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Useful and Effective Diagnosis and Evaluation Tools for Eenvironmental Change in Increased Mill Water System Closure

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ABSTRACT

In the past, abundant and clean water was available for paper mills' use. However, the growth of population and industry made water less available nowadays. Also, environmental regulation limits wastewater discharge, which affects mill operation cost. Therefore, paper mills are under pressure to use more recycled water and mill system closure. As a result, chemical and physical parameters of water are changing and new environment is being created for microorganisms in paper mill system as well. The more soluble or suspended organic materials are increased as more water is recycled and less or scarce dissolved oxygen is available, depending on the degree of recycled water usage. Microorganism flora in paper mill system will be also shifted according to the environmental change of mill system. Anaerobic bacteria, including sulfate reducing bacteria (SRB), will be dominant in the system as very low or almost no oxygen available in the system. Nevertheless, it is common in domestic paper mills that employ the same and old biocides as a means of microbial control, and microbiological control is often less recognized or even neglected. The right biocide selection for increased reductive environment of mills is critical for operation and estimated loss from paper quality defects such as sheet break, holes due to microbiological cause is tremendous compared to the microbiological control cost. It is imperative to investigate and diagnosis the environmental change of mills for right control of cumbersome microorganisms. Several useful diagnosis tools, including new technology employing OFM (Optical Fouling Monitor) *in situ*, are illustrated.

1. Introduction

In the past, abundant fresh water allowed papermakers considerable flexibility in dealing with problems on the papermachine.

Today, plentiful, inexpensive, fresh water can no longer be taken for granted. With the growth in world population and increased life style expectations, today's papermaker comes into competition with the community for the same water. This can lead to increased costs or even allotment limitations.

As an example, environmental regulations severely limiting discharge of the mill wastewater can critically affect water management.

Although water sent to the sewer can be treated to reduce or remove BOD/COD, some mills are no longer able to discharge the

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treated water into streams. Other mills have been allowed to expand the number of machines on the site, but have not been able to increase the total amount of water they are allowed to discharge.

The papermaker's response to these pressures has come to be known as *mill closure* or *water system closure*. Changes made as a result of water system closure can impact the microorganisms that colonize the papermachine.

The objective of this paper is to explore the effect of water system closure on the microbial population and useful monitoring tools.

2. Water reutilization

Water lost through evaporation or carried out with the sheet is not recoverable. Fresh water must be added to make up this water loss. However, certain papermaking practices can intensify water loss. In extremely open systems, it is not uncommon to see fresh water hoses run continuously, even though they are only needed for short periods of time.

The relatively simple act of turning off the hose can conserve water with minimal cost or even net benefit to the papermaker. Conservation is considered the first category in water reuse (Fig. 1) and it requires minimal capital expenditure.

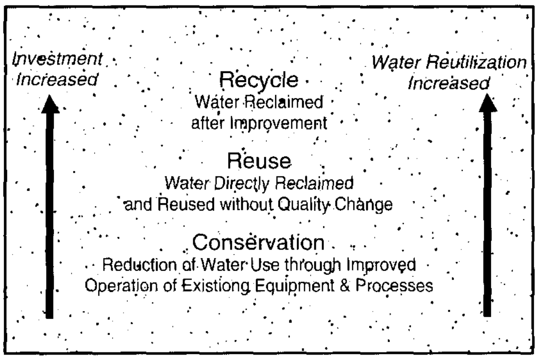

Fig. 1. Increasing stages of water reutilization.

The second stage of water reutilization involves reclaiming water from one source and using it in another process without making changes to the water quality. Fresh water is usually recommended for dilution of additives. As mills try to reduce consumption of fresh water, white water may become a source for dilution of additives such as starch or polymers. In 1993, Brouwer[1] looked at contamination of starch diluted with white water. He found that the amount of time the cooked starch could be held without preservation decreased dramatically when compared to starch diluted with fresh water.

He stated that higher levels of biocide were needed to control the growth of bacteria in the starch made from recycled white water rather than fresh water. The long starch molecules, added for sheet strength, are enzymatically degraded by microbial amylase. Brouwer did not address what happens if the amylase is already present in the white water. Biocide treatment successfully kills the bacteria but does not always denature the enzyme already formed by the microbes.

Fig. 1 indicates that the third stage of water reutilization involves reclaiming the water and improving the quality before using it in other operations. In general, as you move from conservation to recycle, the costs increase. In extremely closed systems, steps toward a high degree of water recycling may include returning water after biological treatment for use in the papermaking process. Unfortunately, some of the bacteria used to stabilize the waste reach the papermaking process where they can continually reinoculate the machine water system. Furthermore, reverse osmosis equipment used to remove undesirable chemical ions can quickly become fouled by unchecked microbial growth.[2]

3. Mill closure

Due to the difficulties caused by this mill-to-mill variation in water sources, process chemicals, microbial species, biocide programs, etc., various conditions should be considered.

The literature contains warnings about problems caused by the growth of anaerobic bacteria in closed mill systems.[3-10] The growth of this population is enhanced by some of the factors that can make closure attractive. For example, water temperature often increases, which is usually a benefit. However, increased water temperature is accompanied by decreased dissolved oxygen content in the water.

Lower concentration of dissolved oxygen allows for more favorable growth of anaerobic bacteria.

The problems associated with growth of anaerobic bacteria in paper systems include odors from organic acids produced as metabolic products, depression of system pH caused by formation of these acids[3,8,10] and even the formation of toxic or explosive gases such as hydrogen and hydrogen sulfide.[7] In addition, as mill closure increases, soluble chemicals increase in concentration in the mill water. These chemicals can serve as nutrients for bacterial growth.

Since the growth of anaerobic bacteria as one of the major factors of problems caused by water reuse, different microbiological control is necessary due to local environmental change. Recovery of water after wastewater treatment is a possible method for improving the quality of water prior to reuse. This is being done at several mill sites. In some areas of California, mills are being asked to use municipal waste water as incoming water after biological stabilization and treatment of the municipal effluent in order to kill pathogenic microbes. The advantage of this process is that the majority of biodegradable compounds are removed and fewer sugars and other nutrients would be available to sustain microbial growth within the papermachine system. There are some disadvantages. Pathogenic microbes are usually much easier to kill than are the filamentous bacteria that cause numerous problems on the machine.

4. Microbial control chemicals

One category of specialty chemicals fitting this description is microbial control chemicals. Microbial colonization of paper

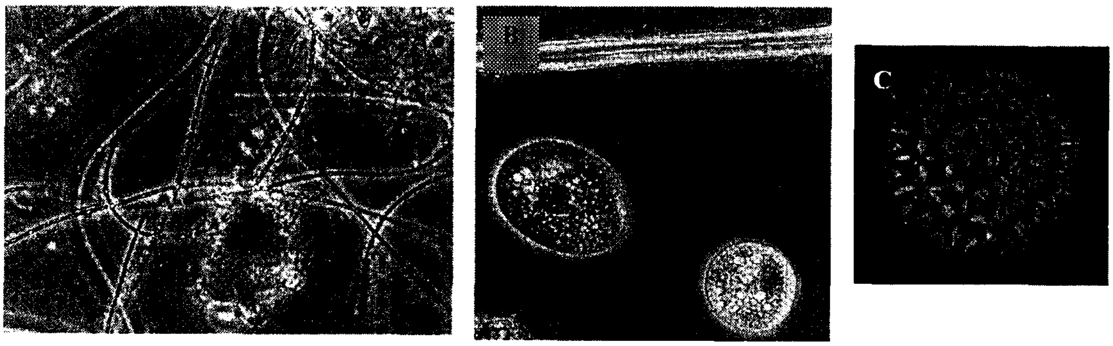

Fig. 2. 400X Phase contrast photomicrographs of microbes from paper machines. A is a deposit containing the filamentous sulfur bacterium, *Beggiatoa*, in addition to single-cell bacteria and filler materials. B compares motile protozoa with a wood fiber and C is an alga.

machines can increase machine downtime, reduce paper quality, and decrease profitability. In the past, deposit control issues were much less of a concern with small machines that ran for short periods of time between grade changes. Microbes were considered to be a minor nuisance that involved simple wash-ups during the frequently taken downtimes. If a small 20-ton a day machine is compared to a high-speed machine making 1,200-ton per day, the profit loss/hour of downtime might be \$232/hour compared to \$14,000/hour based on a \$700/ton paper price. The downtime that was endured on small, old machines, whose capital costs were paid off years ago, cannot be tolerated on the large high-speed machines. This downtime means the difference between a machine making a profit and one that looses money.

The large, high-speed machines are prone to microbial contamination due to increased surface area in the wet end. The high-speed increases the chances of a sheet break if minor pieces of biofilm/deposits are present in the headbox furnish. When coupled with the alkaline pH, the use of starch, high filler levels and the increased demand for lighter grades of paper, problems caused by microbial slime are increased. Most modern machines try to target a minimum of 6 weeks between boilouts. Some, with extensive microbial control programs, run for up to 6 months without a boilout on lightweight, coated machines running well over 1,000 meters per minute. This is in contrast to the small, old machines that take downtime every weekend.

Machine deposits are only part of the problems caused by microbes in the papermill. While microbial deposits are easily seen, it is the less visible microbial spoilage that causes additive and end product problems and malodors. Anaerobic bacteria can produce deadly, toxic and explosive gases that have injured and killed workers.[7] Alkaline papermaking and increased water system closure enhance the ability of anaerobic bacteria to thrive in the papermaking system. In addition, the large new chests typical of the high-speed machine systems have long holding times and often are poorly agitated. This can increase the possibility of spoilage, because the anaerobic bacteria are able to thrive under the stagnant, low oxygen conditions found in these large chests.

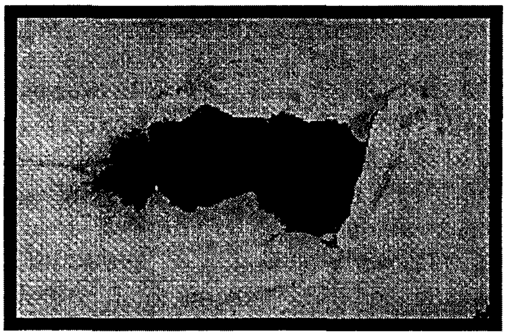

Fig. 3. Digital scan of a paper sheet hole formed by a sloughed deposit. Note the irregularities around the edges of the hole.

Traditional assessment methods for microbial problems provide limited information about the status of microbes in the machine system. Aerobic plate counts performed on agar media or Petrifilms® give limited information about the growth of microbes on surfaces. Typically, a 48-hour incubation period is needed to detect visible colonies on the growth medium. There is a lapse during at least 2 days of production before results are available. Starch spoilage is a fairly typical example of how microbes can cause quality problems. Microbial contamination in poorly preserved starch will cause a pH drop, viscosity loss and loss of sizing. The problem can be intermittent and can take a great deal of time to solve if only traditional plating techniques are used. During this period, the machine may produce a high percentage of unsellable paper.

Reducing down time and quality loss due to microbial activity can be accomplished with proper microbial control programs.

This paper will discuss new tools for monitoring microbes and biocides. It will also discuss new microbial control programs appropriate for world-class machines.

5. Biofilm deposits

In the paper machine ecosystem, microbes attach to surfaces to form a biofilm or deposit. After a monolayer of bacteria colonize a surface, the deposit dramatically increases in size as it entraps the wood fibers, carbonates, clays and other particles used in making paper. Bacteria and fungi bind these materials to form slime deposits (Fig. 2A). The deposits can grow in size to several centimeters in thickness. As long as the deposits remain in place on a surface they typically do not cause problems. However, when they slough they can form holes, defects or even breaks in the sheet.

When this happens it is often necessary to stop the machine to wash up or even stop production for several hours to do a thorough boilout to remove the deposits.

Microbes attached to a surface behave much differently than free-swimming organisms. Characklis and Marshall found that attachment to surfaces is a competitive advantage for microbes.[11] Attached microbes reproduce and are not washed away with the fluids. Fluid flow patterns actually increase nutrient availability. In addition, the variation in oxygen gradients within the biofilm permits growth of microorganisms with different oxygen requirements. Organisms in a biofilm are less susceptible to the effects of anti-microbial agents than are bacteria in the free-swimming state.[12] This is why it is important to prevent attachment through use of biocides to kill or inhibit the organisms or by using a non-toxic product to prevent the microbes from sticking to machine surfaces.[13]

6. Monitoring deposits

Judging the efficacy of a deposit control program can take days or even weeks. For most microbial control programs, the common assessment method is plate counts.[14]

These report the number of microorganisms or colony forming units (CFU)/mL of fluid using a standard microbiological medium. Unfortunately, measurement of microbes in the fluids is only an indirect indicator of the degree of deposit formation. At best, it indicates the potential for fouling or warns of changes in the overall population. Another weakness of these counts is the fact that non-toxic chemistries such as deposit control polymers or biodispersants may dramatically reduce surface biofouling without affecting the microbial plate counts.

Since plate counts do not effectively monitor machine fouling, a variety of methods have been used to directly monitor biofilm accumulation. The simplest fouling monitor is the machine itself. By examining the same area of the machine at regular intervals, it is possible to get an idea of the rate of fouling and predict the need for a boilout. This simple method can be effective, although it is highly subjective. The next level of refinement is to take pictures of the same spot on the machine over time or when the machine is down. The photos can record gross differences in program efficacy. They can help predict the need for a boilout even if they are not suitable for quantifying the deposition rate.

The first attempts utilized coupons to monitor the deposition rate. The most primi-

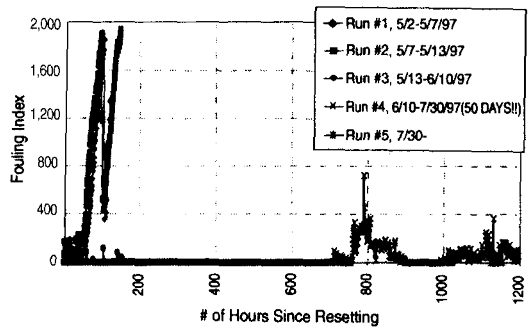

Fig. 4: OFM absorbance curves showing fouling over time. Early runs show steep fouling curves indicative of rapid fouling until program was changed.

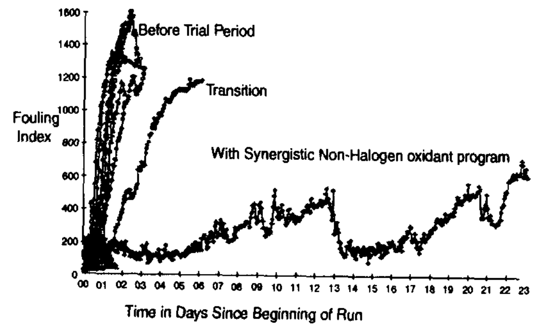

Fig. 5. Fouling Index at a specialty mill changing to a synergistic LAZON program.

tive method involved using a subjective numeric scale to judge slime accretion.[15] Wet weight determinations are subject to errors caused by sloughing of the biofilm on the coupon edges and the large error inherent in a wet weight compared to a dry weight. By using a template and dry weights it was possible to reduce some of the error; however, this method was cumbersome and required destructive off-line testing.[13]

Monitors such as the annular reactor has been very useful for giving on-line information in controlled university laboratory studies or systems containing low levels of solids.[16] Unfortunately, papermill fluids contain sufficient solids to interfere with the test method.

Wetegrove and Banks developed a useful optical fouling monitor (OFM) that provides on-line monitoring of paper machine surface fouling.[17] Fig. 4 shows the accumulation of deposits over several runs. Deposit accumulation was slow during the first days of the early runs in May of 1997. After the initial periods the fouling index climbs quickly. Sharp drops in absorbance signal/fouling index record sloughing events of the biofilm on the OFM deposit accumulation surface.

The final runs in June and July show low deposit accumulation when the deposit control program was changed.

Downtime was reduced as a result of a new LAZON^{sm} program.

In the example shown in Figure 5, the OFM was used to collect biofilm accumulation data prior to starting a trial with a new environmentally friendly program that used a non-halogenated oxidant combined with conventional biocides as part of a synergistic program. This specialty mill had tremendous problems with fouling caused by filamentous bacteria. Although bulk fluid plate counts were relatively low, fouling was severe. The rapid fouling is shown in the "Before Trial" curves in the graph. As the new program was being instituted, the rate of fouling declined. The final curve shows the biofilm accumulation for several days when the new program was fully operational.

In addition to monitoring biofilm accumulation, the on-line OFM is a critical monitoring tool for demonstrating the activity of non-toxic or less toxic programs. Non-toxic substances should not change the plate counts or ATP levels in bulk fluids.

However, they can be very effective in blocking microbial adhesion.[13] Without tools to accurately measure the effect of the chemicals, it is difficult to assess the difference between effective non-toxic chemistries and what might be wishful thinking.

7. Spoilage

Many additives used in making paper can serve as nutrients to support microbial growth. In addition to the cellulose fibers are additives that include: water, starch, alum, clay, carbonates, polymers, proteins, latex, and often complex coating formulations critical to print quality. Papermakers understand the economic benefits associated with deposit control; unfortunately, they tend to overlook spoilage of additives and fibers because it can be difficult to detect. Ignoring these processes can be costly, environmentally risky, and can present a direct safety hazard when toxic or explosive gases such as H_2 or H_2S are generated by the activity of anaerobic bacteria.[7]

Contamination of additives and coatings can come from a number of sources.[8] Not all makeup or quench water used for additive make up is properly treated. In addition, the material enters the mill already contaminated with bacteria and/or fungi. Or a contaminated carryover from a previously spoiled batch or shipment contaminates the next lot when the new additive is added to a chest containing the spoiled material.

8. Spoilage monitoring

The normal microbiological plating process allows enumeration of many spoilage microbes. However, problem solving is hampered by the 24~48+ hours it takes to form visible colonies in the growth medium. Furthermore, if the microbe has exacting growth requirements, it might take a longer period of time to form a visible colony, or the microbe may not be able to grow on the medium used for enumeration and the contamination missed. Microbiologists estimate that less than 0.1~1% of the total bacteria are recovered on agar. Plating methods do, however, provide information on the general type of microbes causing the problem. It is relatively simple to differentiate between a problem caused by fungi, aerobic bacteria, or anaerobic bacteria using specialized plating techniques.

The extended incubation period, necessary in plating techniques, slows resolution of the problem. For example, if a mill only makes sellable paper intermittently because of ongoing sizing problems, the time period needed for incubation of the plates or Petrifilms can result in the loss of significant quantities of paper.

One useful technique for measuring the metabolic activity of microbes is adenosine triphosphate (ATP) analysis using a photometer such as the TRA-CIDE® instrument.

Very simply, ATP can be thought of as the energy current of the cell. When ATP is extracted from cells it can be quickly evaluated by using a luciferin:luciferase reaction.

The light emitted by the reaction can be measured with an instrument called a photometer. In general, a high ATP reading means that numerous metabolically active cells are present, while a low value indicates that either few cells are present or that the cells are at a relatively low metabolic state.

ATP does not give a cell count, nor does it differentiate between the causative organ-

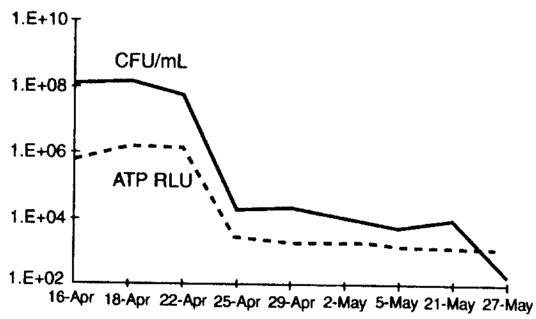

Fig. 6. Comparison of ATP relative light units and aerobic plate counts in CFU/mL on the effluent stock from a broke chest.

isms. This metabolic test does give test results in minutes, which allows corrective actions to be made immediately.

The plate counts and ATP levels (expressed as relative light units (RLU)) correlate fairly well in the example shown in Fig. 6. In contrast, the ATP levels and plate counts shown in Fig. 7 do not correlate very well. ATP analysis is not a direct replacement for plate counts, nor does it give a plate count number. However, by using the information correctly, ATP can be used as a metabolic indicator and early warning for problems. In the example shown in Fig. 7, thermophile bacterial plate counts in excess of 10,000 CFU/mL were a problem in this mill's size press starch. It appears that ATP levels, in this case, did not seem to be a problem if they remained below 800~900 relative light units (RLU). The two excursions, which were during the early problem solving period, had ATP RLU values of 1,460 and 12,350.*

As can be seen in the example shown in Fig. 7, interpretation of ATP results is not as simple as running the test nor as straightforward as the example shown in Fig. 6. ATP does not necessarily correlate with plate counts. Bacteria, inhibited by slow acting biocides, may yield low ATP values and yet be present in high numbers (See Fig. 7: Time 2). In addition to having only an indirect correlation with plate counts, low ATP levels can accompany severe problems caused by anaerobic bacteria. This is because anaerobic fermentation produces low levels of ATP in comparison to the high levels of ATP produced in aerobic respiration.

Some biocides appear to cause a temporary accumulation in ATP. One hypothesis assumes that the biocide makes the cell membranes leaky, allowing ATP to be released from non-viable cells. A second

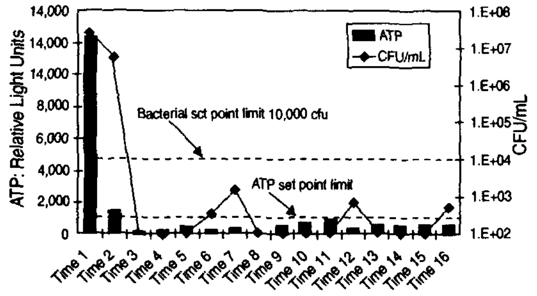

Fig. 7. Comparison of thermophilic plate counts and the metabolic indicator, ATP, in size press starch. Each warning "set point" is specific to the mill and sample site.

hypothesis suggests that the accumulation in ATP is caused, in part, by the mode of action of the biocide. This may temporarily allow ATP to accumulate prior to cell death. In addition, ATP released from lysed cells by the mode of action of certain biocides can persist for a short time in the machine process waters.

To avoid erroneous conclusions, an individual familiar with the microbiology of industrial paper systems must carefully evaluate the ATP data. Used correctly, ATP can be a valuable tool and can significantly reduce the time it takes to resolve a problem.

9. Monitoring biocides

TRA-CIDE ATP can be used to determine if a biocide has an effect against the population; however, it is not a direct indicator of the presence of a toxicant. Gas chromatographic and high performance liquid chromatographic analysis are accurate, but rarely available at a mill site. The toxicity (TOX) mode of the TRA-CIDE photometer can be used to monitor levels of antimicrobial substances in a paper system.[19] The

• Due to variations in test methods, reagents, bacterial populations, etc. the ATP levels shown here are not meant to represent a "safe" level or value for all mills. ATP levels must be calibrated to the sample point and the specific situation.

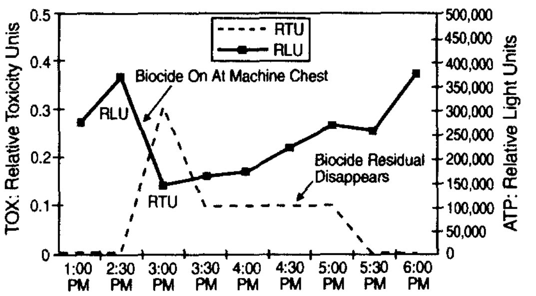

Fig. 8. Headbox relative toxicity and ATP readings conducted over a biocide feed cycle.

instrument is used to measure the initial light output of a bioluminescent bacterial suspension. An aliquot of water from the process is mixed with the bacterial cells and allowed to incubate for a set time period. A second light reading is then taken. Any decrease in light output is recorded and the proprietary software converts the readings into relative toxicity units (RTU).

Fig. 8 shows the ATP RLU levels and TOX RTU in a headbox over a 5-hour cycle on a paper machine in Asia. In the two sampling points prior to the biocide feed at 1:00 PM and 2:00 PM, the ATP RLU levels are high and the RTU toxicity units are not detectable. Shortly after 2:00 PM, a biocide was dosed into the system at the machine

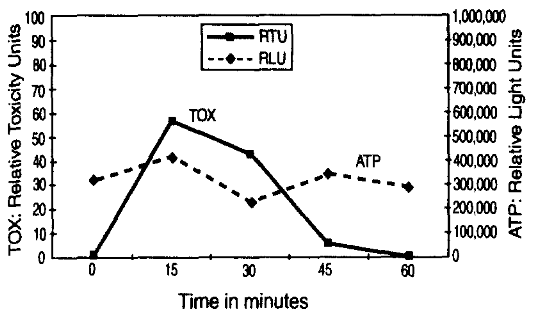

Fig. 9. Toxicity RTU indicates significant biocide present during the 1-hour cycle. Biocide was introduced just after the Time 0 sample. ATP RLU values do not change significantly during the same time period.

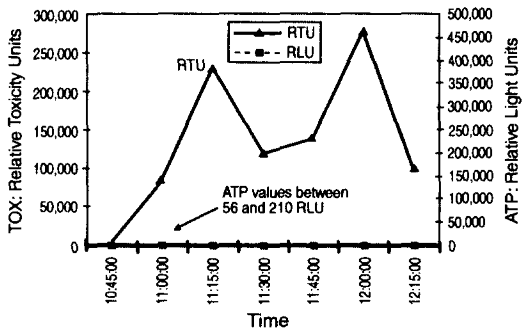

Fig. 10. Example of extreme over-feeding of biocides. TOX values are exceptionally high and the ATP levels are quite low.

chest. At the 3:00 PM sampling, the RTU and CFU/mL showed inversely proportional spikes in response to the biocide dosing. As the toxicant level rises, the ATP levels decrease. While the biocide levels decrease after the slug feed, the ATP levels gradually rise. By 5:30 PM, the biocide has completely disappeared and by 6:00 PM, ATP has returned to the pre-dosing levels.

In the cycle shown in Fig. 9, the TRACIDE TOX values show that significant levels of biocide are present following a slug feed to the machine chest. The ATP values do not change significantly during this time period. This indicates that the biocide is being added at levels insufficient to achieve a response against the microbial population present in the mill fluids. This type of curve can be observed when a biocide is underdosed or when the population has become tolerant of the biocide active.

The graph in Fig. 10 is from data collected in a central European board mill. Multiple oxidizing and non-oxidizing biocides were fed at numerous dosing points at the wet end of the machine. The biocides in this system were added at extremely high levels.

ATP values ranged from 56-210 RLU. This program was neither economical nor was it environmentally responsible.

10. Summary

Water system closure can be beneficial to the papermaker. In addition to reducing the amount of water, it can help with recovery of desirable chemicals and wood fines that may have been lost to the wastewater treatment system in the past. In order to take full advantage of water reuse potential, microbial problems must not be ignored during the planning stages of water system projects. In order to maximize the reuse of water one must keep in mind how the water will be reused and the microbial problems that can occur. Several conclusions follow:

1. The cycling up of various ions and possible nutrients may enhance the growth of different microbial populations. This may cause an increase in problems caused by anaerobic bacteria.
2. No single measurement will determine the degree of closure even when the fresh water is considered. A chemical with a tracer may be used if quantitatively determining the cycling up of the system is necessary.
3. Although papermakers are concerned about the microbes causing deposits, the problems of soured water, pH depressions, spoiled additives, corrosion, odor in finished products, hazardous gases, etc. may be of far greater consequence in closed mills. Papermakers and suppliers need to pay attention to the potential for these problems during the planning stages in the closure process.
4. Problems with filamentous bacteria may increase on the papermachine when water is recovered after biological stabilization. This has the potential of causing increases in deposits and plugging of showers and felts.
5. With increased water reuse, the demand for antimicrobial oxidants and certain proprietary biocides may increase. Biocides that work well in an open system may not be the product of choice under closed conditions. Product selection tests should be conducted periodically to determine if optimal products are used for the level of water reuse found at the mill site.

Uncontrolled microbial growth in the papermaking process adversely affects both paper product quality and machine runnability. Specialty chemicals are employed to prevent microbiological spoilage of additives, reduce the formation of hazardous gases, avert malodors and reduce formation of deposits on machine surfaces. The pursuit of improved control programs such as LAZON has prompted the development of targeted monitoring tools and novel treatment programs. These maximize machine performance while minimizing or eliminating adverse environmental impacts. The least expensive option may not always return the most profit to the papermaker.

Large, new high-speed machines are more susceptible to the microbiological problems than are small machines. Ignoring microbial contamination on the large machines can cause a greater loss of profitability. Downtime must be minimized.

Control of the adverse effects of microbial growth can be achieved through proper application and targeted dosing of biocides within the machine system. With the newly developed OFM, it is possible to determine the rate of fouling on machine surfaces and the effect of non-toxic chemicals on the deposit-forming microbial population. The OFM can be used to indicate the need for adjustments to the deposit control program and predict the need for boilouts.

The TRA-CIDE unit permits rapid assessment of microbial metabolism by monitoring ATP. In addition, TRA-CIDE diagnostics can also be used to monitor relative levels of biocides in mill fluids. By combining both tests, it is possible to quickly determine if sufficient biocide is present to inhibit microbial activity or to discover the presence of a resistant or tolerant population. This makes it possible to avoid biocide over-feeding or the use of ineffective biocides. Collection of data within minutes, rather than the days required using traditional methods, allows rapid program optimization. The combined use of monitoring tools and novel treatment chemistries can provide world-class control of paper machine microbes along with the best environmental stance.

Petrifilm is a trademark of 3M Corporation; TRA-CIDE and LAZON are trademarks of Nalco Chemical Company.

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