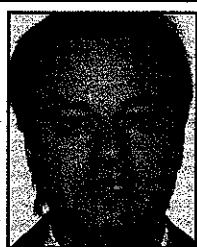


CATIONIC DEMAND SURVEY OF PAPER MACHINES: HOW CAN WE MEASURE AND USE IT?

A practical method is described

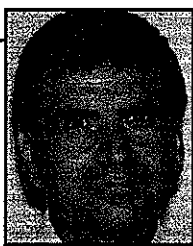
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- Zeta potential measurements;
- Colloidal titration ratio (CTR);
- Charge demand titration (CDT).

We will briefly describe each of these techniques and discuss some of the problems associated with their use.

ZETA POTENTIAL

The issue of charge measurement on paper machines has been addressed by many authors [1-5]. For many years zeta potential was measured with different electrokinetic techniques such as streaming current potential, micro-electrophoresis, electro-osmosis or sedimental potential. These tests were used by numerous mills and were the only tools available.

The technique most often used in paper mills is micro-electrophoresis where the movement of the charged solid (colloidal) phase is caused by an electrical field, with the liquid phase remaining stationary. The zeta potential, in millivolts, is then calculated, using standard equations from the mobility measurement.

Lindstrom [5] published a good overview of this technique and its theory for the papermaking system. Amongst other things, the technique can determine neither the amphoteric character of a surface nor the quantitative amount of soluble anionic charge in the system because it can only analyze the particles within the colloidal domain.

In our experience, this measurement can be fairly demanding for inexperienced personnel with the results being both operator- and technique-dependant. The dilution factor, the conductivity level (ionic strength), the technique for screening the colloids, the choice of a particle to monitor, the temperature of the sample, as well as the plane in which the operator focuses and normal eye fatigue — all may influence the results.

CTR TECHNIQUE

The CTR technique was first developed by H. Tewrayama in 1948 [3]. The technique basically treats the sample of the papermaking furnish with an excess of either a cationic or anionic standardized polymer, followed by its back-titration with a standardized polymer of opposite charge, to an endpoint indicated by the color change of o-toluidine blue.

Strazdins [1] and Patton [4] have pointed out some of the problems associated with the CTR technique such as premature color change induced by certain types of polymers, inability to measure the magnitude of charge (mobility), and the inability to properly record the rate of charge decay. Other problems associated with this method are the difficulty of determination of endpoint for colored stocks, the dependence of the color of the endpoint on the conductivity of the medium, the variation in adsorption tendencies of polymers, the fact that polymers adsorb on a surface regardless of their charge and resulting uncertainty of assumption of full stoichiometry of charge neutralization.

The CTR technique is fairly easy to perform and has the advantage of requiring little in terms of capital investment. Our experience indicates that when done in a consistent and rigorous manner it can give a good approximation of the charge associated with colloidal and soluble fraction and good trends for the wet-end conditions in different papermaking systems.

CHARGE DEMAND

The most recent development in wet end charge measurement is the particle charge detector (PCD) — which is based on the streaming current principle — and its use for the endpoint determina-

PAPERMAKING STOCK is a complex mixture of various components of different sizes, amounts and chemical nature. The proper retention of these components is a critical factor for the efficient operation of a paper machine.

The presence of soluble anionic material, often referred to as anionic trash, will interfere with cationic components such as retention aids and starches.

The amount of cationic material needed to neutralize part of the negative charges present in the papermaking stock must be strictly controlled in order to obtain a certain window of final charge, where the fibres, fillers and chemicals added in the wet end will be efficiently retained.

The configuration of the various feeding points is also an important factor and may affect the effectiveness of different retention programs.

Many instruments and techniques to measure the charge in the wet end are available to guide technical personnel in paper mills. The three offline techniques most commonly used to measure the wet end or system charge are:

tion in polyelectrolyte titration [7].

It was found that using a specific method of the charge demand technique, we could predict the effects of various chemical additives and get a better understanding of the charge interactions occurring in the wet end of a paper machine.

The effect of turbulence and kinetics in modern paper machines on the distribution of additives between charged and uncharged surfaces cannot be precisely simulated by any existing method. In this situation we believe that one of the best tools available today for the papermaker is the charge demand technique, assuming that adequate precautions are taken in doing the test.

The CDT principle is relatively simple. The charge of the suspension is measured qualitatively and quantitatively by titration of a known volume of a sample with a standardized solution of polymer of opposite charge to the isoelectric point detected with the streaming potential-based particle charge detector. The sample is placed in a cell and the colloidal particles adhere to the Teflon wall. The flow of the liquid phase, caused by the mechanical action, generates a streaming potential. Theoretical background is available in recent literature on this technique [4,7,8].

One of the most important aspects of this method is that the titration is done using diluted $1 \times 10^{-5}N$ or $1 \times 10^{-4}N$ solutions of the cationic polymer, poly diallyldimethyl ammonium chloride (DADMAC). This low-molecular-weight, linear, highly-charged cationic polymer, especially in highly diluted, low conductivity solutions, provides good stoichiometry and fast kinetics of the charge neutralization.

In the method discussed in this paper, a sample of stock is diluted with distilled water to the consistency allowing the particle charge detector to operate (if the

consistency is too high it hinders movement of the piston). Diluting the studied samples is not ideal as it changes conductivity, which has an important effect on polymer adsorption characteristics. Dilution may also affect the kinetics of the polymer interactions - diffusion of the macromolecule to the surface, its adsorption and reformation.

On the other hand, dilution, as opposed to removing fibres, also has positive aspects in that the lower conductivity increases the magnitude of the streaming potential signal and provides a sample containing all the elements of the papermaking furnish, better representing the real life situation.

In these studies, complete stock furnishes were always used. However if the cationic demand of only a certain stock fraction is of interest (e.g., fibres, fines, colloidal or dissolved fraction), the sample could be filtered appropriately before titration. Although we limit this presentation to the total cationic demand surveys of paper machines, the application of this method to measuring the cationic demand of particular fractions of the stock is informative and could be the wet ends of a separate presentation.

PCD METHOD

1. The instrument must be at room temperature and in good working condition. This should be periodically checked by doing a titration with the standardized solutions of cationic and anionic polymers. Our experience shows that it is extremely important to keep clean the Teflon titration container and the piston and electrical contacts between the container and the apparatus. It is recommended that a test tube brush and tap water be used to clean the cell, followed by rinsing with deionized water and dry-

ing with a clean (white) paper towel [6].
2. The use of a single concentration of titrant, within a series of measurements, is preferred. Changing the concentration of titrant will lead to the need for lengthy washing of the cell, especially when the change from higher to lower titrant concentration takes place. The minimum volume amount for the titrant should be 1 mL and the maximum 10 mL, in order to minimize the effect of dilution on the charge (the ideal volume is close to 5 mL).

Our experience shows that within these volumes of titrant the technique is reproducible within 5%. We found that using $1 \times 10^{-5}N$ or $1 \times 10^{-4}N$ solutions of DADMAC is preferable for the papermaking suspension.

3. Since high-consistency pulp must be diluted to prevent piston blockage, it is recommended that all the other samples be diluted by a similar factor, conductivity having an effect on the potential. Diluting by the same factor results in all tested samples having the same order of conductivity.

4. Our experience shows that dilution by a factor of five to 10 is generally sufficient in fine paper systems for good operation of the PCD because the consistency is low enough for unobstructed movement of the piston.

The dilution factor is commonly much higher in stocks having a very high cationic demand, such as deinked or groundwood fibres, and can be as high as 100 when a $1 \times 10^{-5}N$ DADMAC solution is used. Alternatively, the $1 \times 10^{-4}N$ DADMAC solution can be used and the dilution factor reduced proportionally.

Cationic demand values calculated for the original, undiluted samples measured using different concentrations of DADMAC solutions and different dilution factors, were compared routinely and showed good agreement.

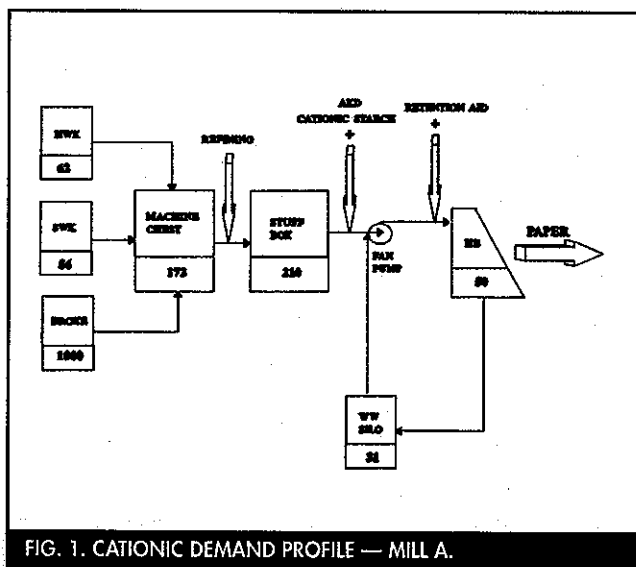


FIG. 1. CATIONIC DEMAND PROFILE — MILL A.

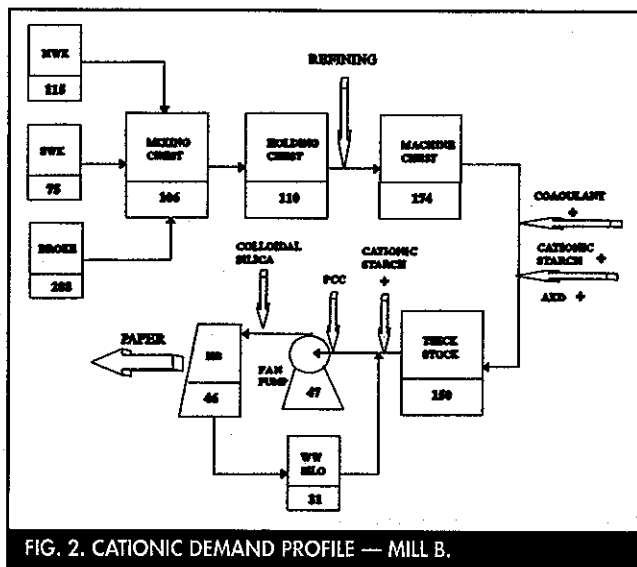


FIG. 2. CATIONIC DEMAND PROFILE — MILL B.

5. The anionic or cationic demand of the water used for dilution of the stock sample should be checked in order to correct all the demand values if necessary. This will also avoid possible contamination with a poor quality dilution water. The conductivity and pH of diluted samples should be recorded to assure that the same conditions are used throughout the study.

6. The PCD method normally works well under alkaline and neutral conditions within reasonable conductivity levels.

Under acid conditions, where large quantities of alum are used, the PCD should not be used without taking into consideration that the charge characteristics of alum vary depending on the pH, and that dilution will, by affecting pH, change the amount of charge provided by the aluminium species. Our experience with alum shows that the problems associated with its use are negligible for stocks with less than 2 kg/t of alum and/or operating at a pH above 5.5.

7. Using a scoop for thick stock samples and an Eppendorf-type pipette for diluted samples, a certain amount of furnish is weighed on a laboratory scale (0.01g). The Eppendorf pipette has a large opening to prevent blocking. The sample is then diluted by weight by the desired dilution factor using distilled or deionized water.

8. It is strongly recommended that all the diluted samples be titrated as soon as possible within a similar time, ideally two to three minutes. We do not recommend holding stock samples for more than half hour before testing them.

It was found that over time, some ionic species may move in or out of the fibres, changing the actual total charge of the sample. Koethe and Scott have shown the influence of time on polymer-fiber interactions [9]. In a recent paper, Wagberg et al. presented data on the stoichiometry and kinetics of polymer-fiber interactions [10].

9. Once a representative sample has

been acquired and properly diluted with distilled water, 10 mL of this suspension is pipetted into the white Teflon cell using an Eppendorf-type pipette. Always remember to place the Teflon ring over the piston so that no suspension splashes out during the titration.

10. Once the instrument is on, the operator should wait to see a stable potential reading before starting the titration with the polymer. The initial potential reading should be used only to determine which polymer solution should be used to titrate the sample (anionic or cationic).

The initial potential reading is often irreproducible as it depends on different external factors such as the conductivity, temperature and viscosity of the suspension, the size of the colloidal particles adhering to the Teflon wall of the cell and the dimensions of the sample cell.

11. Titrate the sample slowly and regularly at a speed near 1 mL/min preferably using a peristaltic pump or an automatic titrator. The operator can slowly reduce the titration speed near the isoelectric point. Ideally, the test should be completed within five minutes or less. If a volumetric syringe is used (which is weighed before and after the test) the operator must be careful to obtain a fairly constant flow to obtain a definitive end-point.

12. As soon as the titration is finished, the operator should clean the piston and the cell as described previously. The piston and the cell must be cleaned at the end of every day using a mild soap solution if necessary and rinsing thoroughly with plenty of water.

When the particle charge detector is used in different furnishes it is recommended to have a few spare pistons and cells so that when a cell is dirty it can be properly cleaned and rinsed over night. Unfixed dyes in a suspension can adhere to the cell wall and the piston and color them: we recommend using acetone to clean them thoroughly.

13. The following calculation is used to

find the cationic demand of the original, undiluted sample if $1 \times 10^{-5}N$ DADMAC solution is used:

$$CD(Eq/L) = \frac{V(mL) * 1 * 10^{-5}(Eq/L) * DF}{10(mL)} \quad (1)$$

where:

CD = cationic demand;

V = volume of titrant used (mL);

DF = dilution factor of original sample.

If the cationic demand of the studied sample is expressed in Eq/L then:

$$CD(\mu Eq/L) = V * DF \quad (2)$$

If the case where normality of the titrant solution is $1 \times 10^{-4}N$ and the sample volume is 10 mL, this equation will be modified to:

$$CD(\mu Eq/L) = 10 * V * DF \quad (3)$$

DISCUSSION

Various types of paper mills were surveyed for cationic demand. These surveys are presented to provide information about the typical pattern of cationic demand map in various types of mills. These patterns are characteristic of the type of production and are a function of the furnish and additives used as well as factors such as the degree of mill closure, white water recirculation, and so on. The cationic demand surveys should be a frequent exercise in the mill because they provide better understanding of the entire system and correlations amongst its various components.

The cationic demand of the headbox or white water should be monitored continuously, but frequent testing with the PCD titration device in the back of the system is also recommended. The value of these measurements will become even more evident when the correlation is established between measured cationic

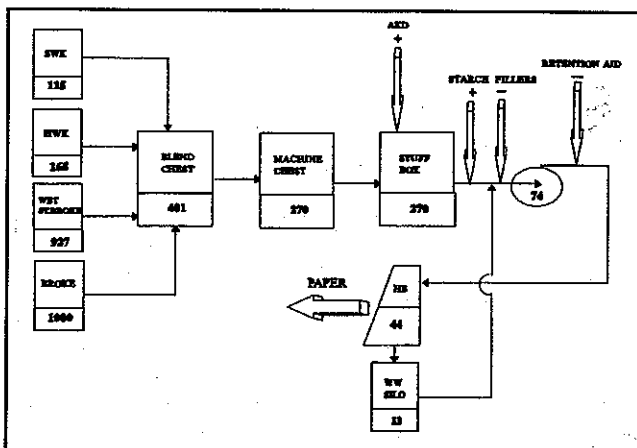


FIG. 3. CATIONIC DEMAND PROFILE — MILL C.

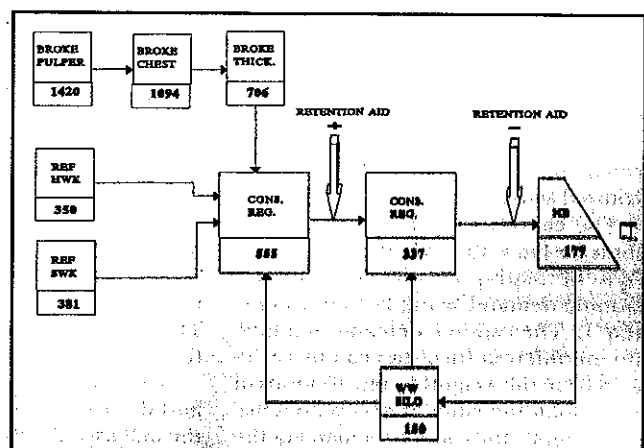


FIG. 4. CATIONIC DEMAND PROFILE — MILL D.

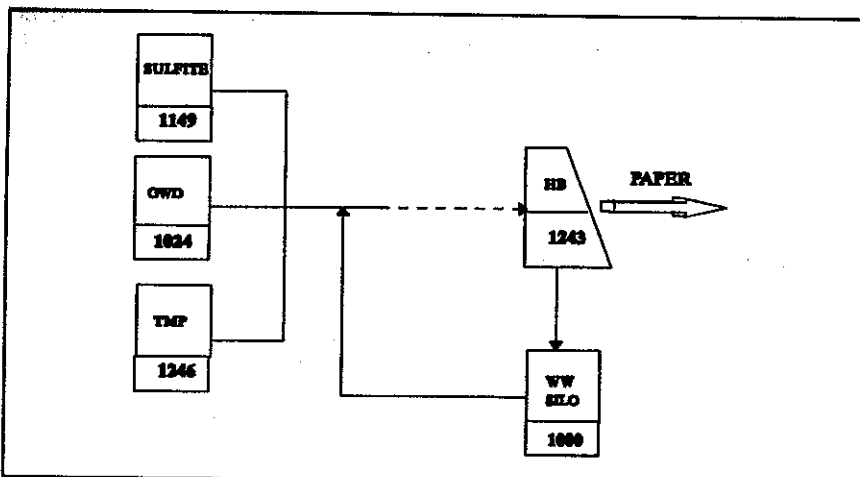


FIG. 5. CATIONIC DEMAND PROFILE — MILL E.

demand values and some relevant operation conditions.

A knowledge of the system characteristics under the typical operating conditions will pay off when upsets or process changes occur, for example the introduction of recycled content, white water closure, change of additives or change of the feed point configuration. The mill's technical personnel will be able to deduce the cause of an upset, correct it, or predict the effect of planned changes on the system.

Following are examples of the cationic demand patterns of several mills of different types. These data represent results of our numerous on-site surveys. These surveys resulted in typical cationic demand profiles which, once established for good operations, can be used in the case of upsets. Cationic demand profiles are blocked in four groups — fine paper, groundwood specialties, newsprint and board. These patterns are presented in the form of simplified diagrams showing stock flow, sampling point location and the feeding points of the most important additives. The cationic demand of samples is always expressed in Eq/L of total original stock sample with no correction being made for the consistency.

FINE PAPER MILLS

Mill A: This mill, Fig. 1, uses both hardwood kraft (HWK) and softwood kraft (SWK) as furnish as well as its own broke which contains optical brightener and oxidized starch.

The cationic demands of the kraft fibres are low with HWK of 62 μ Eq/L and SWK of 56 μ Eq/L, with the broke's cationic demand being as high as 1000 μ Eq/L. The cationic demand of a mixture of different furnishes can be calculated from the weighted contributions of the cationic demands of its components. For example, the cationic demand of the machine chest stock can be calculated in the following way, assuming that HWK,

SWK and broke are mixed in the ratio of 60:30:10, respectively:

$$CD_{mc} = 62 \cdot 0.60 + 56 \cdot 0.30 + 1000 \cdot 0.10 = 154 \mu\text{Eq/L} \quad (4)$$

This calculated value agrees well with the observed value of 172 μ Eq/L. This type of calculation must include white water dilution wherever it occurs.

The increase of the cationic demand between the machine chest and the stuffbox, can be attributed to refining of the stock.

Refining increases the concentration of soluble anionic material, content of fines fraction and the accessibility of the titrant polymer to the negative charges inside the structure of the fibres. After the dilution of the stuffbox contents with the white water and treatment with the AKD cationic starch and cationic retention aid the demand is lowered to 50 μ Eq/L in the headbox.

The removal of solids in the formed sheet lowers the cationic demand of white water to 31 μ Eq/L. The difference in cationic demand between the headbox and white water, especially if the white water were sampled directly from the forming section, might provide an interesting insight into the amount of charge removed with the sheet and the distribution of charge among fibres, fines and soluble material. This difference might also serve as a basis for setting the charge balance for a machine operating in steady-state. In practice, this is often difficult to discuss due to the significant and varying flow of fresh water into the system.

The cationic demand has been monitored on this machine for a long time. The numbers presented are characteristic of good operation of the machine.

When an upset affecting retention and drainage characteristics occurred, the mill used the cationic demand data it had collected to investigate and address the source of upset. During this upset,

the cationic demand of the headbox was significantly higher than typically measured. An anomaly was found in the cationic starch dilution system. The problem was corrected and this eliminated the retention and drainage difficulties. The cationic demand value of the headbox returned to normal.

Mill B: This mill, Fig. 2, uses a similar furnish to mill A. However, the cationic demand for kraft fibres is slightly higher and for broke it is much lower than the values measured in the mill A. As observed previously, refining increases the cationic demand and the addition of cationic starch lowers it. The numbers presented on the diagram are typical of the operations on this one-grade paper machine system.

The cationic demand was surveyed for a year and showed very stable operations. Note that if more than one grade is produced, then cationic demand baseline data have to be established for each grade separately.

Mill C: In addition to furnishes used in mills A and B, this mill, Fig. 3, also uses wet-strength broke. Wet-strength broke has a high cationic demand because of the chemical treatment necessary for its disintegration. The cationic demand values for kraft pulps are slightly higher than in mill B.

Mill C's cationic demand profile is affected by the variety of chemical additives used. They include cationic materials (AKD which contains a cationic polymer and cationic starch) as well as anionic additives (dispersed fillers and a high-molecular-weight polymer used as the retention aid).

The addition of an anionic flocculant results in reduced cationic demand; this effect may be explained in terms of the flocculation of fillers reducing the total available surface area for the polymer adsorption.

This is another example in which cationic demand titration helped to solve a problem. Feeding AKD promoted with a cationic polymer simultaneously with a coagulant, caused an overcationization of the system. This resulted in impaired retention and, consequently, lower sizing efficiency. Diagnosis of a root-cause was based on the cationic demand studies; the doses of coagulant were adjusted and the problem corrected.

GROUNDWOOD SPECIALTY

Mill D: This specialty mill, Fig. 4, produces coated paper and uses furnish elements different from those in mills A, B and C.

The cationic demand of the broke is high, which is characteristic of coated broke systems. Most of this high cationic demand may be attributed to soluble and colloidal materials, so that the cationic

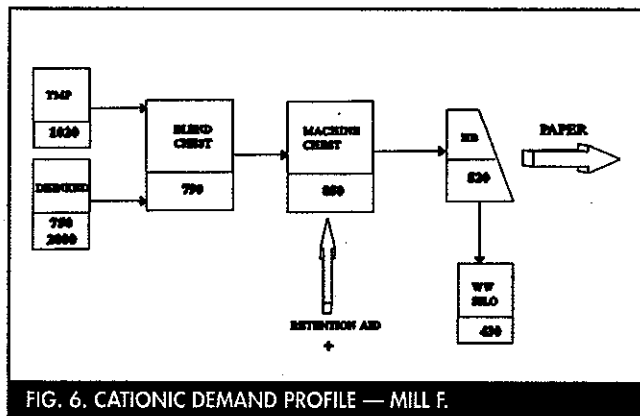


FIG. 6. CATIONIC DEMAND PROFILE — MILL F.

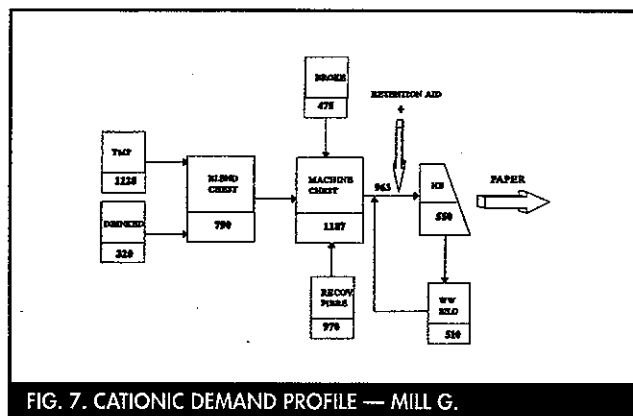


FIG. 7. CATIONIC DEMAND PROFILE — MILL G.

onic demand of broke rapidly decreases on thickening, with the cationic-demand-inducing-species accumulating in the process water.

The cationic demand of SWK is much higher than in mills A, B and C most likely as an effect of a higher level of the cationic demand of recirculated white water. Bleached groundwood has a typically high cationic demand. Adding cationic retention aid and diluting with white water, lower the cationic demand between the consistency regulators.

Cationic demand of the headbox drops as a result of the addition of anionic retention aid and dilution with the white water. A relatively small drop of cationic demand between the headbox and white water suggests that there is quite a large amount of soluble anionic and colloidal material accumulated in the white water loop. This could result in problems with runnability and sheet quality.

The mill had periodically experienced these types of upsets which, to a large extent, are now controlled by treating the coated broke with a coagulant. Since this study was done, the mill is buffering the charge of the headbox from the variations in the charge of the coated broke. This allows it to keep the cationic demand of the headbox within a certain window of operation, improving machine efficiency and product quality.

NEWSPRINT MILLS

Mill E: This mill, Fig. 5, represents a typical example of a cationic demand profile for a newsprint mill operating without any retention aid.

The typical values of cationic demands of sulphite, groundwood and thermomechanical pulps and a high level of cationic demand of the white water loop, are observed. The high cationic demands of the elements of the stock furnish and the lack of any charge-neutralizing additives cause the steady-state charge balance to be reached at a much higher level of cationic demand in the headbox and the white water loop. The accumulation of anionic trash in the

white water loop, more pronounced with the higher degree of closure, will eventually lead to such problems as deposits, holes, linting or brightness loss.

The mill experienced these difficulties and has introduced a retention aid program to the operations.

Mill F: This newsprint mill, Fig. 6, uses large quantities of deinked pulp which exhibited a wide variability of cationic demand. The cationic demand of such pulps varied between 700 and 2000 $\mu\text{Eq/L}$, most likely as a result of upsets in deinked pulp production, varying quality of washing or varying quality of raw materials. It may be useful to monitor regularly the cationic demand of deinked pulp, both prepared on-site or purchased, especially where it forms a large part of the furnish.

Some pretreatment with a coagulant for highly anionic deinked pulps may not only improve charge balance on the machine with all its positive consequences, but also prevent problems with pitch and stickies.

Typical values for the headbox cationic demand of about 500 $\mu\text{Eq/L}$ were observed, although there were no obvious changes in retention, speed and runnability for cationic demands between 400 and 700 $\mu\text{Eq/L}$. Therefore, the charge sensitivity of newsprint mill systems is much lower than that previously observed for fine paper mills which use mostly wood-free pulps and operate at lower levels of cationic demand.

Mill G: This is another example of a newsprint mill, Fig. 7, using TMP and deinked pulp. The low value of cationic demand of the deinked pulp once again demonstrates the large variability in the quality possible with this furnish stock. Cationic demand values for headbox and white water are quite similar to those observed in mill F.

BOARD MILLS

Mill H: This mill, Fig. 8, produces coated board. Cationic demand surveys in board mills are time-consuming because they have to include surveys of the stock

flow for each ply.

The mill experienced a problem associated with size retention and inter-ply bonding. The cationic demands of top and bottom liners and the filler ply are presented. The two set of numbers represent two independent surveys done several months apart.

Both top and bottom plies are built of identical furnishes and treated identically. Therefore, it was quite unexpected to see the large differences in the cationic demands of headbox and white water loops during the problem-solving survey. The differences were caused by an imbalance in the white water circulation system, and after this was corrected the problem was largely reduced.

The survey has been repeated often since then and showed top and bottom liner headbox samples having close and low cationic demand values around 10 $\mu\text{Eq/L}$. Close monitoring of the cationic demand of the top and bottom liners ensures that no over-cationization takes place.

Mill I: This is another example of a board mill, Fig. 9, using 100% recycled furnish in the filler ply and 100% virgin pulps in the liner.

The mill uses a microparticulate retention program and operates in a very closed system. The cationic demand values presented are typical and steady over a long period of time.

CONCLUSIONS

- Using the streaming potential PCD colloidal titration method for the determination of the total cationic demand of a papermaking furnish provides improved accuracy and sensitivity compared with other techniques.

- Standardization of the method, and calibration of both equipment and solutions are essential for the success of long-term monitoring of a papermaking system. They are even more crucial if comparison of data for various systems is attempted. Unfortunately, the level of awareness on this issue among paper-makers and suppliers is generally low.

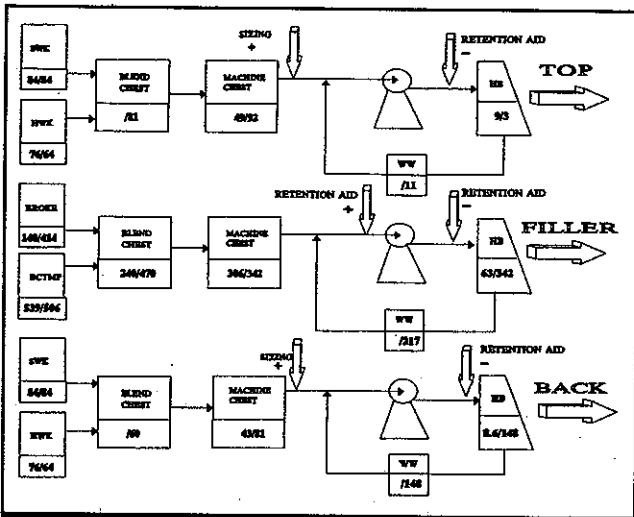


FIG. 8. CATIONIC DEMAND PROFILE — MILL H.

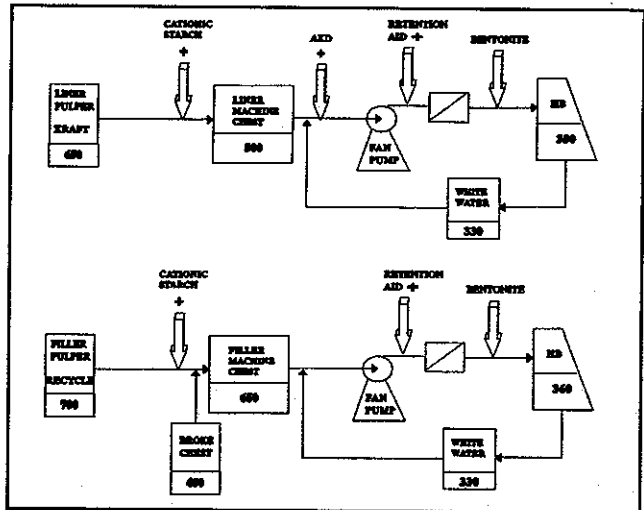


FIG. 9. CATIONIC DEMAND PROFILE — MILL I.

Résumé: Cette communication décrit une méthode de mesure de la demande cationique faisant appel à un détecteur du potentiel d'écoulement comme indicateur du point de virage du titrage. D'autres techniques utilisées pour déterminer la quantité de charge dans le papier sont également passées en revue. La méthodologie et des exemples d'enquêtes sur la demande cationique sont présentés pour des usines de papier fin, de papier spécial à base de pâte mécanique, de papier journal et de carton. Des exemples d'interprétation de ces études sont montrés du point de vue des additifs, des procédés et des problèmes d'exploitation.

Abstract: A method for the measurement of the cationic demand, using a streaming potential detector as an indicator for the equivalent point of the titration, is presented. Other techniques used to determine the charge state of papermaking stock are also reviewed. The methodology and examples of mills' cationic demand surveys are presented for fine paper, groundwood specialty, newsprint and board mills. Examples of the interpretation of these surveys are shown from the point of view of the effect of additives, processes and upsets in operations.

Reference: GRATTON, R., PRUSZYNSKI, P. Cationic demand survey of paper machines: How can we measure and use it? *Pulp Paper Can* 96(3): T103-108 (March 1995). Paper presented at the 80th Annual meeting of the Technical Section, CPPA, at Montreal Qc on February 1 to 4, 1994. Not to be reproduced without permission. Manuscript received November 25, 1993. Revised manuscript approved for publication by the Review Panel August 24, 1994.

Keywords: WET ENDS, CATIONIC COMPOUNDS, FURNISH, MEASUREMENT.

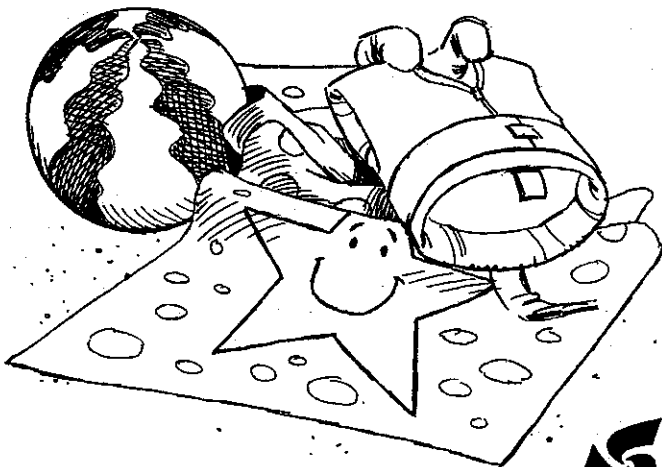
Colloidal titration is strongly method-dependent and following procedures consistently is absolutely necessary.

- Surveys should be done routinely under normal operation in order to build statistically-relevant relationships.
- The method improves understanding of the whole furnish system and can be used for its optimization.
- We encourage others to do their own mill cationic demand surveys and share their results. This will permit the industry to develop a better understanding of charge related phenomena in paper-making.

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Just swimming
around...



...active living makes
a healthy difference!

