

Root cause analysis of cationic polymer additive efficiency decline in virgin and recycle containerboard mills

DAVIT E. SHAROYAN AND CORY W. ROSS

ABSTRACT: It is well known that retention, drainage and strength polymers struggle to perform (if at all) in virgin containerboard mills. In-depth studies have been undertaken in this area for more than seven years, investigating the issue from all directions.

A key finding of this work is that soluble lignin is detrimental to chemical efficiency. A strong correlation exists between decreased chemical efficiency and high soluble lignin. Both recycled systems and virgin systems have been studied, and this correlation holds true regardless of furnish. The primary area of concern is virgin containerboard, because these mills tend to have the highest lignin levels. Some highly closed recycled mills can also build elevated lignin levels that can negatively affect chemical efficiency.

Application: The main objective of our studies was to discover the primary reason for polymeric efficiency decline in paper mill process waters and look for ways to overcome the problem. It was found that increased levels of soluble lignin is the main reason for polymeric efficiency loss. If lignin levels are high, there are ways to remove some of the soluble lignin and to improve on efficiency of polymeric additives and paper machine runnability.

The global containerboard market continues to grow as demand for paper-based packaging increases. Primarily, the demand is due to consumer preference for corrugated boxes, which are widely accepted as a more sustainable packaging alternative than plastic. Corrugated boxes provide excellent protection for transporting goods and can easily be recycled or composted.

North America has the highest concentration of virgin containerboard mills in the world and one of the best fiber sources. For years, North American mills have produced some of the strongest corrugated boxes available. With a large percentage of virgin boxes in the recycle stream, the North American market benefits from very high-quality OCC (old corrugated containers). Because of strong demand, virgin containerboard mills have been expanding production at existing mills for many years by maximizing pulp mill production and adding recycle plants. Consequently, many mills that are operating at capacity and looking for more opportunities to increase production are willing to consider chemical options. These mills historically have not used much wet end chemistry; thus, this is a big change for them. Unfortunately, using chemical additives—and making them work—is often challenging and can result in very high costs for somewhat marginal performance. For these reasons, chemical efficiency has become an important topic.

With sustainability at the forefront, many recycled containerboard producers manage their water consumption very closely. Water consumption has been decreasing as mills continue to close up their systems. This trend has created more

challenging conditions for the application of chemical additives. As more and more water is recycled, the contaminants cycle up. This reduces chemical efficiency.

With increased water closure, the amounts of dissolved, colloidal, and suspended impurities (e.g., dissolved salts and organics, volatile fatty acids, fines, etc.) in process water grow exponentially and cause many problems for paper production [1-7]. Conductivity is one of the parameters that dramatically increases with water reuse; it is believed to harm the efficiency of chemical additives (e.g., strength, retention, and drainage polymers; and sizing agents) [8-11]. It is a well-described phenomenon that in waters with higher conductivity, polymer configuration changes and performance drops [8].

Among others, Johnson and Harrington [9] reported the effects of mill water circuits' closure and conductivity increase on first pass retention and drainage. The effects of different salts and increasing conductivity on polymer performance were described as well [9]. Studies were conducted by adding a 50/50 mixture of sodium chloride and sodium sulfate to a 50/50 mixture of hardwood kraft pulp and softwood kraft pulp. The lab results indicated that retention and drainage characteristics were adversely affected at higher conductivities (studied up to 5000 $\mu\text{S}/\text{cm}$). Mill data also indicated that an increase in conductivity resulted in a reduction in first pass retention, an increase in cationic polyacrylamide (CPAM), and an increase in alkyl ketene dimer (AKD) usage.

Using TMP newsprint furnish, Pruszyński and Jakubowski [10] studied in detail the effects of conductivity and cationic demand (from polygalacturonic acid) on drainage proper-

ties. The negative effects of specific ions (i.e., sulfate, sulfite, chloride, sulfate, phosphate, borate, silicate, ethylenediaminetetraacetic acid [EDTA], and pentetic acid [DTPA]) were investigated. Bentonite combined with CPAM was found to be highly sensitive to the presence of silicates, phosphates, and anionic trash. Polyethylene oxide (PEO)-based programs showed low sensitivity to single ionic salts; they were affected more by increasing cationic demand.

The effects of conductivity on paperboard and machine performance were summarized in a detailed review by Xu et al. [11]. Their manuscript elucidated many aspects of the topic of interest: water closure and contaminant buildup; sources of conductivity; typical conductivity values in different segments of the papermaking market; the effects of conductivity on colloidal stability and polymeric structure; and the effects of specific salts and ions on performance of papermaking additives.

Nevertheless, conductivity is not the only parameter that can affect polymer efficiency. The other components of process waters that can harm chemical performance are negatively charged components of organic origin, such as dissolved lignin, degraded starch, volatile fatty acids or organic acids, or fatty acid triglycerides, and so forth, which are commonly referred to as “anionic trash”. They can also reduce polymer efficiency [12-16].

Among others, studies reported by Lee et al. [12] indicated that colloidal substances in process water were more critical and detrimental for sheet strength (e.g., tensile index and Scott

bond) than dissolved substances (including inorganic ions) [12]. Springer et al. [13] found organic components had a greater effect on reducing tensile strength than inorganic contaminants. The relative order of contaminants from the most to the least detrimental was defoamer, kraft lignin, water-soluble wood extractives, and lignosulfonates from the neutral sulfite semichemical (NSSC) pulping process [13].

Several authors demonstrated that water closure affects, to varying degrees, the efficiency of different polymers. For example, Allen et al. [3] investigated the effectiveness of five retention polymers (i.e., bentonite/anionic polyacrylamide, poly-DADMAC, polyethyleneimine, PEO/phenolic resin, and chitosan) in white water systems, representing three degrees of closure (with fresh water makeup at 55, 20, and 3 m³/ton of pulp). The system closure increased cationic demand and impaired the performance of cationic polymers, while the performance of non-ionic polymers remained relatively unaffected or even improved. Polverani et al. [4] observed similar trends with changes in fiber source when 100% OCC was converted to 50:50 mixture of OCC and NSCC pulp. Increase in cationic demand resulted in poorer performance of cationic polymers and more efficient performance of PEO/phenolic resins.

The relative contributions and the negative effects of the individual components in process waters have not been thoroughly investigated. Identifying the most troublesome component(s) and finding ways to remove them from the mill water may increase the efficiency of chemical additives.

Analyzed Components	Minimum Values, ppm	Maximum Values, ppm	Average Values, ppm
Sodium	126	2540	740
Calcium (as CaCO ₃)	166	4790	1510
Magnesium (as CaCO ₃)	23	243	100
Hardness (as CaCO ₃)	189	4940	1610
Aluminum	<0.5	14.5	3
Chloride	177	2360	690
Sulfate	83	2240	830
Acetate	120	3100	660
Propionate	<25	860	210
Butyrate	<25	750	240
Total inorganic carbon	41	267	113
Total organic carbon	1032	15570	4165
COD	2720	70800	16900
Starch	3	5753	~2400
Lignin	109	895	390
Mutek charge, µequ/L	-97	-1645	-520
pH	5.5	7.4	6.1 - 6.4
Conductivity	1270	13800	5400
CaCO ₃ = calcium carbonate ; COD = chemical oxygen demand.			

I. Average values and the range for major contaminants from analyses of white water samples from 17 mills.

INVESTIGATING CHEMICAL EFFICIENCY: EXPERIMENTAL METHODS AND RESULTS

To identify the troublesome components and evaluate their effects on polymer efficiency, extensive mill water analyses were undertaken and a synthetic furnish/water model was developed. The composition of the model was based on white water analyses from 17 paper mills in North America, Asia Pacific, and Europe. Derived model white water included several inorganic components, such as sodium, calcium, chloride, sulfate, and acetate salts; and organic components such as fines, starch, lignin, fatty acids, and others; for a combined total of 12 components. The quantities of the majority of components in white water were derived from averaging 17 mill water analyses results. Averaged values, as well as the range from min to max values of selected components from water analyses, are summarized in the **Table I**. After the model was developed, extensive drainage studies were undertaken to identify the effects and contributions of organic and inorganic components on drainage times and drainage polymer aid efficiencies. CPAM was used as a polymer aid for drainage studies. Drainage testing was conducted using DDA (Dynamic Drainage Analyzer, PulpEye; Domsjö, Sweden) by making 500 ml 0.7% consistency stocks with synthetic white water of the desired recipe.

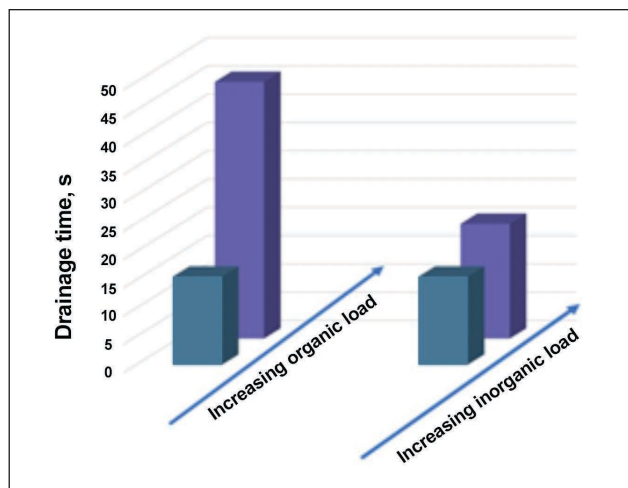
In addition to preparing model water that contained average levels of organic and inorganic components, we studied synthetic white waters with increased and reduced levels of organic and inorganic components. Manipulation of organic and inorganic loads helped us better understand the relative contribution of organic and inorganic impurities to the decline in chemical efficiency.

As Eq. 1 shows, drainage aid efficiency or percent improvement was calculated based on the difference (ΔT) between drainage times with (T_2) and without drainage polymer (T_1) over the drainage time without drainage polymer (T_1) and expressed as a percentage:

$$\% \text{ Improvement} = \frac{T_1 - T_2}{T_1} \times 100\% \quad (1)$$

We found that small ions such as sodium, calcium, chloride, sulfate, and acetate contributed as much as 95% or more of total conductivity; whereas the contribution of charged organic components to conductivity was minimal. **Figure 1** shows the relative effect of organic and inorganic component levels on drainage. We also found that a four-fold increase in inorganic components (and hence an increase in conductivity from 3000 to 9500 $\mu\text{S}/\text{cm}$) did not result in a significant increase (from 15.58 to 20.25 s or 30%) in drainage times. On the contrary, a four-fold increase in organic load did not result in any significant change in conductivity (from 3000 to 3200 $\mu\text{S}/\text{cm}$); however, this led to a significant increase in drainage times (from 15.58 to 45.36 s, an increase of almost 200%).

Drainage test results indicated that drainage polymer aid is more efficient in systems with low organic content. A four-



1. Relative effect of organic and inorganic loads on drainage.

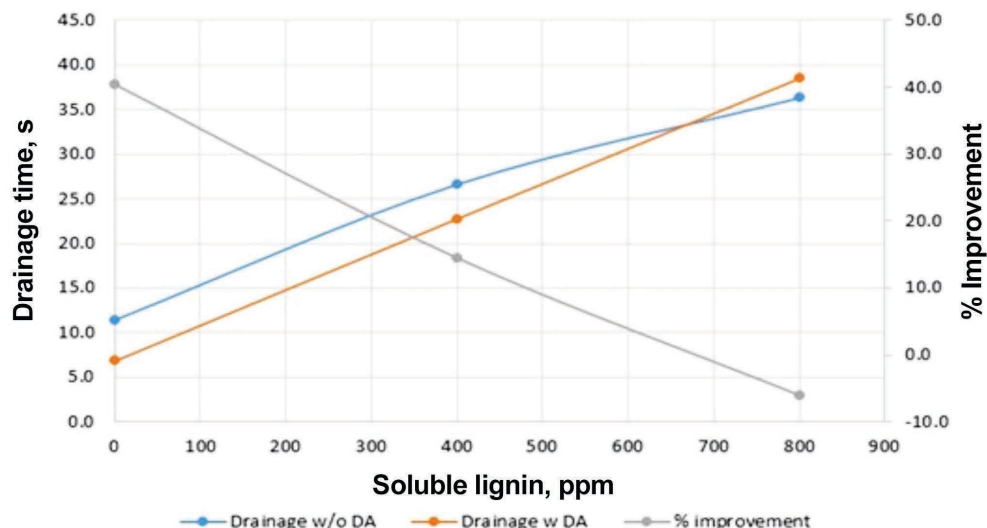
fold increase in inorganic load increased chemical efficiency from 30.6% to 49.9%, whereas a four-fold increase in organic load decreased chemical efficiency from 30.6% to 19.2%. Experimental data consistently indicated that high organic load was a much bigger problem for drainage than high inorganic load and conductivity.

When paper strength parameters and the effects of dissolved and colloidal contaminants on strength polymer efficiency were assessed, similar results were obtained. Ring crush and Mullen burst values were measured in handsheets made in model waters with different levels of organic and inorganic contaminants. The results clearly indicated that strength polymer efficiency was higher in systems with low organic content in water and lower in systems with high organic load.

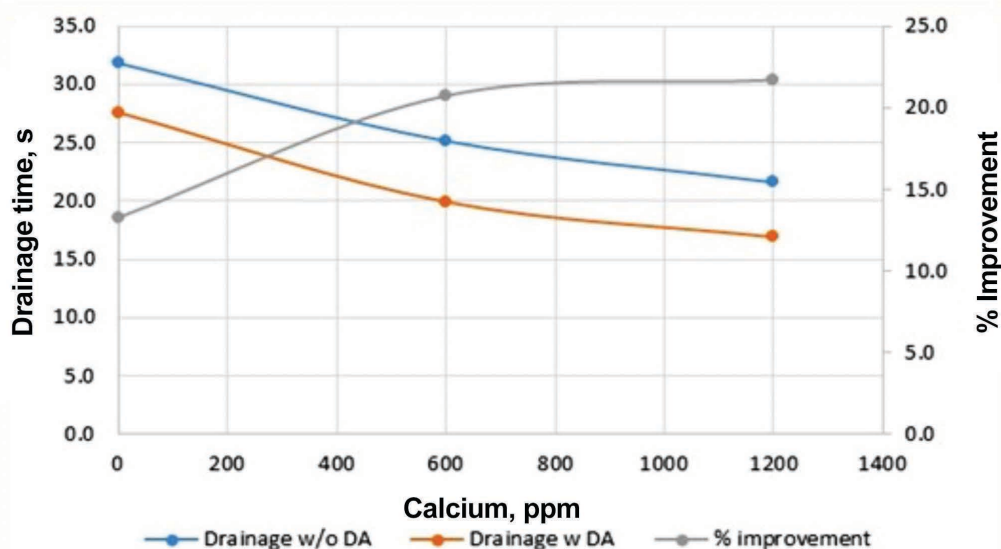
Next, we separately explored the effects of all individual components on drainage in the designed white water. Drainage times were measured with and without drainage aid and drainage aid efficiencies were calculated using Eq. 1. Throughout testing, the total organic and inorganic loads were kept constant so that overall conductivity ($\sim 5000 \mu\text{S}/\text{cm}$) would not be affected by variations in the concentration of the tested component. For each component, three conditions were tested: 0 ppm; X ppm (where X is the average load of the component in the designed white water recipe); and 2X ppm.

Out of 12 components studied, soluble lignin showed the strongest negative effect on drainage. **Figure 2** shows that drainage times increased both with and without drainage aid. As lignin levels increased, drainage aid efficiency steadily declined and practically disappeared when the load of soluble lignin reached 650 ppm or higher (polymer efficiency declined from 40% at 0 ppm lignin to almost 0% at 650 ppm lignin). Soluble lignin appears to be the most problematic component in the model white water.

For comparison, **Fig. 3** shows the effects of soluble calcium concentration on drainage times and drainage polymer efficiency. The effect of calcium ion (Ca^{2+}) was studied in the range from 0 to 1200 ppm (at constant conductivity). The de-



2. Effect of soluble lignin on drainage time, with and without drainage aid (DA).



3. Effect of calcium ion on drainage time, with and without drainage aid (DA).

cline in Ca^{2+} concentration was compensated for by the addition of other ions (sodium salts) to keep the overall conductivity constant. Test results indicated that drainage times with drainage polymer and without drainage polymer declined with an increase in calcium concentration. This can be explained by the complexation of soluble lignin with calcium ions and its partial removal from the aqueous phase [17]. As a result, polymer efficiency is not negatively affected. On the contrary, it is slightly supported by an increase in the calcium ion load in the white water.

Additionally, the effects of three parameters (i.e., soluble lignin, anionic starch, and conductivity) on drainage were investigated in more detailed design of experiment (DOE) studies performed with Design-Expert software (Stat-Ease

Inc.; Minneapolis, MN, USA) using regular 2-level factorial with 3 factors and 4 repeats of mid-points. In the DOE studies, the levels of all three factors varied independently and within a wide range. Soluble lignin varied from 70 to 730 ppm (400 ppm midpoint); anionic starch varied from 430 to 4370 ppm (2400 ppm midpoint); and ionic load varied from 820 to 8200 ppm (consequently, conductivity varied from 1300 to 8800 $\mu\text{S}/\text{cm}$). In these studies, drainage times with and without drainage aid were measured, as well as percent improvement (i.e., polymer efficiency), which was calculated using Eq. 1. Cationic polyacrylamide was used as a drainage aid at 2 lb/ton.

Table II summarizes the experimental results from the DOE studies. Raw data demonstrate that the highest polymer

Run	Starch, ppm	Lignin, ppm	Ionic load, ppm	Conductivity, uS/cm	Drainage Time without DA, s	Drainage Time with DA, s	ΔT , s	% Improvement
1	2400	400	4510	4800	24.61	20.86	3.75	15.24
2	430	730	820	1400	25.39	21.32	4.07	16.03
3	4370	70	8200	8720	13.67	8.75	4.92	35.99
4	430	730	8200	8800	16.67	17.00	-0.33	-1.98
5	2400	400	4510	5300	25.38	21.41	3.97	15.64
6	430	70	8200	8800	9.73	6.30	3.43	35.25
7	430	70	820	1270	11.30	6.21	5.09	45.04
8	2400	400	4510	4880	25.55	20.77	4.78	18.71
9	4370	730	820	1450	35.10	42.25	-7.15	-20.37
10	4370	730	8200	8740	49.27	46.21	3.06	6.21
11	4370	70	820	1420	18.66	10.12	8.54	45.77
12	2400	400	4510	4900	29.16	23.63	5.53	18.96

II. Drainage times and efficiency (percent improvement) at various loads of starch, lignin, and ionic load (conductivity). (DA = drainage aid.)

efficiencies (35% to 45%) are reached when lignin amounts are minimal.

Analysis of variance (ANOVA) performed with Design-Expert software (Stat-Ease Inc.) led to significant models for drainage and percent improvement (drainage aid efficiency) functions. Final equations in terms of actual factors were derived. The drainage model function (with model P -value 0.0049) identified both lignin and starch, and their interaction factors were significant factors. P -values for starch, lignin, and starch and lignin interaction factors were 0.0026, 0.0010, and 0.0125, respectively. P -values of 0.05 or less are considered statistically significant. For percent improvement, the function derived model (P -value 0.0054) identified only lignin, with a P -value of 0.0005, as a significant factor. Neither drainage or percent improvement functions depend on ionic load, and hence, conductivity. Conductivity was found to be an insignificant factor for both drainage (P -value 0.8592) and percent improvement (P -value 0.1428) model functions. These findings are similar to the results and observations from earlier tests. The regression equations obtained were:

$$\text{Drainage} = +10.196 + 1.016\text{E-}3^*[\text{S}] + 0.0133^*[\text{L}] + 5.962\text{E-}6^*[\text{S}]^*[\text{L}] \quad (2)$$

$$\% \text{ Improvement} = + 43.777 - 0.061^*[\text{L}], \quad (3)$$

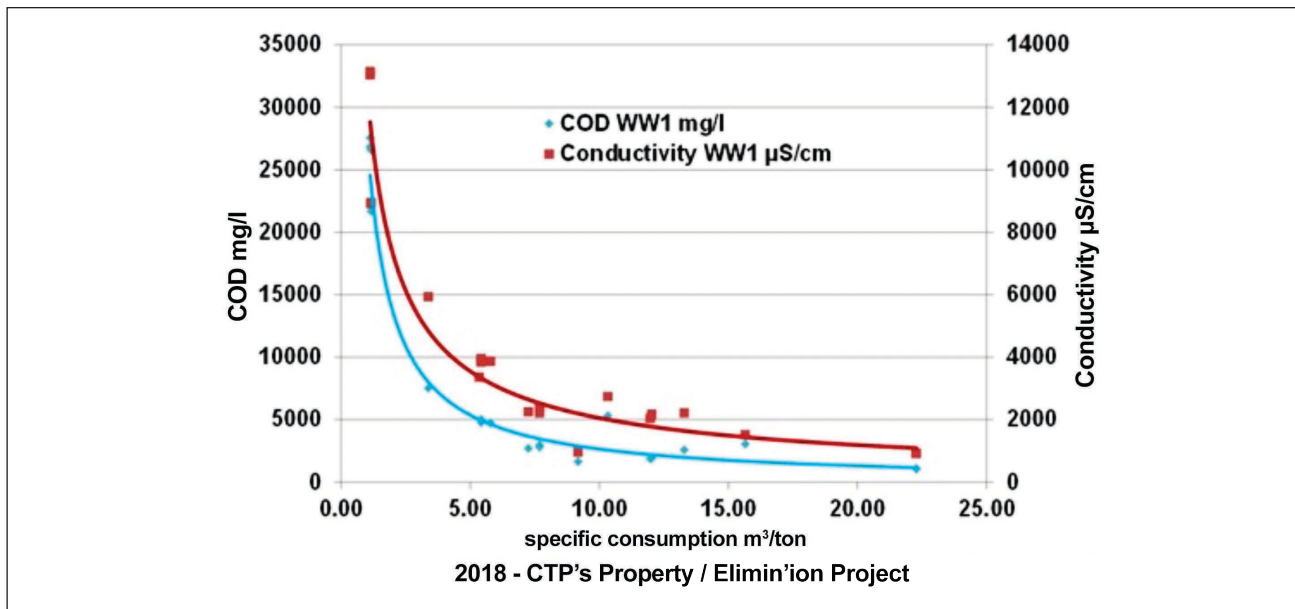
where [S] is starch and [L] is lignin.

Using Design-Expert software (Stat-Ease Inc.) tools and derived equations, drainage and percent improvement functions

were optimized to find conditions where the drainage times were minimal and percent improvement (drainage aid efficiency) values were maximal. The optimization process indicated that a reduction in soluble lignin (from, e.g., 400 to 200 ppm) could lead to an increase in polymer efficiency of approximately 30% (percent improvement). Therefore, even with only moderate reductions in lignin levels (i.e., partial removal of lignin from process water), significant improvements in drainage polymer aid efficiency can be expected.

The DOE analyses led to different conclusions than the previously reported studies. For example, prior statistical studies were conducted in a similar fashion and they also used the averaged levels of contaminants [6]. However, values of soluble lignin (from 25 to 175 ppm) used in those studies and the values of other components (e.g., sodium up to 5000 ppm) did not fully reflect the lignin and other contaminants' trends that we observed in recycled and virgin containerboard mills. Consequently, the prior studies overlooked the effects of lignin.

Some researchers [8-10] explored the effects of soluble salts on polymer performance with evaluations that used pure or tap water. While the information on conductivity or specific ions' effect on polymeric structure and performance is very important, other factors were beyond the scope of those investigations. Many studies that demonstrate the negative effects of conductivity on performance of papermaking additives (e.g., drainage, retention, strength aids, and sizing agents) fail to consider the increasing levels of organic contaminants that simultaneously occur with increases in conductivity. Conductivity measurement is quick and easy; however, analyses of organic constituents (e.g., starch, lignin, fatty acids, etc.) in process waters require equipment that is



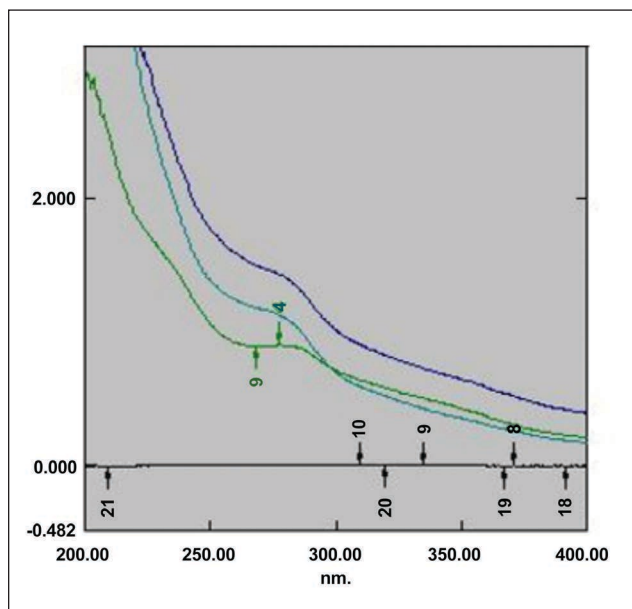
4. Conductivity and chemical oxygen demand (COD) in process water for old corrugated containers (OCC) mills (with permission from CTP) [18].

more sophisticated. Consequently, these analyses typically are not or cannot be conducted in paper mills; thus, all the attention and “blame” falls on conductivity. Nevertheless, limited studies of organics (e.g., chemical oxygen demand [COD]) along with conductivity indicate that organic constituents accumulate to the same extent with water closure and should not be overlooked (**Fig. 4**) [5,18].

Moreover, water analyses of virgin linerboard mills indicate that conductivity values on average are lower than those of highly or fully closed recycled mills. However, the efficiency of polymeric additives in virgin mills is very low or nonexistent; overall, it is significantly lower than in recycled mills. Therefore, conductivity cannot explain the lack of polymer efficiency in virgin linerboard paper mills.

The identification of soluble lignin as the most problematic component in paper mill waters helps to explain polymeric efficiency loss in specific market segments. The water systems of tissue and towel mills contain very low levels of soluble lignin. As a result, efficiency of polymeric additives is not affected at all. Lignin levels are significantly higher in recycle and virgin mill waters. Soluble lignin content varies from 100 to 600 ppm in recycle mills and from 400 to 600 ppm in highly and fully closed mills. Increased levels of soluble lignin decrease performance of polymeric additives. Efficiency loss can be moderate to high. Lignin levels can be significantly higher, reaching 2000 ppm or even higher, in virgin linerboard mills. When lignin levels are greater than 700 ppm, polymer efficiency declines to practically zero, making polymer aid applications essentially ineffective.

To solve the problems associated with soluble lignin, lignin must be analyzed and quantified. Several methods can be used to measure lignin in the lab setting and in the field. The most convenient and conventional method is based on ultraviolet-



5. Ultraviolet-visible spectroscopy (UV-VIS absorbance) of model white water (green line) and two white water samples from highly/fully closed recovered linerboard (RLB) mills (blue and purple lines).

visible spectroscopy (UV-VIS absorbance) measurements of white water samples at 280 nm. In that region (260 to 300 nm), lignin absorbance shows up as a local maximum on the spectral line (**Fig. 5**). **Table III** summarizes the average values and ranges of soluble lignin obtained by analyses of white water filtrates from several tissue, recycle containerboard, and virgin containerboard mills. Indulin AT kraft lignin was used in those measurements as a reference.

To overcome the effects of high lignin in process waters,

Tissue and Towel Mills		Recycle Containerboard Mills		Virgin Containerboard Mills	
Range, ppm	Average, ppm	Range, ppm	Average, ppm	Range, ppm	Average, ppm
0-55	30	110-605	390	140-2135	980

III. Lignin survey results from recycle and virgin containerboard mills.

specific additives must be used. We recently developed a new technology that reduces soluble lignin as a pretreatment step and enables polymeric additives to work well in water systems where they previously failed. This will be the subject of our next publication.

CONCLUSIONS

High organic loading of white water has a dramatic effect on retention, drainage, and chemical efficiency in general. The effect from organics is greater than from inorganics. Of the organic contaminants, lignin was identified as the major contributor to poor chemical efficiency. Lignin can be measured and monitored easily, and new chemical approaches can help to reduce soluble lignin and better manage wet end chemistry to achieve a variety of performance requirements. **TJ**

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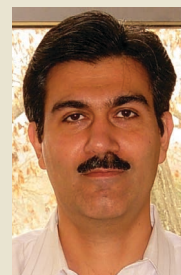
ABOUT THE AUTHORS

Water closure and polymeric efficiency decline remain a big challenge for the paper industry. The main objective of this study was to discover the primary reason for polymeric efficiency decline and look for ways to overcome the problem. Current research adds to a wealth of knowledge accumulated on polymer efficiency decline in process water. However, with a different and more comprehensive testing approach, different results were obtained here.

The most difficult part of this study was to overcome the established perception that increased conductivity is the main reason for polymeric efficiency decline. The most interesting and surprising finding was that increased levels of soluble lignin, and not conductivity, are the main reason for polymeric efficiency decline.

Paper mills can benefit from monitoring levels of soluble lignin in their process waters. If lignin levels are high, there are ways to remove some of soluble lignin and to improve the efficiency of polymer additives and machine runnability. Our next step would be to introduce efficient chemical approach(es) to remove soluble lignin from process water and to boost chemical efficiency.

Sharoyan is principal scientist, Global Technology, and Ross is North American regional marketing lead and strategic market manager, Packaging, for Solenis LLC in Wilmington, DE, USA. Email Sharoyan at desharoyan@solenis.com.



Sharoyan



Ross

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