Time Variations of Macrostickies and Extractable Stickies Concentrations in Deinking

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The stickies content, both macrostickies and stickies extractable in a solvent, was determined for samples taken at short time intervals from deinking lines, producing deinked pulp for newsprint production. The study was carried out at three mills on different continents, with each having a different source of recycled paper as raw material. The short-term variations in extractable stickies in the incoming raw material were quite extreme, with differences of 100% being seen within hours. Despite this, the final deinked pulp contained fewer sudden variations and had no correlation to the incoming stickies content. While the raw material appeared to affect the incoming stickies content, a well-optimized deinking line was able to buffer the raw material variability, and the final stickies content was more dependent on the deinking process. This result was seen for the two mills examined for this phenomenon, despite a different raw material supply. Macrostickies were found to exhibit the same tendencies, although with smaller and less sudden variations. However, the variations of macrostickies and extractable stickies never correlated, even when both were measured for the same pulp fraction, thus confirming that solvent extraction is not an appropriate method for the determination of macrostickies and is more a reflection of microstickies.

Introduction

Stickies are still a concern in deinking and are among the most detrimental contaminants in recovered paper recycling, affecting both the process efficiency and the quality of the final pulp. Stickies control and treatment are thus an important part of deinking line operations.[1] Assessment of the actions taken requires measurement of both the macrostickies and microstickies, the latter being located mostly in the fines fraction and most responsible for deposition and impaired paper machine performance.[2–4] Assessment can involve either stickies determination in conjunction with stickies control trials or mapping of stickies throughout deinking lines or at the paper machine.

With rapid changes in raw material, the varying stickies content entering the deinking line can affect the interpretation of mill trials even if they last only a few hours, and this paper's origins lie in an attempt to gauge the magnitude and speed of possible variations. In turn, mapping of stickies across a deinking line allows determination of the removal efficiency of different process stages and gives a good overview of the stickies control management in the mill.[5]

Mappings usually involve up to 10 or more sampling points. A few mappings have been published for total extractable detrimental substances[5–7] but also specifically for either stickies[3,8,9] or other specific detrimental components, such as fatty and resin acids.[10] These mappings give a picture of the concentrations of the components in question throughout the process and the removal efficiencies of various stages, which help to determine the behavior of the stickies or other detrimental substances in different stages and conditions. However, these pictures are only snapshots of the process—the situation at any one given moment in time—and do not reflect any variations in the process itself. These variations can be even more revealing because changes in the incoming raw material, changing unit operations, and changing process chemistries can play a large role in the operations of the deinking line and their effectiveness in removing detrimental substances. It can also be difficult to ascertain if these snapshots are representative of normal process conditions.

Some studies carry out the mapping two or three times and then mix the samples to determine an average value, but while this helps, it does not give an accurate picture of how the process can vary with time. Miranda and coauthors carried out two mappings at a time interval of approximately 1 month in order to compare a period of time in which the mill was experiencing deposit problems to a time in which the deposit problems were minimal.[5] While this gives an indication of the extent of the differences in the stickies content that could occur, it does not reveal how quickly these changes could occur. As seen by Banerjee and coauthors, conditions that could potentially lead to runnability problems, as measured by the microorganic content, can change rapidly within hours.[11] It is quite reasonable that the content of stickies could experience similar variations, and a comparison of processes without an idea about the variations of the stickies content can lead to misleading conclusions.

The main reason for this lack of knowledge is the nature of the analysis method. While many characteristics of the process are measured online, stickies measurements are offline, complex, and time-consuming because of the several labor-intensive sample preparation and measurement steps that most methods require. One common method to measure stickies at mill scale is to determine the macrostickies, considered to be the solid and tacky contaminants larger than 150 μm. The effort required usually limits mills to selecting one or two sampling points being sampled once a day, or even more seldom.

Another method is to measure or analyze the extractable content of a sample, such as determination of the total amount

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Table 1. Basic Information for Mills Surveyed and Samples Taken[a]

mill	location	paper supply	furnish ONP:OMG	pulping chemistry	flotation collector	sampling points	stickies measurements
A	USA	USA, Canada	60:40	reduced alkaline	synthetic	cleaner feed, final DIP	extractable and macro
B	Europe	UK, Western Europe	50:50	reduced alkaline	fatty acids	screens feed, screw press accept	extractable and macro
C	China	China, USA	50:50	alkaline	fatty acids	headbox	extractable

[a] ONP = old newspapers; OMG = old magazines. Reduced alkaline refers to either little or no caustic added to the pulper.

soluble in an organic solvent[5,6] or determination of the amount of specific compounds after further analysis of the extract by Fourier transform infrared (FTIR) spectroscopy,[2,12] high-performance liquid chromatography (HPLC),[13] or pyrolysis-gas chromatography (PyGCMS).[8,13] The extractable components include recycled-fiber-based stickies, such as styrene, acrylate, and vinyl acetate based binders and adhesives, as well as wood pitch components from the wood fibers themselves or process chemicals such as deinking soaps.[3,12,13] These methods are even more time-consuming and usually require equipment or expertise not found at the mills, so samples are instead sent to external research institutes. For this reason, few measurements of this type are available for mills, which makes the scope of the work produced in this study unique.

To determine the extractable stickies content in this study, a previously published method was used and consisted of a combination of solvent extraction with tetrahydrofuran (THF), followed by component separation and quantification of the component groups by HPLC.[13] THF was selected based on a previous study for comparison of the solvents.[13] While it is known that THF will not extract all components related to stickies, especially cross-linked and high-molecular-weight polymers, previous experiments involving the extraction of deposits from paper machines have shown that the THF-soluble fraction of a deposit shows extreme tackiness and contains a wide variety of compounds, such as styrene- and acrylate-based polymers, as well as (vinyl) acetates.[13] The nonsoluble fraction, meanwhile, exhibits no tack whatsoever. Therefore, THF will extract the majority of stickies tacky in nature and most likely to actually deposit, which are the most relevant type for a study of this nature.

This method presents the advantage of being able to more accurately determine the stickies content—the components originating from adhesives or binders in recycled fiber—without interference from other soluble components such as wood extractives. Other methods for stickies determination exist. However, many, including microscopic analysis, are even more time-consuming and subjective. One main advantage of chemical analysis over optical analysis is the ability to identify individual components based on their chemical composition as opposed to simple broad categories of stickies based on their general size, shape, or tackiness. Many stickies are too small to be seen, and even visible ones can be hidden or missed because of their attachment to and inside the complex fiber and inorganic particle matrix found in most pulp samples. Despite this, most mills employ a relatively rapid optical stickies determination method using a common computer scanner and specialized software. However, because there is no microscopy involved, the stickies determined are only the relatively large ones, generally greater than 150 μm, and are thus considered to be macrostickies. For this reason, macrostickies determinations were carried out as well, according to mill standard methods.

Both methods, macrostickies and extractable stickies analyses, have been demonstrated as being important and complementary: Blanco and coauthors have demonstrated that an integrated approach is necessary for the full characterization of stickies[7] because different methods give different information. The determination of macrostickies allows us to monitor the visible contaminants that could lead to decreased product quality,[14] while determination of the extractable stickies gives information about potential destabilization of microstickies and dissolved and colloidal material. For this reason, both analysis methods were applied to the same samples when possible.

To investigate the variations with time, samples were taken at two mills at an interval of only a few hours for several days in a row. When many samples across a short time span are analyzed, it is revealed how quickly variations in stickies concentrations can occur. At the third mill, daily samples were taken for approximately 1 month to see the variations that could occur over a longer term. All three mills used a furnish of 100% recycled fiber, using different proportions of old newspapers (ONP) and old magazines (OMG) for newsprint production. However, it should be noted that, despite the fact that all three mills use a fairly similar proportion of ONP:OMG, the actual raw material could vary more because of the fact that each mill receives its recycled paper from different sources. Not only would the original paper production lead to differences in the recycled paper, such as the stickies content, but the different collection programs, including sorting, storage, and transportation time, lead to different qualities of recycled paper. This can not only affect the stickies content but, more importantly, the deinking line operations required to remove the ink, the primary purpose of the deinking line. This, in turn, will affect the stickies removal from the paper and therefore the amount of stickies material in the water systems. In general, however, OMG would be expected to lead to a higher stickies content in the water systems. This is due not only to the presence of binders in the coating of light-weight-coated (LWC) papers, which make up a significant portion of OMG mixtures, but also partly due to stickies being more easily detached from the coating, as opposed to the more difficult detachment of stickies directly from the fibers and fiber networks. Stickies also originate from various glues, labels, and glued-in inserts used in magazines. The raw material types and source locations are listed in Table 1, along with other pertinent details.

Because of the time and effort required to analyze such a large number of samples, the traditional mapping of approximately 10 points was decreased to either one or two: at the beginning and end of the deinking line (mills A and B) or from the headbox (mill C). The locations of these points are shown in a schematic, highly simplified for illustrative purposes, of a deinking line (Figure 1). As seen, the process is divided into three water circuits or loops, with each circuit being fairly isolated from the others in terms of water circulation and quality. The pulp flows forward to each successive loop, but the majority of the process water is circulated within each loop itself, with only a fraction (a few percent of the total water volume in each loop) being sent forward with the pulp fibers. This leads to an accumulation of primarily water-borne components in each circuit, while fiber-borne components are carried forward. The tendency for the components to accumulate means that each

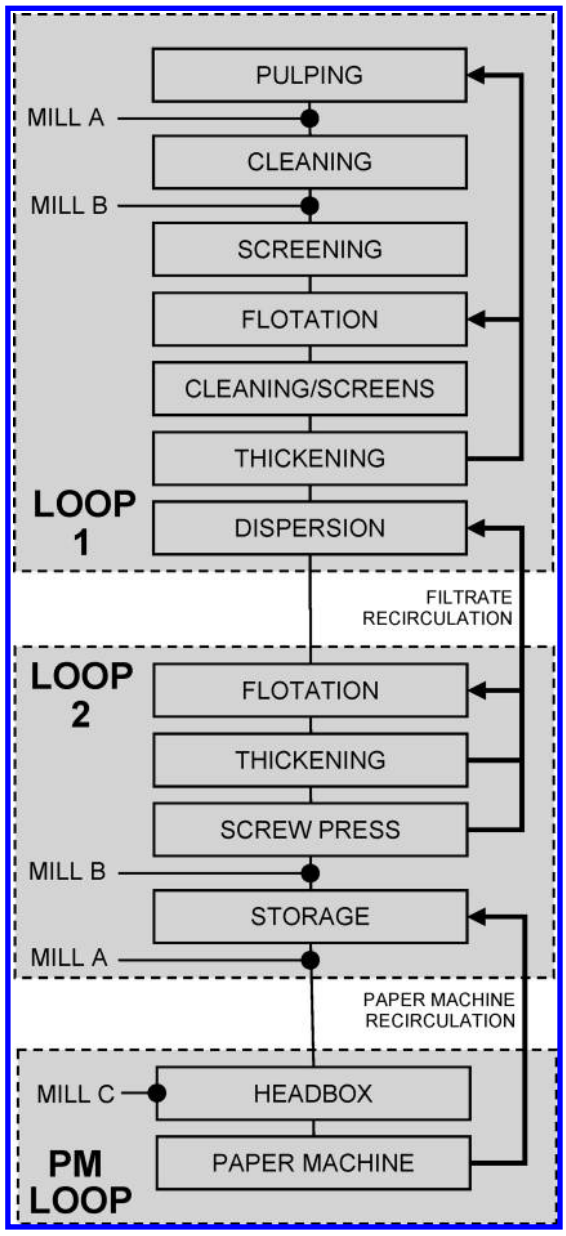

Figure 1. Schematic of a deinking line with sampling points.

successive circuit is cleaner (in terms of chemical oxygen demand, anionic trash, etc.) than the previous.

With a large volume of water in comparison to the volumes entering and exiting, each circuit could be regarded as a separate continuous stirred-tank reactor (CSTR). In this study, however, sampling points are taken at the beginning of loop 1 and at the end of loop 2. The whole deinking line is therefore considered to be one CSTR. This is in accordance with how the issue has sometimes been approached in the literature.[15,16] During regular operation, the residence time of fibers through each circuit would be less than 1 h and even less than 2 h for all three loops. However, because of the various dewatering stages and recirculation of water, the mean residence time of stickies, especially microstickies, would be higher.

In addition to the water circulated within each loop, process water is circulated countercurrent to the pulp flow, as is also seen in Figure 1. The most important recirculation of process water when examining these results from this study is the paper machine (PM) recirculation water, which, although cleaned, contains an accumulation of water-borne components, such as microstickies. This obviously affects the stickies content of the final deinked pulp at the storage tower (final DIP).

Materials and Methods

Macrostickies. Macrostickies were measured by first screening the samples according to TAPPI 275 sp98. Samples were diluted to 1% for screening through a Somerville screen (150 μm) and the screen rejects removed by pressurized water.

The water/rejects sample from the previous step was diluted and filtered through a black wet-strength filter by a Rapid Köthen sheet former. The filter was dried and heated in a press while in contact with a coated paper. The tacky material stuck to the coating during the pressing, and upon removal of the coated paper, the tacky particles were colored white against the black background of the filter paper. Particles not strongly attached to the filter were washed off with water (10 L/min at 1 bar of pressure). The filter was dried and analyzed according to INGEDE Method 4 99-12 using an Epson Expression 1680 Pro with SENTINEL Paper Scan software at 400 dpi resolution and a contrast of 88%. The results were given by the software as millimeters squared, and the macrostickies concentration was given as millimeters squared per kilogram of dry pulp.

Total Extractable Stickies. Samples were first filtered through standard paper filters used for making brightness pads in the mill laboratory. Extractable stickies were analyzed by extracting the air-dried filters in an Accelerated Solvent Extractor 200 (2×5 min cycles, 200 psi) supplied by Dionex using THF as the solvent. The solvent was collected and an aliquot evaporated to 1 mL and injected into an HPLC system, consisting of a size-exclusion column (Jordi 550A) and an evaporating solvent light scattering detector (Sedex 80, nebulizer temperature = 40 °C), with an eluent flow of 1.0 mL/min of THF. The polymer fraction, or the extractable stickies content of the pulp, was separated by the HPLC column from the wood extractives and quantified by a known styrene calibration solution. The method is described in more detail by MacNeil and coauthors.[13] The standard deviation for 10 replicates was previously determined to be less than 4%.

Microstickies. Microstickies (mill B) were determined by taking the difference (in mg/g) between two analyses: analysis of the total extractable stickies (as described above) and analysis of the extractable stickies in the fiber fraction. The latter was obtained by filtering a parallel sample through a dynamic drainage jar with a 100-mesh wire (~150-μm holes). The material remaining on the wire was collected and dried for extraction and analysis (as described above for the total extractable stickies). The difference between the extractable stickies in the total sample and in the fiber fraction was defined as microstickies.

Results

Variations in Extractable Stickies.

Mill A. At mill A, samples were taken at 2−8-h intervals from the feed to the high-consistency cleaner, in other words, close to the beginning of the flotation line and from the final storage silo for deinked pulp. The results for extractable stickies are shown in Figure 2. The feed to the cleaner revealed a steady increase of (extractable) stickies during the 4-day trial, with an almost 4-fold increase from 2 to 8 mg/g. Smaller fluctuations were seen within the period, with changes of 20−30% possible within hours. During this time, operations of the deinking line were kept as steady as possible, with no changes in the pulping

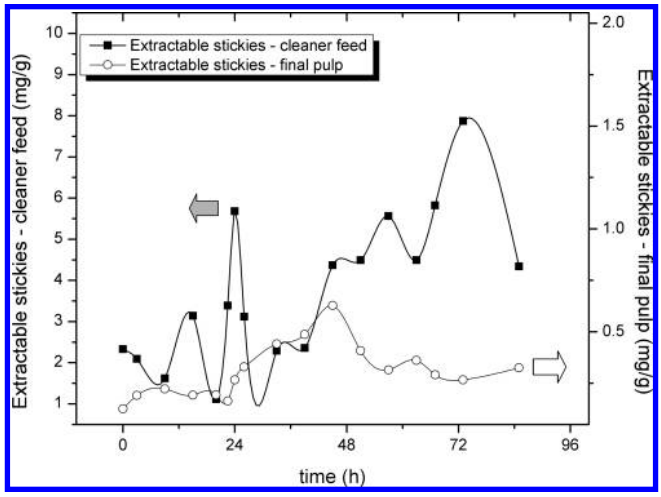

Figure 2. Extractable stickies content for the cleaner feed sample (black squares) and the final pulp sample (white circles) for mill A.

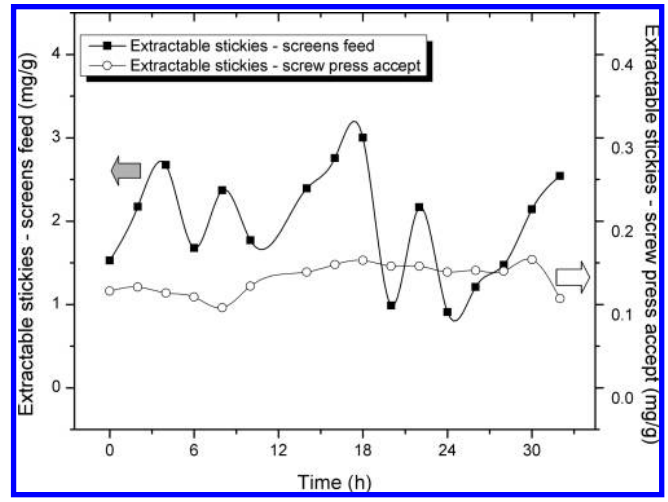

Figure 3. Extractable stickies content for the screening feed sample (black squares) and the screw press accept sample (white circles) for mill B.

Table 2. Extractable Stickies Removal Rates across the Deinking Lines for Mills A and B

mill	first sample	second sample	duration of study (h)	min removal (%)	max removal (%)
A	cleaners inlet	final DIP	85	81	95
B	screens inlet	screw press accept	32	85	95

or flotation chemistry because of the trial underway, which indicates that variations were due to the raw material.

The data from the final pulp samples (Figure 2) showed an even larger difference in stickies, from 0.12 to 0.62 mg/g, or a 5-fold increase, but the samples had smaller short-term fluctuations than the samples from the beginning of the deinking line. It appears that the deinking line is robust and is able to "absorb" variations in the incoming pulp with regard to stickies.

In addition, despite taking into account the retention time of the pulp through the deinking line, no correlation in the stickies content between the incoming and outgoing pulp was found. However, this point is also affected by paper machine operation because the pulp is diluted with paper machine white water, which could increase any variations in stickies concentrations depending upon the white water contents.

Because of the lack of correlation of the stickies content between the beginning and end of the deinking line, the stickies removal capability of the deinking line was not constant, and itself fluctuates, from 81% to over 95% (removal rates of the deinking lines are listed in Table 2). Often deinking lines are reported in the literature or reports as having a certain stickies removal rate, but it is clear that the removal rate is highly dependent on the incoming raw material, perhaps much more than previously believed. The correct comparison of different deinking lines is obviously not easily done with only a few measurements.

Mill B. The first trial from mill B (Figure 3) covered a shorter amount of time but included sampling at slightly shorter time intervals. Two sample points were measured: both the coarse screen accept, again close to the beginning of the deinking line, and the second-loop screw press accept. In this case, the screw press accept was taken instead of the final deinked pulp to help avoid interference from white water from the paper machine used as dilution water, as was the case for mill A.

The concentration of the stickies in the incoming pulp decreased somewhat during the trial, but the fluctuations mostly stayed within almost ±50% of the average, with the highest value almost 3 times the lowest, which is similar to the results from mill A. However, the stickies concentrations in the screw press samples were more stable, staying within ±20% of the average. This is in contrast to the final deinked pulp samples from mill A, which had variations up to 150% of the average value. This could be explained by the lesser influence from dilution waters from the paper machine in mill B, where the screw press accept pulp sample is taken before dilution from paper machine white water.

Again, there is no correlation between the incoming and outgoing stickies from the deinking lines, and the removal rate of stickies in the deinking line varies significantly with time (Table 2). In these trials, however, there was no attempt to keep the operation of the deinking line constant with respect to this study, and the results reflect normal operation and any possible changes that occur. This also could be a reason for a more stable stickies content in the final pulp. As published by Sarja and coauthors, the removal methods of ink and stickies in flotation are closely related, either through stickies attachment to the ink particles or because of similar surface properties, allowing them to be removed to the same extent in flotation.[9] Because an important factor in deinking line operations is to keep the ink content in the final pulp low and stable, changes to achieve this could also inadvertently keep the stickies content low and stable as well.

The analysis method used above measured both stickies attached to fibers or other large particles, as well as stickies free in the water phase, either colloidal or suspended, and defined as microstickies in this study. Because microstickies are most likely to be the cause for deposition and are therefore more detrimental,[2–4] microstickies were determined by measuring the stickies on the whole sample and the fiber fraction and calculating the difference (as described in more detail in the Materials and Methods section).

As seen in Figure 4, the replotted total extractable stickies content in each sample is nearly identical with the microstickies content, with microstickies making up about 90% of the total stickies for nearly every point. This is in the same range as that reported before, where two different pulp samples had 86 and 97% of the stickies passing a 150-μm wire and were analyzed using the same analytical method.[17] As such, it seems that the majority of the variations in stickies in the process are coming from microstickies. Because microstickies are more likely more easily removed in flotation

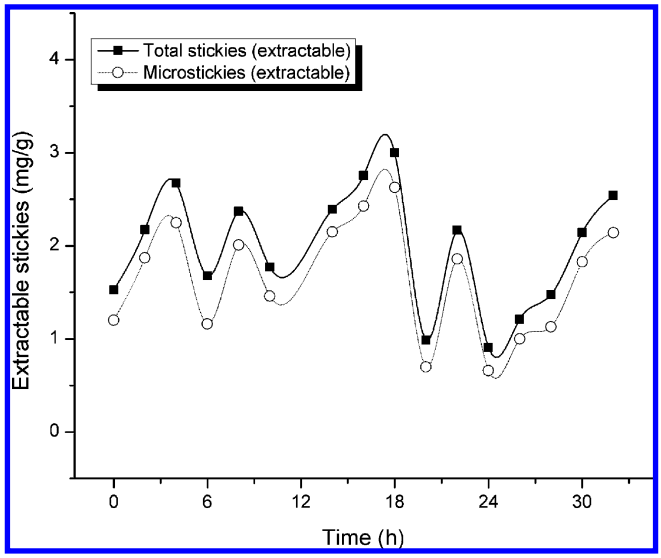

Figure 4. Total extractable stickies content (black squares) and extractable microstickies content (white circles) on the same sample (mill B screw press accept).

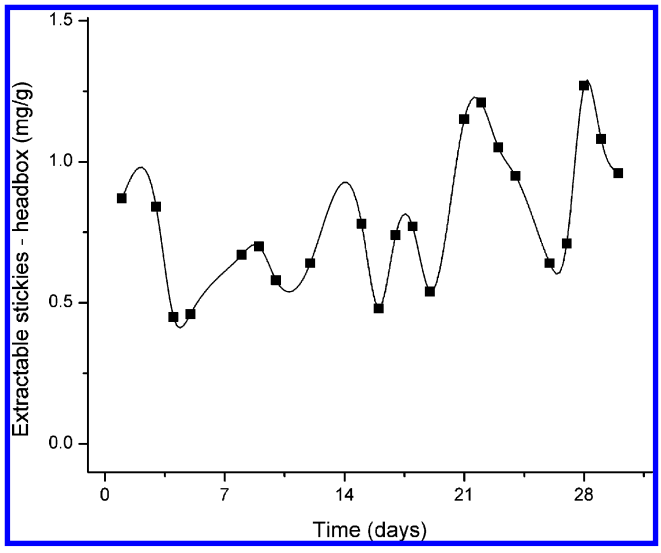

Figure 5. Extractable stickies content in headbox samples for mill C.

units,[2,5,7] it seems reasonable that the flotation cells are able to remove more stickies when more are introduced to the process in the raw material.

Mill C. The trial at mill C involved sampling of the headbox for a period of 1 month. During sampling days, samples were taken throughout the day and mixed at the end for one common, pooled sample to represent the average for that day. These pooled samples were analyzed for extractable stickies and are plotted in Figure 5. As seen, the stickies varied 3-fold across the month, including up to 100% changes on a daily basis. The location of the sample, the headbox along with the overall length of time of the study, makes it difficult to determine the reason behind the variations because both mill operations and raw material can play a role. The mill reported the switching of recycled fiber sources during the month, so the reason is most likely a combination of the two.

Variations in Macrostickies. A second trial at mill B was carried out with samples taken from the inlet to screening every 4 h for 4 days. Both macrostickies and total extractable stickies were measured and are shown in Figure 6. The macrostickies

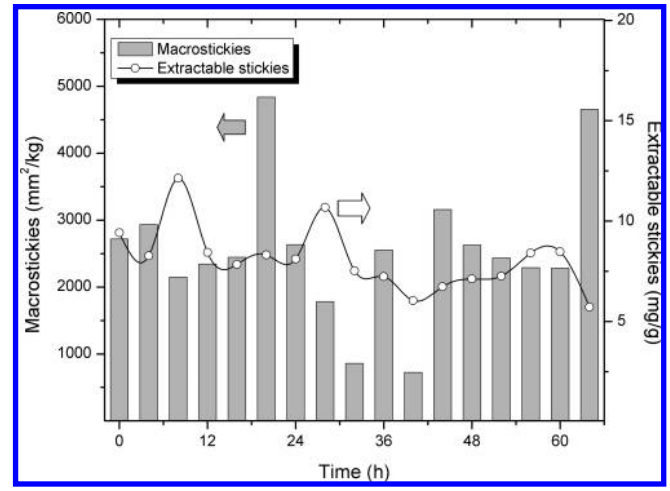

Figure 6. Macrostickies (gray columns) and extractable stickies content (white circles) for mill B (screening stage inlet pulp), first trial.

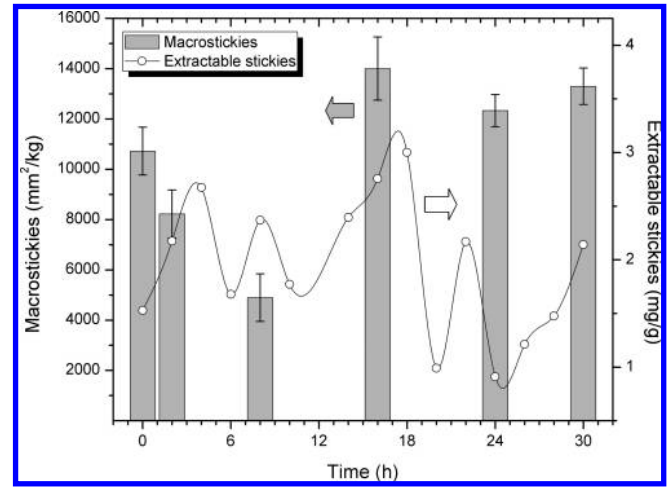

Figure 7. Macrostickies (gray columns) and extractable stickies content (white circles) for mill B (screening stage inlet pulp), second trial.

seemed to have fewer variations with only three or four exceptions, although those exceptions were quite significant in magnitude. Extractable stickies were measured as well and had fewer variations than the previous trials, but again with a few significant exceptions. However, when the two are examined, the exceptions did not correlate with each other: i.e., there is no relationship between macrostickies and extractable stickies concentrations in the raw material.

The study was carried out again, but this time three replicates for the macrostickies were performed, in order to ensure that the exceptions were not outliers due to analytical error (Figure 7). The area of the macrostickies was larger during this trial—from 3 to 4 times higher—although the extractable stickies were actually lower than those in the previous trial. In any case, the error bars show that the variations far exceed any error due to analysis. Again, the changes within a single operations shift at the mill could be 100% for both extractable stickies and macrostickies.

The macrostickies content at the end of a deinking line was also measured at mill A, with samples taken every 4 h for nearly 4 days (Figure 8). Here, the macrostickies varied in amount much more than those for mill B, even though the deinking lines were thought from previous results to even out fluctuations, at least for the extractable stickies. In fact, even differences of 300% from one sample to the next, a period of several hours, could be seen. When the extractable stickies were plotted on

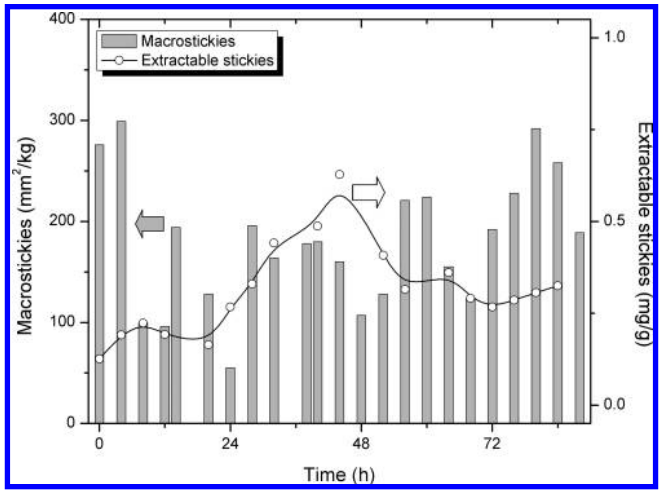

Figure 8. Macrostickies (gray columns) and extractable stickies content (white circles) for mill A (final deinked pulp).

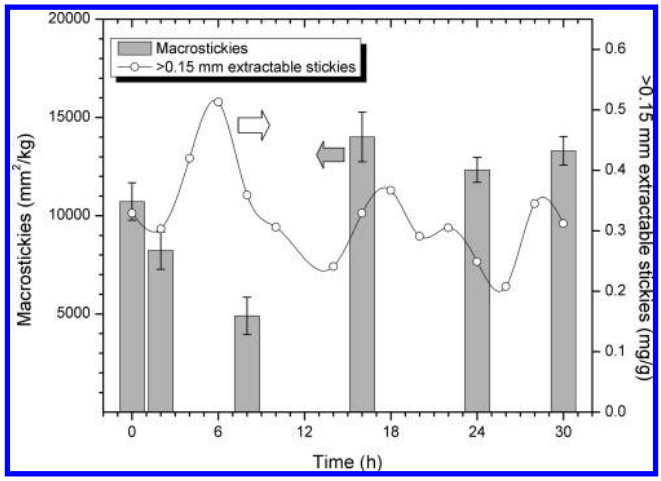

Figure 9. Macrostickies (gray columns) and extractable stickies content (white circles) on the fiber fraction (>0.15 mm) for mill B (screening stage inlet pulp).

top, it is seen that the variations are much less between any 2 consecutive samples. However, it is seen that macrostickies leaving the deinking line, once again, do not correlate with extractable stickies. This lends credence to the complementary approach of different stickies determination methods suggested by Blanco and coauthors.[7]

In Figure 9, the macrostickies data from Figure 7 is replotted (columns), while the extractable stickies retained by the 150-μm wire are plotted on top (black line). If macrostickies are fully extractable in THF, the two sets of data should in theory correlate because they are both measured on the reject of 150-μm screens. However, it is clearly seen that no such correlation exists. Because it has been seen that macrostickies are at least partially soluble in THF,[18] this lack of correlation is most likely due to extractable (THF-soluble) stickies attached to fibers influencing only the extractable stickies determination because the fibers are washed away only during the macrostickies analysis. In any case, the macrostickies content, on any fraction of pulp, cannot be determined by dissolution in THF.

Conclusions

Stickies entering the deinking lines are seen to fluctuate with time, with daily variances in the concentration up to 100% or more as seen in two mills with deinking lines using recycled fiber for newsprint production. The reason for these fluctuations seems to be the raw material entering the mill, even if an attempt is made to keep the quality of the raw material supply constant. This is despite the fact that all three mills, while using nearly the same raw material type, have very different raw material sources: North America, Western Europe, and China.

However, despite these fluctuations in the incoming raw material, the deinking lines were seemingly able to cope well and, in general, removed more stickies when more contaminated pulps were used, thus evening out the stickies concentrations in the final deinked pulp. This was possibly due to an inherent capability of the units, but it could also be due to the slight changes made in the deinking line operation in response to other factors. In any case, the final deinked pulp had a much more stable concentration of stickies than the raw material. Apparently, while the stickies content entering the deinking line is dependent upon the raw material, the final stickies content is more dependent upon the operation of the deinking lines.

It is clear that more extensive measuring programs are required if representative results are to be obtained. Measuring sample points only a few times, far apart in time, will not give a representative picture of the process when considering both the stickies content and stickies removal in industrial deinking lines. This fact should be considered when the performances of different mills or different sampling times are compared.

It is also seen that the macrostickies contents in pulps, both entering and leaving the deinking lines, have wide variations, on the same scale as extractable stickies. However, there is no real correlation in concentrations between the two in this study. It appears that even if macrostickies are partially soluble in an organic solvent, extraction of pulps is not sufficient for macrostickies determination. Therefore, a complementary approach is required.

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