STICKIE REMOVAL USING NEUTRAL ENZYMATIC REPULPING AND PRESSURE SCREENING

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ABSTRACT

Removal of stickie contaminants is currently a major focus of paper recycling research. Medium consistency alkaline repulping followed by pressure screening has proven to be effective for stickie removal. There is, however, an alternate method that is equally effective and more environmentally benign. This study compares the effectiveness of this alternative method, neutral enzymatic repulping, with conventional alkaline repulping for contaminant removal from a controlled office paper furnish spiked with typical pressure-sensitive adhesives. Neutral repulping on a pilot scale was performed using a mixture of cellulase and lipase preparations at the ambient pH of the paper furnish. Sequential pressure screenings, flotation, and washing comprised the deinking process, and contaminant count on handsheets prepared after each step was used for these evaluations. We found that pressure screening was substantially more effective for contaminant removal when the pH was neutral. The enzyme mixture further enhanced both the freeness and brightness of the deinked pulp. Process water from the enzyme treatment also contained fewer microstickies, an added benefit for improved water quality and mill performance.

INTRODUCTION

The environmental benefits of recycling paper under neutral conditions have been well established by others (1,2). These advantages have become even more important with the trend towards closure of mill water systems. Previous work with enzymatic deinking at neutral pH confirmed that neutral deinking works and that there is no detrimental impact on process water (3,4). Recently, we have found in laboratory trials that enzyme preparations used at the ambient pH of the paper furnish are more effective than alkaline processing for the removal of stickie contaminants (5). In those experiments, we used vibrating flat screens and flotation for stickie removal. Because pressure screening requires a large quantity of pulp for processing, we did not at that time evaluate the enzyme-treated pulps with pressure screens.

In this study, we report an intermediate scale up of a controlled postconsumer office paper furnish spiked with 5% typical pressure sensitive adhesives (PSAs). Here, we observed the influence of the interaction of printing inks with mixed stickies on contaminant removal with pressure screens. Pressure screening through 0.30-mm and 0.15-mm slotted screens was followed by flotation and washing. To evaluate pressure screening without using the USDA, Forest Service, Forest Products Laboratory’s (FPL’s) 1,500-L capacity hydropulper, we repiped the pressure screen unit to permit recirculation of pulp that was repulped in a 50-L capacity pulper. This adaptation simplified the process of comparing neutral and alkaline repulping. Three trials were run: (a) alkaline repulping without surfactant added, (b) neutral enzymatic repulping with surfactant included in the pulper, and (c) neutral pulping with surfactant only. Samples were taken after each step in the processing to determine the efficiency of stickie removal at each stage. Efficiency of removal was assessed by contaminant count of handsheets made after each stage. The microstickie content in process water from each screening run was also measured.

EXPERIMENTAL

Materials

We used a 50-50 blend of unprinted copy paper and a sorted high quality mixed office paper containing both laser and offset printing for the paper furnish. We applied small pieces of PSAs, typical of those found in reclaimed office papers, to the unprinted paper. Based on oven dry (OD) weight of the paper, a total of 5% PSAs was added: 3% the adhesive currently used by the U.S. Postal Service (USPS) for their self-sticking postage stamps, 1% of an acrylic- based adhesive, and 1% of commercial address labels. The adhesive used for postal stamps and the acrylic- based PSA were obtained as adhesive-coated paper rolls made for the USPS/FPL study of PSAs. Address labels suitable for laser printing were purchased from an office supply store. The percentage adhesive applied in all cases was based on the weight of the adhesive and its paper backing. All paper was shredded prior to pulping.

The enzyme preparations used were Novazym 342, a cellulase preparation also containing various hemicellulase activities and Resinase A 2X, a lipase designed for pitch control. Both enzymes were provided by Novo Nordisk Biochem of North America (Franklinton, NC) and were used at a level of 0.05% each on OD pulp. A total of 0.1% nonionic surfactant (BRD 2340, Buckman Laboratories (Memphis, TN)) was added to each trial. Because of the synergy existing between an enzyme preparation and surfactant in the pulper, the surfactant addition was split between the pulper and the flotation cell in Runs 2 and 3. All of the surfactant was added into the flotation cell in Run 1.

Methods and Equipment

A 50-L capacity high consistency pulper (Voith Sulzer (Appleton, WI)) was used for repulping each 6.5-kg batch of shredded paper. Soft city water was added during a 5-mm fiberization after which the temperature and consistency were adjusted to achieve a final blend of 13% consistency at 45 to 47°C. Sodium hydroxide was added to bring the pulp to pH 10 for the alkaline trial (Run 1). Pulping continued for 20 min. For Run 2 (the neutral pH enzyme trial), paper was fiberized for 5 min with 0.05% nonionic surfactant. After fiberization, we added a dilute mixture of enzymes (0.05% each of the Novazym 342 and Resinase A based on OD pulp) and pulped for 25 min at the ambient furnish pH, 7.9 to 8.0. A total of 0.1% combined enzyme concentrates as received from the supplier was used. Run 3 followed the procedure described for Run 2 excluding the enzymes. The additional 5-min pulping time was required to completely eliminate fiber bundles in the neutral pH runs. Apparently, 20 min was adequate for complete repulping under the alkaline conditions of Run 1 using this pulper at medium consistency. When a larger capacity pulper is used, no compensation time should be necessary.

After pulping was complete, the pulp was transferred to a 150 gal (0.57 m$^3$) holding tank and diluted with 40°C soft city water to achieve a target consistency of 1.1% for pressure screening. The pressure screen (Voith Multifactor Model 00) was started and the system was equilibrated by returning both the accepts and rejects back to the original stock tank. No dilution water was used in the screening process. The feed pressure to the 0.30-mm slotted screen was held at 0 psia with approximately 2 to 3 psi (13.8 to 20.7 kPa) pressure drop across the screen. The accepts flow was adjusted to 200 to 225 L/min, while the reject flow was set at 20 to 30 L/min. Once the flows were maintained, accepts were directed to an empty tank and the reject flow was diverted to a waste screen box. Accepts from the 0.30-mm screen were subsequently passed through a 0.15-mm slotted pressure screen using the procedure described above.

Accepts from the 0.15-mm screen were floated at 1% consistency in a Denver Co. (Colorado Springs, CO) flotation cell. Surfactant was added to meet a 0.1% total charge on OD pulp (0.1% for Run 1 and 0.05% for Runs 2 and 3). Flotations were done at ~43°C. Flotation accepts were dewatered on a 150 mesh (0.104-mm) sidehill screen. Dewatered pulp was washed at low consistency and dewatered and rinsed over a 150 mesh screen. Representative samples were taken from each step described above for handsheets needed for contaminant analysis.

Standard TAPPI handsheet making protocol (T-205-om 88) was followed with modified pressing. Sheets were pressed only once for 2 min to prevent stickie contaminants from spreading or sticking to handsheet disks according
to the protocol developed by Ross Sutherland for the USPS work (6). Air-dried sheets were dyed with Morplas blue dye, which stains contaminants dark blue. Sheets were counted in the 0.02 to 4.0 mm² range on an Optomax (Hollis, NH) speck check scanner at 100 detection level, 600 dpi resolution, no shade compensation.

Microstickies were measured in process water after pressure screening following a modified Doshi method (7), which uses three-ply polyfoam to adsorb microstickies. Because the presence of fiber interferes with microstickie adsorption on the foam, we removed fibers from 8 L of low consistency screen accepts by dewatering it over a 150 mesh screen. This process water was then heated in a stainless steel bucket to 65°C and was slowly stirred at the same time. Weighed polyfoam strips were suspended in the water and microstickies were collected for 30 min. The strips were removed from the bucket, dipped into cold water to “set” the microstickies, and oven dried prior to weighing. Increase in the weight of the polyfoam was assumed to be microstickies contained in the process water after screening a normalized weight of fiber, 30 g.

**REPULPING RESULTS**

Results of the three runs are summarized in Table I. Each run was sampled at five points in the deinking process: from the pulper, after the 0.30-mm slotted screen, after the 0.15-mm screen, from flotation accepts, and from the final washed and dewatered pulp. Even though a comparable paper mix spiked with identical levels of PSAs was used in the three runs, initial pulper counts differ widely, ranging from 1,781 ppm for Run 1 (the alkaline control) to 4,026 ppm for Run 2 (the neutral pH enzyme trial). The contaminant count of the pulper in Run 3 was 3,115 ppm. After the first (0.30-mm) pressure screening, the contaminant level for Run 1 more than doubled (4,355 ppm). In comparison, however, screening lowered the stickie level to 1,540 and 1,590 ppm, respectively, for Runs 2 and 3. Subsequent screening through the 0.15-mm slots reduced the contaminant level in all three runs but most significantly in Run 2, mainly due to the larger mean particle size (Table II) of the contaminants in that run. Better detachment of ink and stickies by the enzymatic treatment and the resulting enhanced pulp freeness also contributed to superior contaminant removal. Flotation was most effective in Runs 1 and 3 in which the contaminants were decreased to 460 and 91 ppm, respectively. This might be explained by the mean particle sizes, which were more appropriate for removal. Handsheets from Run 2 contained 203 ppm contaminants after flotation. Washing was very effective in reducing the contaminants in Run 2 to 13 ppm compared with 328 and 73 ppm for Runs 1 and 3. This was attributable to the small particle size after flotation and enhanced freeness which facilitates removal of detached contaminants.

The large differences in the initial contaminant counts in the three pulper trials need explanation. We assume that the alkaline medium in Run 1 increases the tackiness of the stickie contaminants and partially solubilizes some of them. Tacky contaminants tend to ball up during medium consistency pulping which exposes less surface area for detection in a handsheet. Solubilized stickies remain as colloidal or microstickies with the pulp and are partly removed on the sheet mold. Unlike flat, thin toner ink platelets, stickies are more three dimensional, which makes them more difficult to quantify accurately by measuring only the surface of a handsheet.

Pressure screening usually is an excellent means for stickie removal (8,9). Unexpectedly, the stickie count increased dramatically with screening in the alkaline run. It is clear from the counts in the accepts after the first pressure screening (4,355 ppm) that Run 1 contained a similar amount of stickies as the other trials. This dramatic increase in stickies after the 0.30-mm screening might be explained by extrusion of macro stickies plasticized by alkaline pH through the pressure screen, by adsorption of colloidal or solubilized microstickies onto fibers after the pH dropped during dilution prescreening, or by fragmentation of stickies by mechanical shear (10,11). Subsequent screening through the 0.15-mm screen reduced the count somewhat; however, the contaminant level was still well above the original pulper count. Flotation was extremely effective in reducing contaminants; however, these accepts were still unacceptably dirty compared with flotation accepts from the neutral pulping runs. Subsequent washing was not very helpful in Run 1, suggesting that small contaminant particles were still intimately associated with fibers.

Contaminant removal observed during the processing of Run 2 was similar to that of Run 3 (Runs 2 and 3 were both neutral pulping runs), and contaminant removal from these runs was uniformly greater than that from Run 1. Although the actual numbers detected on handsheets from comparable processes vary, a common trend is evident.
As expected, contaminants are reduced in each sequential process in Runs 2 and 3. Pressure screening is the single most important step for contaminant removal in these runs, reducing the initial contaminants by almost 88% for Run 2 and 65% for Run 3. Gentle attrition in the pulper at neutral pH kept the contaminants large and less elastic, which assured good screening. The mean average particle sizes in Runs 2 and 3 are significantly larger than those in Run 1, facilitating removal by the screens. Enhanced freeness of the enzyme-treated pulp further improved separation. Flotation and washing built upon efficient screen removal, bringing the final count down to 13 ppm for Run 2 and 73 ppm for Run 3.

In addition to the contaminants counted within the official dirt count range of 0.02 to 4.0 mm$^2$, we compared the contaminants smaller than 0.02 mm$^2$ and larger than 4.0 mm$^2$ that were detected but not averaged into the reported residual contaminants for each run. These data are summarized in Table II. All samples taken from Run 1 contained substantially more residual contaminants both smaller than and larger than those detected in Runs 2 and 3 pulped under neutral conditions. Examining counts detected outside of the TAPPI standard range further magnifies the reduced contaminant removal under alkaline conditions. Particles larger than 4.0 mm$^2$ that passed through the 0.30-mm screen in Run 1 resisted removal in subsequent processing. These large particles are clearly visible and need to be separated from the pulp and removed from the furnish. Many of these smallest particles are still within the visible range, which is aesthetically unacceptable as well as being problematic for agglomeration and redeposition in subsequent phases of deinking or papermaking.

Microstickies were also measured in the process water accepts following pressure screening. As expected from our previous work (5), more microstickies were collected from a sampling of ~30 g OD pulp from the alkaline run than from the neutral enzyme Run 2. These results are summarized in Table III. These colloidal stickies contained in process water can resurface as agglomerated stickies when the temperature or pH is altered in subsequent processes in a deinking or papermaking mill.

Brightness and freeness also were measured on the deinked pulp. Final pulp from the enzymatic trial, Run 2, had a brightness of 85.2% ISO, 2 points brighter than the alkaline trial and 1 point brighter than Run 3. This improvement is consistent with the use of xylanase-containing enzyme preparations as well as low dirt count, which also influences brightness of a pulp furnish. The freeness of Run 2 was 403 Canadian standard freeness (CSF), approximately 70 points higher than either of the other runs, a typical result of repulping with a cellulase (3,4).

CONCLUDING REMARKS

These trials are a positive endorsement for neutral pulping. Repulping at the ambient pH of the paper furnish resulted in superior stickie contaminant removal as well as fewer microstickies in the process water compared with conventional alkaline repulping. Addition of cellulase and lipase to the pulper improved contaminant removal, pulp freeness, and pulp brightness. Pressure screening was extremely effective in stickie removal from trials pulped at ambient pH of the paper furnish. We plan to run an additional trial to determine the role of surfactant addition in the pulper at pH 10.

REFERENCES


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Table I. Residual contaminants measured on handsheets sampled at five points during processing

<table>
<thead>
<tr>
<th>Point in process</th>
<th>Run 1 (ppm)</th>
<th>Run 2 (ppm)</th>
<th>Run 3 (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pulper</td>
<td>1,781</td>
<td>4,026</td>
<td>3,115</td>
</tr>
<tr>
<td>After 0.30-mm screen</td>
<td>4,355</td>
<td>1,540</td>
<td>1,590</td>
</tr>
<tr>
<td>After 0.15-mm screen</td>
<td>3,178</td>
<td>660</td>
<td>1,245</td>
</tr>
<tr>
<td>Flotation</td>
<td>460</td>
<td>203</td>
<td>91</td>
</tr>
<tr>
<td>Washing</td>
<td>377</td>
<td>13</td>
<td>73</td>
</tr>
</tbody>
</table>

Table II. Mean average particle size of contaminants 0.02 to 4.0 mm\(^2\) counted by speck check and particles less than 0.02 mm\(^2\) and greater than 4.0 mm\(^2\), but not included in contaminant count

<table>
<thead>
<tr>
<th>Mean average particle size (mm(^2))</th>
<th>Residual particles (ppm)</th>
<th>Residual particles (ppm)</th>
<th>Residual particles (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>&lt;0.02 mm(^2)</td>
<td>&gt;4.0 mm(^2)</td>
<td></td>
</tr>
<tr>
<td>Run 1</td>
<td>Run 2</td>
<td>Run 3</td>
<td>Run 1</td>
</tr>
<tr>
<td>0.07</td>
<td>0.19</td>
<td>0.13</td>
<td>1,095</td>
</tr>
<tr>
<td>0.08</td>
<td>0.14</td>
<td>0.11</td>
<td>1,811</td>
</tr>
<tr>
<td>0.06</td>
<td>0.09</td>
<td>0.08</td>
<td>1,689</td>
</tr>
<tr>
<td>0.12</td>
<td>0.10</td>
<td>0.11</td>
<td>112</td>
</tr>
<tr>
<td>0.08</td>
<td>0.05</td>
<td>0.08</td>
<td>146</td>
</tr>
</tbody>
</table>

Table III. Microstickies measured by adsorption on polyfoam from process water after screening 30 g of pulp through 0.30-mm and 0.15-mm pressure screens and present on handsheets made from pulp at comparable stages of processing

<table>
<thead>
<tr>
<th>Sample</th>
<th>Microstickies</th>
<th>In process water (mg/30 g pulp)</th>
<th>On handsheets(^a) (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.30-mm screen</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Run 1</td>
<td>3.6</td>
<td>1,811</td>
<td></td>
</tr>
<tr>
<td>Run 2</td>
<td>2.9</td>
<td>300</td>
<td></td>
</tr>
<tr>
<td>Run 3</td>
<td>5.6</td>
<td>391</td>
<td></td>
</tr>
<tr>
<td>0.15-mm screen</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Run 1</td>
<td>4.7</td>
<td>1,689</td>
<td></td>
</tr>
<tr>
<td>Run 2</td>
<td>1.7</td>
<td>208</td>
<td></td>
</tr>
<tr>
<td>Run 3</td>
<td>3.3</td>
<td>425</td>
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\(^a\) Contaminants <0.02 mm\(^2\).
### Pressure Screen Trials

<table>
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<tr>
<th>stickies-ppm</th>
<th>Run 1</th>
<th>Run 2</th>
<th>Run 3</th>
<th>Run 4</th>
<th>Run 5</th>
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</thead>
<tbody>
<tr>
<td>pulper</td>
<td>1781</td>
<td>4026</td>
<td>3115</td>
<td>6413</td>
<td>3956</td>
</tr>
<tr>
<td>12 cut</td>
<td>4355</td>
<td>1540</td>
<td>1590</td>
<td>2597</td>
<td>1755</td>
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<tr>
<td>6 cut</td>
<td>3178</td>
<td>660</td>
<td>1245</td>
<td>1285</td>
<td>539</td>
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<tr>
<td>flotation</td>
<td>460</td>
<td>203</td>
<td>91</td>
<td>279</td>
<td>129</td>
</tr>
<tr>
<td>wash</td>
<td>377</td>
<td>13</td>
<td>73</td>
<td>261</td>
<td>71</td>
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</tbody>
</table>

**mean size in sq mm**

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<thead>
<tr>
<th></th>
<th>pulper</th>
<th>12 cut</th>
<th>6 cut</th>
<th>flotation</th>
<th>wash</th>
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</thead>
<tbody>
<tr>
<td>0.07</td>
<td>0.19</td>
<td>0.12</td>
<td>0.06</td>
<td>0.12</td>
<td>0.05</td>
</tr>
<tr>
<td>0.08</td>
<td>0.14</td>
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<td>0.09</td>
<td>0.11</td>
<td>0.09</td>
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<td>0.12</td>
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<td>0.07</td>
<td>0.06</td>
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<td>0.05</td>
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<td>0.09</td>
<td>0.06</td>
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</table>

**>0.02 sq mm**

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<th>flotation</th>
<th>wash</th>
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<tbody>
<tr>
<td>1095</td>
<td>650</td>
<td>738</td>
<td>1417</td>
<td>1021</td>
<td>355</td>
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<tr>
<td>1812</td>
<td>300</td>
<td>391</td>
<td>476</td>
<td>381</td>
<td>360</td>
</tr>
<tr>
<td>1689</td>
<td>208</td>
<td>425</td>
<td>381</td>
<td>92</td>
<td>50</td>
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<tr>
<td>147</td>
<td>14</td>
<td>38</td>
<td>55</td>
<td>50</td>
<td>50</td>
</tr>
</tbody>
</table>

**total ppm detected**

<table>
<thead>
<tr>
<th></th>
<th>pulper</th>
<th>12 cut</th>
<th>6 cut</th>
<th>flotation</th>
<th>wash</th>
</tr>
</thead>
<tbody>
<tr>
<td>2876</td>
<td>4576</td>
<td>3853</td>
<td>7830</td>
<td>4977</td>
<td>355</td>
</tr>
<tr>
<td>6167</td>
<td>1840</td>
<td>1981</td>
<td>3073</td>
<td>2110</td>
<td>381</td>
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<tr>
<td>4867</td>
<td>888</td>
<td>1670</td>
<td>1666</td>
<td>899</td>
<td>329</td>
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<tr>
<td>573</td>
<td>258</td>
<td>109</td>
<td>329</td>
<td>221</td>
<td>111</td>
</tr>
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<td>524</td>
<td>27</td>
<td>111</td>
<td>316</td>
<td>121</td>
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**Freeness**

<table>
<thead>
<tr>
<th>Run 1</th>
<th>Run 2</th>
<th>Run 3</th>
<th>Run 4</th>
<th>Run 5</th>
</tr>
</thead>
<tbody>
<tr>
<td>Freeness</td>
<td>Brightness</td>
<td>Ash</td>
<td></td>
<td></td>
</tr>
<tr>
<td>ml CSF</td>
<td>%</td>
<td>%</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Run 1</td>
<td>335</td>
<td>82.9</td>
<td>9.1</td>
<td></td>
</tr>
<tr>
<td>Run 2</td>
<td>404</td>
<td>85.2</td>
<td>7.5</td>
<td></td>
</tr>
<tr>
<td>Run 3</td>
<td>330</td>
<td>83.0</td>
<td>8.1</td>
<td></td>
</tr>
<tr>
<td>Run 4</td>
<td>360</td>
<td>84.3</td>
<td>9.5</td>
<td></td>
</tr>
<tr>
<td>Run 5</td>
<td>330</td>
<td>84.2</td>
<td>10.4</td>
<td></td>
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</tbody>
</table>

### removal efficiency

<table>
<thead>
<tr>
<th></th>
<th>Run 2</th>
<th>Run 3</th>
<th>Run 4</th>
<th>Run 5</th>
</tr>
</thead>
<tbody>
<tr>
<td>12 cut</td>
<td>61.7%</td>
<td>49.0%</td>
<td>59.5%</td>
<td>55.6%</td>
</tr>
<tr>
<td>6 cut</td>
<td>84.6%</td>
<td>60.0%</td>
<td>80.0%</td>
<td>86.4%</td>
</tr>
<tr>
<td>flotation</td>
<td>95.0%</td>
<td>97.1%</td>
<td>95.6%</td>
<td>96.7%</td>
</tr>
<tr>
<td>wash</td>
<td>99.7%</td>
<td>97.7%</td>
<td>95.0%</td>
<td>98.2%</td>
</tr>
</tbody>
</table>

**total area objects detected**

<table>
<thead>
<tr>
<th></th>
<th>in sq mm</th>
</tr>
</thead>
<tbody>
<tr>
<td>pulper</td>
<td>457</td>
</tr>
<tr>
<td>12 cut</td>
<td>1194</td>
</tr>
<tr>
<td>6 cut</td>
<td>822</td>
</tr>
<tr>
<td>flotation</td>
<td>98</td>
</tr>
<tr>
<td>wash</td>
<td>83</td>
</tr>
</tbody>
</table>

**Neutral Enzymatic Repulping for Stickie Removal**

Sykes, Klungness, Gleisner, Abubakr

**Additional data**