

**Quarterly Progress Report For: Replacing Chemicals in Recycle Mills with Mechanical Alternatives****Covering Period:** October 1, 1999 to December 31, 1999**Date of Report:** January 13, 2000**Contractor:** Institute of Paper Science and Technology**Award Number:** DE-FC36-99GO10381**Contact:** Sujit Banerjee (404) 894-9709; s.banerjee@ipst.edu**Project Team:** DOE-HQ contact (Valri Robinson), AF&PA contact (Conni Kunzler), project mentor (Tom Friberg, Weyerhaeuser, Jim Ramp, Southeast Paper).

**Project Objective:** The objective of this project is to explore potential applications of underwater pulsed power technology to the paper industry. These included fiber refining, disinfection, stickies dispersion, and stickies control.

**Background:** In pulsed power a spark is discharged underwater. Present commercial applications of the technology include the detonation of land mines, zebra mussel control, and water disinfection. In preliminary work we have found that stickies are rapidly dispersed under the influence of pulsed power. The purpose of this project is to explore other applications of the technology in the recycle paper industry, especially in applications where the technology can replace potentially expensive chemical use.

**Status:** We have found that sparking improves the screenability of stickies in the presence of pulp. Sparking is also able to detackify pitch coated on metal surfaces. This is potentially a major finding since it extends the range of the application from recycle to virgin mills. We will need to determine if the effect also occurs for pitch particles suspended in whitewater. Some stickies such as Robond, a PSA used in the paint industry, are relatively unaffected by spark treatment. We find that this polymer is oxidized to a lower extent. A sparker unit has been purchased and installed at IPST. A licensing agreement with Sparktec Environmental, Stoney Creek, Canada, the manufacturer of the device, has been negotiated.

Details are provided in the Attachment.

**Plans for the Next Quarter:**

- Run experiments with stickies from cleaner rejects. These have been obtained from Consolidated.
- Verify the improvement in stickie screenability with a real mill furnish. This has been obtained from Abitibi, Tacoma.
- Determine the effect of sparking on zeta potential.
- Spectroscopically characterize the stickie surface before and after sparking.
- Determine the range of the process.
- Plan field trials.

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ID Number	Task/Milestone Description	Planned Completion	Percent Completion	Actual Completion	Comments
1.1	Laboratory studies on stickie detackification.	12.00	20		
1.2	Study of stickies attached to machine surfaces.	12.99	75		
1.3	Mechanism of stickie passivation.	12.00	40		
1.4	Application to stickies dispersion.	6.00			
1.5	Depth profiling of detackification.	3.00	20		
1.6	Comparative study of other means of inducing detackification.	12.00	10		
2	Studies on fiber refining.	12.99	100	6.99	
3	Laboratory studies on biocidal activity.	12.99	100	6.99	
4.1	Planning for field work.	12.99	20		
4.2	Field work for stickies detackification.	9.00			
4.3	Field work for biocidal activity.	12.00			
5	Economic analysis.	3.01			
6	Final report.	3.01			

## Replacing Chemicals in Recycle Mills with Mechanical Alternatives

(DE-FC36-99GO10381)

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### *Accomplishments*

- Sparking improves the screenability of PSA stickies at pulp consistencies of 0-2%.
- Sparking is able to detackify pitch coated on metal surfaces.
- Sonication or sparking oxidizes PSA and Robond.
- A sparker unit was purchased and installed at IPST.

### *Screenability of stickies*

In the preceding report we showed that sparking improved the screenability of free PSA stickies, i.e. in the absence of pulp. We have now repeated the measurements in the presence of bleached softwood kraft pulp. Cured stickie (Carbotac PSA) was mechanically homogenized in acetone. Water was added to the suspension, which was allowed to equilibrate, and most of the solvent was removed. More water was added and the process was repeated several times to wash out the acetone. Mixtures of pulp (0.1, 1.0, and 2.0 % consistency) and stickie (0.1% by volume of water) were sparked 50 times in a 2.5-gallon tank, and the rejection percentage determined with a Pulmac analyzer fitted with a 0.006" (150  $\mu$ ) screen. Prior to sparking the stickie suspension was filtered through a black filter paper and image-analyzed (with an ImagePro system) to determine the size distribution. A total of 1,300 particles were counted; the results are provided in Table 1.

Table 1: Size distribution of stickies	
size range ( $\mu$ )	percent
0-50	24.3
50-70	13.2
70-80	6.83
80-90	4.86
90-100	4.94
100-200	25.7
200-300	7.37
300-400	3.87
400-500	2.81
500-1,000	6.07

<b>Table 2: Screening efficiency of stickies in the presence of pulp</b>			
<b>pulp consistency (%)</b>	<b>percent screen rejects before sparking</b>	<b>percent screen rejects after sparking</b>	<b>percent reduction in accepts stickies</b>
0	50.6	53.1	5.1
0.1	59.2	61.4	5.4
1.0	59.4	59.6	0.5
2.0	71.7	73.0	4.6

<b>Table 3: Effect of sparking on contact angle and pitch detachment</b>		
<b>no. of sparks</b>	<b>contact angle</b>	<b>percent of pitch detached</b>
0	73.3	0
10	70.6	0
20	69.5	10
50	69.2	20
100	70.6	30

The results of the screening experiments are provided in Table 2. The final column is the percentage reduction of stickies in the accepts stream after sparking. The initial screen rejects increase with consistency owing to stickie-fiber agglomeration. Although the rejects increase after sparking, the improvement is *nominally* quite small. However, the actual improvement is much better as per the following analysis. It can be seen from Table 1 that half the stickies are less than 150  $\mu$  in size. The screening efficiency in the absence of pulp is also 50%, which indicates that stickies of < 150  $\mu$  in size pass through. Now, the largest stickies will be rejected, regardless of whether or not they are sparked. Unfortunately this fraction is not known. However, if we estimate that stickies of size greater or equal to 350  $\mu$  (twice the slot width) are always rejected, then only one-third of the stickies are potentially treatable. If so, then the final column in Table 2 should be multiplied by a factor of three.

### ***Effect of sparking on pitch***

A sample of pitch was obtained from Georgia-Pacific's, Vienna, GA, particleboard mill as a deposit taken just after the dryer. The material was ground, heated, and then coated on 2 x 2 inch metal coupons. The coupons were sparked at room temperature up to 100 times at 5,000 V (50,000A) at a 1-inch distance from the spark gap. Since the pitch was not tacky at room temperature, changes in the surface properties were measured through contact angle at room temperature, and tack measurements at higher temperature. The contact angle results are presented in Table 3. The effect is marginal, probably because surface wettability is relatively unchanged. Some of the pitch detached from the coupon during sparking, and an estimate of the amount removed is included.

Tack measurements with a Polyken tack tester were made after the coupon was immersed in boiling water for 10 seconds to make it tacky. Readings were taken as the coupon cooled, with the surface temperature being monitored with a thermocouple. The results, listed in Table 4 and illustrated in Figure 1, demonstrate a substantial reduction in tack at the higher temperatures.

Table 4: Effect of sparking on tack					
temperature (°C)	tack (grams)				
	0 sparks	10 sparks	20 sparks	50 sparks	100 sparks
35	38	24	20	23	22
36.3	44	42	30	35	25
36.8	78	89	34	35	41
37.5	93	90	53	42	54
38.4	128	119	94	44	81
39.5	209	120	124	50	108
40	280	212	176	67	108
41	384	300	235	86	110
43	430	354	301	119	159
44	516	603	499	144	190
46	637	660	680	238	200
51	642	750	808	353	286
57	660	751	936	445	458
65	1315	1315	1070	1062	802

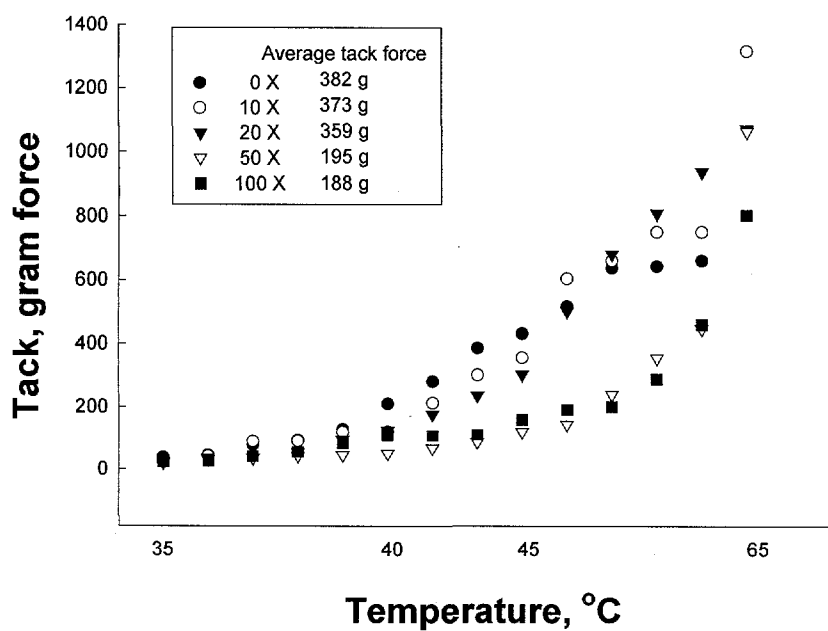


Figure 1: Effect of sparking on tack.

<b>Table 5: Effect of sparking on brightness</b>		
<b>sparks</b>	<b>Robond 90</b>	<b>Carbotac</b>
0	61.55	16.13
10	61.23	22.23
30	56.22	29.67
50	60.06	37.66
100	63.19	38.85
150	60.47	41.24

### ***Generation of aldehyde groups during sparking and sonication***

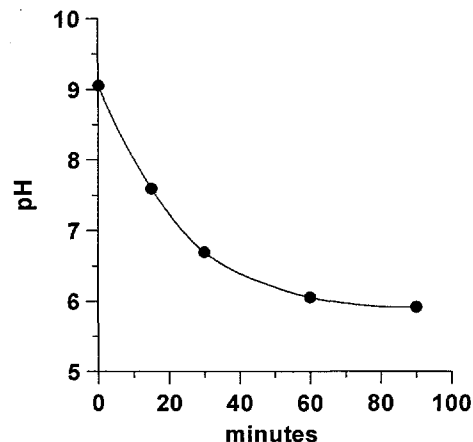
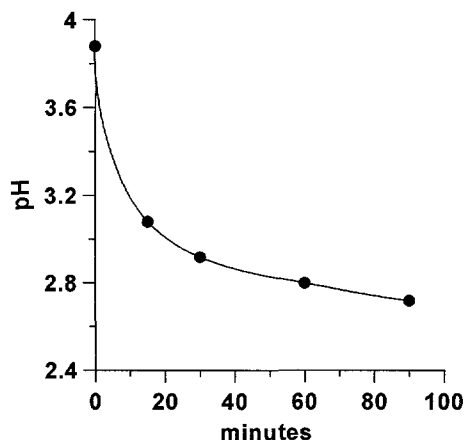
It was felt that the detackification of the polymer surface was caused by oxidation, and we treated 1 mL of a PSA emulsion (Carbotac) with Schiff's reagent (0.1 mL) before and after ultrasonic treatment. This reagent turns purple in the presence of aldehydes. The initial emulsion turned purple indicating that aldehydes were present in the original stickie. The emulsion was coated on metal coupons, dried and sparked 0, 40, and 60 times at 5,000 V, after which a drop of Schiff's reagent was added to the surface. No color change occurred over 2 hours, probably because the reagent was unable to diffuse through the stickie surface. However, purple spots appeared on the surface after 24 hours.

The emulsion was then coated on 3 x 1 inch strips of bleached softwood pulp at a level of 0.3 g/strip and dried at 100°C for 12 hours. Some of the emulsion penetrated into the body of the paper. The strips were sonicated for 2 hours at 22°C, and re-dried at 100°C for 12 hours. They were then treated with Schiff's reagent for 30 minutes. While the initial strips contained aldehydes, the more highly sonicated strips did not, suggesting that the aldehydes were oxidized to carboxylic acids. Since the Carbotac emulsion penetrated into the matrix of the pulp, these results apply to not just the surface, but also to the body of the pulp, confirming that the presence of pulp does not significantly affect the process.

Sparking led to similar results. Carbotac and Robond emulsions were coated on 3 x 1 inch strips of bleached softwood pulp at a level of 0.3 g/strip, and dried at 100°C for 12 hours. The strips were sparked up to 150 times at 5,000 V, and dried at 100°C for 12 hours. They were then treated with Schiff's reagent for 30 minutes. Results from brightness measurements (TAPPI Test Method T-452) are provided in Table 5. The Robond samples remained white throughout; i.e. no aldehydes were present under any conditions. Aldehydes were initially present in Carbotac as indicated by the purple color, but the color faded as the samples were sparked.

In order to confirm that sonication led to oxidation, the pH of a suspension of Carbotac and Robond 90 was monitored during sonication. The results, presented in Figure 2 demonstrate the formation of acidic species. These results demonstrate that the aldehydes initially present in Carbotac are removed through sonication, probably through oxidation. The pH of the Robond decreased, also suggesting oxidation to an acid. Hence, both sparking and sonication appear to induce oxidation of the polymer surfaces.





**Figure 2: pH changes during sonication of 1% Carbotac (left) and 1% Robond (right) during sonication.**

### ***Installation of a sparker unit at IPST***

The work reported to date was conducted at the facilities of Sparktec Environmental, Stoney Creek, Canada. In order to accelerate the effort, a unit was purchased and installed at IPST, and significant effort was devoted to the install. We expect the work to proceed at a much more rapid pace from now on.

### ***Publications and technology transfer***

A paper entitled *Control of Stickies through Electrotechnology*, by H. Corcoran, D-J. Sung, and S. Banerjee, was presented by Howard Corcoran at the 1st CTP-PTS Packaging, Paper and Board Symposium, Grenoble, France, 1999.

An agreement has been signed with Sparktec Environmental, Stoney Creek, Ontario, Canada for commercializing the sparker application for stickies.