% solids to a simulated headbox sample at 0.34% solids.

This device provides an objective, numeric value for the degree of flocculation and de-flocculation, which is complementary to a gang stirrer test. Traditional beaker tests provide subjective values for flocculation but also provide a means of

measuring actual turbidity and settling rate. Since the device reports a numeric value and it is sensitive to small changes in polymer dosage, it may be an excellent tool for online monitoring and control of any flocculation-based process. Another benefit of this technique is that it measures the state of flocculation continuously and allows the user to record the signal as a function of time, which is not easily obtained by other techniques. This feature has many practical benefits. For example, one could use the data to determine optimum addition points based on time and chemical reactivity. Another advantage to recording 120 data points in a 2 min experiment is a higher degree of confidence in the interpretation of an experiment. After running a traditional drainage jar experiment, the user has one or two data points (i.e., filtrate consistency and turbidity). With a 120-point curve, we have a trend that can be more easily analyzed and outlier points can be more easily identified. Finally, the data are available for the user immediately rather than waiting for a consistency pad to dry in an oven.

There are, however, some limitations to the technique that should be mentioned. The most significant is that it measures torque or viscosity of a treated sample and not retention or drainage. We have shown that a relationship exists; however, there may be situations in which a large floc or a shear-resistant floc is not the optimal solution. The speed of the Britt jar impeller can be adjusted to simulate the fines retention on a paper machine, whereas this flocculation analyzer can not. Finally, these devices are significantly more expensive than the traditional lab techniques mentioned above.

Since all of the work reported here was collected on-site at various mill locations, we feel confident that this device is rugged enough for in-mill chemical screening and optimization experiments. The data generated by the instrument are repeatable, and we have shown that the data correlate well with traditional lab techniques such as free drainage tests and Britt jar data.

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INSIGHTS FROM THE AUTHOR

As water consumption decreases throughout the industry, polymer use and optimum polymer product selection are critically important to the success of many mill operations. As part of Ashland's continuing research commitment to our customers, we chose to investigate this novel flocculation analysis tool. Understanding and optimizing this technique has allowed us to successfully select polymers to solve difficult problems for our pulp and paper customers.

This work complements studies by Laleg and Collins [1]. They reported on the laboratory technique and I took it to the field. This paper describes results from in-mill polymer evaluations. The most difficult aspect of this effort was to develop a general procedure for all types of samples and then to interpret the data generated from this procedure to make it meaningful for mill operational personnel.

I was most surprised by the correlation between the results from the flocculation analyzer and Britt jar retention data. I expected the results to be related, but not with a high correlation coefficient.

In this work we used the peak height as a measure of the flocculation intensity and the area under the curve as a measure of floc strength or resistance to shear. One area of future investigation will be to further investigate the shape of the response curve or the topography of a three-dimensional (load, time, and chemical dosage) response surface. There may be valuable additional information and many other possible interpretations to be discovered from this line of inquiry.

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