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# AN ANALYSIS OF THE WASH AND FLOATATION DEINKING PROCESSES

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A Thesis submitted in partial fullfillment of the course requirements for The Bachelor of Science Degree

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#### ABSTRACT

The purpose of this paper is to obtain data, so that we may compare the ability of Floatation vs Wash deinking to produce a paper of equal standards as the original unprinted basestock.

It was determined that both floatation and wash deinked stock with the addition of 40% fresh pulp can produce a paper of equal physical and optical properties as the unprinted stock from which it was made.

Floatation deinked stock was found have a higher materials cost, however, it displayed better strength and opacity properties than wash deinked stock. The difference is thought to result from a large fines loss in the wash deinking process.

The need and consumption of paper is steadily increasing and the supply of raw materials and energy may be in question. This rise in demand has placed a premium on the price of virgin pulp. As a result, there is an increasing interest in the recycling of paper and specifically the deinking of the finer white printed papers. Estimated costs of construction for integrated mills are approximately \$200,000 - 250,000 per daily ton. A 1000 tpd mill would then require an investment of 1/4billion dollars, a huge investment considerable for only the larger pulp and paper corporations. These high investment costs are also to be considered in the future availability of virgin pulps. A recycling mill does not require the extra equipment such as digesters, evaporators for accumulation of spent liquors, recovery boilers, and kilns to reconstitute pulping chemicals. Therefore, the lower capital costs for recycling and deinking mills are more attractive to many paper manufacturers.

Utilization of secondary fibers is not new. Their use in the production of paperboard and newsprint has a long history. However, virgin bleached chemical pulps are the dominant material for fine printing papers. It is this type of paper and its reuse is where my study will be centered. Optimum recycling of these papers calls for a product of equal quality to permit the manufacture of identical or commercially equivalent paper grades. (1) The resulting deinked stock is then used as a substitute for virgin pulp in the paper's remanufacture.

#### DEINKING OVERVIEW

Deinking comprises of two specific operations:

- 1) Removing ink particles from the repulped fibers.
- 2) Eliminating the detached ink particles from the pulp slurry.

Most common deinking methods begin with the same basic operation of repulping the waste paper in water and adding chemicals at elevated temperatures.

The objectives of this first operation are the breaking of the hydrogen bonds holding the fibrous structure together and detaching the ink particles from the fiber. This is a Mechanico-chemical process. Printing ink consists of carbon or pigment particles dispersed in a solvent or oil binder. The binder is the force that must be overcome to detach the carbon particles from the fiber. Since water, elevated temperatures and mechanical action alone is often not sufficient to release the pigments, chemicals must be added.

The fundamental deinking chemical is an alkali which saponifies and dissolves the binders to release the ink particles from the fibers. The type of alkali used depends on the wood content of the paper. Wood-free paper can successfully be deinked with caustic soda, however, wood containing papers will produce a yellow tinted pulp when using caustic soda alone. Therefore, sodium peroxide is also added. The active  $0_2$  of the peroxide prevents yellowing and slightly bleaches the fiber. Sodium Silicate is also commonly used, it acts as a dispersant, penetrant, and pH buffer in caustic soda solutions. The repulping operation usually lasts from 20 to 60 minutes. Then to obtain complete ink dispersion, the fibers must be allowed to soak in its chemical medium for about 90 minutes or until desired results are obtained. The resulting pulp called "grey stock" is then passed through sand traps, and screens to remove heavy or large debris. A deflaker then breaks up any large fiber bundles and centrifugal cleaners can then separate smaller contraries by weight or specific gravity variations. The resulting stock is a complex mixture of fibers, fines, ink particles and perhaps fillers.

The second operation of eliminating the ink particles from the suspension can be done in two different processes, either wash or floatation deinking.

#### WASH DEINKING

The wash deinking process is based on the simpler principals of either screening or squeezing the water away from the fiber. Their success depends on how well the ink particles have separated from the fibers and remained dispersed in solution.

Typical thickeners which concentrate stock by a factor of six remove 85% of the water from the stock. Ideally then, in the course of three washings, 99% of the ink in perfect solution should be removed. In practice, however, not all ink particles  $\rho g^{\circ}$ are so finely dispersed that they behave as a solute. Ink particles become trapped among the fibers during dewatering resulting in residual ink and lower brightness pulp. Ink can also redeposit on the fibers after the pulping operation. This is particularly true when using a high-consistency dispersion process. There seems to be a "grinding" effect in which ink particles are ground into adhesive contact with the fibers.

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Optimum results are obtained when dilution between water removal stages is performed with clean water. The recirculation of inky water from subsequent washing stages can lower final brightness of the pulp. Water consumption, however, is minimized in this form of counter-current washing. Fresh water is used for the final dilution, the effluent from the final dewatering is used as dilution water for the second stage and so on.

The need for fresh water in the wash deinking process is  $\rho_{ij}$  of considerable importance. Large amounts of water are used and the effluent contains ink particles, fines, and fillers in very low concentrations. Disposal of this effluent creates a large load on the waste water treatment systems and low concentrations make the contaminants difficult to remove.

Typical yields of the wash deinking process are approximately 70 - 80% by weight. The losses for wash deinking are considerably higher than for the floatation method. This loss is due mainly to fillers and fines which pass through the screens.

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Since most book and magazine papers contain considerable amounts of fillers and coatings, losses are higher for these grades. The clays and mineral fillers, however, are thought to aid in the deinking process. These particles stabilize the colloidal dispersions and help prevent the re-deposit of ink on the fibers. In some deinking processes, bentonite and diatomaceous earth are added to aid in the ink removal process.

The chemical composition of the repulping and ink dispersion before the wash process is varied from one installation to another. However, no additional special chemicals are needed to make this simple process work. Because of this, operating costs can be kept to a minimum as compared to the floatation process, in which special chemicals must be added.

Sidehill screens are the predominant type of wash deinking equipment. This is based primarily on the fine job they do, but also influenced by the relatively low initial cost, low operational expense, and the small amount of maintenance required. The major variables to consider are, wire length and width, mesh size and angle of slope.

Wire sections eight to sixteen feet long are used, the width depends on the capacity required. For most deinked stocks the capacity ranges from 5.0 to 6.25 tons/day/foot of screen width. First section washers usually use a higher mess screen, This requires a longer section to enable proper dewatering. Second and third stages are shorter and use larger mesh wire. Typical first stage screens are of 100 mesh, with 80 - 60 mesh on the following two stages.

The angle of the wire section is somewhat critical. If the slope is too flat, the stock will not continue down the wire after dewatered. If the slope is too steep, the tendency

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is for the stock to move too fast resulting in insufficient time for dewatering. A slope of  $38^{\circ}$  from horizontal is common for most types of stock.

#### FLOATATION DEINKING

The floatation process for deinking pulp has won great acceptance in Europe due to its high yields and low water usage.

The floatation principal was first developed for use in the mining industry for the separation of metallic elements from ore. Pierre Hines was the first to consider floatation for fiber deinking in the mid-1930's.

The floatation deinking process is based on the physical process of selective floatation and the differing wettability of the components to be separated in a complex solid-liquid system.

The suspension of fiber, filler, fines, and ink particles must be separated so that only the ink particles rise to the surface, while the fiber remains in suspension. This is achieved because the ink particles have a poorer wettability than the fibers.

To get a selective process for removing ink particles, a floatation agent must be added to the grey stock usually in the pulper. Floatation agents are long chain molecules containing hydrophobic (water hating) and hydrophilic (water loving) groups at the ends. One end attracts the ink particle, the other end attracts an air bubble and floats to the surface.

The the intake of the floatation cell, turbulence and diffuse flow ensure uniform dispersion of grey stock and air. The froth rising to the surface containing ink particles are skimmed off the surface mechanically.

A typical floatation system consists of six to ten primary cells connected in series. The actual number of cells is determined by the retention time required for desired results. An average retention time in each cell is three minutes for a total of 30 minutes in a ten cell system.

The froth skimmed off of the primary cells is diluted and pumped to the secondary stage for fiber recovery. The recovered fibers are pumped as secondary accepts back to the mixing cell or holding tanks.

The final floatation froth has a consistency of about two per cent, it is dewatered in a screw centrifuge or similar equipment to 30 - 40% solids. Ink disposal then is no problem due to its high consistency and resulting low volume.

The cost of installation of a floatation deinking system is substantially greater than that for a wash process without effluent clarification equipment. However, no extra effluent treatment is needed.

Chemical costs are slightly higher than a wash system. A floatation agent, which is unrecoverable, must be added along with normal deinking chemicals. The amount of this agent added is on the order of about .3% by weight.

Yields from a floatation process are very good, usually about 90 - 95%. Because of the nature of the system and its intent to selectively remove ink particles, most fines and fillers are passed as primary accepts. When considering the beneficial role played by fillers for removing ink, the retention of these fillers in the floatation process might suggest a lower brightness pulp.

There are many variables associated with floatation deinking

other than paper stock, ink type and chemicals. Some of these include temperature of deinking, deinking time, water hardness, filler quantity and pH. On the whole, floatation deinking is much more sensitive to change and conditions than the wash process. These factors must be carefully and continuously monitored.

#### CHARACTERISTICS OF RECYCLED PULPS

In determining the differences between wash and floatation deinked pulp, it is important to know the effects of repulping and caustic treatment might have on the fiber as a basis for further comparisons.

The first major point of consideration is the effect on fiber length and freeness. Previous studies by Horn (3) indicate that at the third recycle or repulping stage, the stock was considerably slower than in previous cycles. Horn found that "the most critical limiting factor to the recycling of wood fiber may be the drainage properties of the pulp furnish on the paper machine regardless of strength considerations."

Repulping in water alone was found to result in the decrease of most strength properties. However, tear strength was actually increased through the second cycle of repulping in water. Horn explained this phenomenon as a result of fiber hornification during drying. This hardening of the fiber, makes it less flexible and of poor bonding potential, creates a situation in which fiber rupture is less likely to occur and fiber pullout will predominate.

Throughout this experiment conducted by Horn, it was apparent that bonding strength (2-tensile) was lost to a greater proportion than fiber strength (zero-span). However, treatment with

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NaOH seemed to restore some of this bonding potential by exposing new bonding area on the fiber.

Opacity was generally found to increase through repeated recycling due to shorter fiber length and accumulation of fines.

#### EXPERIMENTAL PROCEDURE

The purpose of this experimental procedure is to obtain data, so that we may compare the ability of Floatation vs. Wash deinking to produce a paper of equal standards as the original unprinted basestock. Physical and optical properties of these two deinked pulps were statistically compared against the basestock and each other.

Fresh basestock pulp was added to the deinked pulps in varying percentages, attempting to improve their physical and optical properties to the level of the unprinted basestock. The amount of fresh pulp addition required to achieve these properties, is then used as a basis of economic comparison between the processes. Chemical costs are also a consideration in the final evaluation of the two deinking processes.

The paperstock used in this experiment was obtained from Western Michigan University Printing Services. The sample consisted of both printed and unprinted sheets, except for the printing, the paper was identical, as it was selected from the same skid. The black offset printing was of medium coverage on both sides. The paper was made from a bleached chemical pulp. Initial ash tests indicated no coating or mineral fillers.

The unprinted paper (basestock) was repulped in a laboratory Morden Pulper. The hottest tap water ( $\approx 48^{\circ}$ c) available was used without any additional chemicals. The consistency, based on oven dry fibers, was 2.6% during basestock repulping.

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#### BASESTOCK REPULPING

Laboratory Morden Pulper Tap Water ≈48°c Pulping Consistency: 2.6% Pulping Time: 20 Minutes

The resulting pulp was sampled for testing. The Canadian Standard Freeness was found to be 360 ml. However, when corrected to 20<sup>°</sup>c, the final freeness was calculated as 325 mls. Clark Fiber Classification was also performed to later compare with the fiber classification of the pulps after deinking.

Noble and Wood handsheets, of the standard 2.5g weight were made from the repulped basestock, pressed and dried. The "Brecht" Wet Web test was performed at this time. A mold, that resembles a cookie cutter, is placed on the handsheet wire. The sheet is then formed as usual. The mold is removed and the sheet is pressed. A strip, who's edges were formed by the mold, is carefully peeled off the wire and placed on the jaws of the "Brecht" Wet Web tester. The weights are placed on top of the pulp strip to hold it in place during the test. The stopcock is opened and water flows into the suspended pan which serves as the moving force in the test. Test results are recorded in milliliters of water needed to pull apart the wet web.

The handsheets formed from the basestock were tested to obtain the basestock values. The tests performed were:

Mullen
Tensile
Tear
Opacity
Brightness

Printed stock preparation followed two different pulping formulas, one for wash deinking, and one for floatation deinking. The original experimental plan did not call for two separate pulping formulas. However, due to limiting factors, discussed

#### later, the change was made.

#### WASH DEINK STOCK PULPING FORMULA

- 1) Pulper Morden Lab Pulper
- 2) Hottest Possible Tap Water  $\approx 48^{\circ}$ c
- 3) NaOH Addition 2.5% by Weight of Bone Dry Fiber. Sodium Silicate Addition .5% by Weight of B.D. Fiber.
- 4) 20 Minute Pulping Time
- 5) pH 11.3
- 6) Consistency 2.8%
- 7) Pulp Allowed to Soak In Pulper For Two Hours, No Agitation

The pulp was then removed from the pulper and a portion of it was diluted with tap water to about .8% consistency. The stock was poured down the sidehill screen. A tap water hose was used to wash the fibers off of the screen into the catch tray. The stock was rediluted to .8% and again, poured over the screen. A total of three passes was made of the same stock over the screen. Each time the hose washed the remaining fibers down the screen and rediluted the stock. The laboratory sidehill was covered with 80 mesh wire and is about 42° to the horizontal. The resulting pulp was sampled for Canadian Standard Freeness, and Clark Fiber Classification. Handsheets of standard weight were made of this deinked stock. The Brecht Wet Web Test was also performed. The physical and optical tests outlined for the basestock were carried out on all resulting pulps.

#### FLOATATION DEINK STOCK PULPING FORMULA

- 1) Pulper Morden Lab Pulper
- 2) Hottest Possible Tap Water
- 3) NaOH addition 2.0% by weight of B. C. Fiber Sodium Silicate addition 3.0% by weight of B. D. Fiber.
- 4) 20 minutes pulping time
- 5) Consistency 2.8%
- 6) Pulp allowed to soak in pulper for two hours, No agitation.

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The pulp was removed from the pulper and a portion was diluted to .4% consistency. Then, an additional chemical was added. Triton CF-10 manufactured by Rohn and Haas Company, was added at .3% by weight B.D. fiber, to serve as collector, frother, and surfactant. The decreased use of NaOH and increase of Sodium Silicate in this pulping formula was due to previous unsuccessful floatation deinking attempts. The high pH of 11.3 as in the wash deink formula caused excessive foaming in the floatation cell. The proper amount of CF-10 could not be added without over foaming the cell resulting in high fiber losses. Lowering the percent of NaOH and increasing the us of Sodium Silicate which acts like a buffer, the pH was lowered to 9.4 and overfoaming problems did not occur.

The pulp was allowed a 30 minute retention time in the Voith laboratory floatation cell. This is equal to ten, three minute cells in series. After this time, the cell was drained, stock samples taken for Canadian Standard Freeness, and fiber classification. Handsheets were formed of this stock and test in the usual manner.

At this point, the handsheets prepared from, basestock, wash deinking, and floatation deinking were tested. From the results, it was decided to make the first fresh pulp addition of 15% basestock to 85% floatation and wash deinked stocks. The second addition was 25% basestock, 75% deinked, and the third addition was 40% basestock, 60% deinked. Handsheets from these three levels of fresh stock addition, were formed and tested in the same manner as above.

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#### RESULTS

Examination of results from the experimental portion of this report are comparitive in nature. Since the two deinking processes are to be compared against each other and the basestock, tabular and graphical from present clearer representations of the data.

#### TABLE I

Summation of Analysis Of Variance: Base vs Float, Base vs Wash

In the following tables a "yes" indicates a 95% chance of a significant difference between the deinked, Fresh pulp mixture, and the Basestock Value based on the data obtained.

### Deinked Pulp + 25% Fresh Stock

TEST	fi w	WASH		FLOAT
Opacity	×.	No	1.18	Yes
Brightness		Yes		Yes
Mullen	×	Yes	ల	Yes
Tensile		Yes		No
Wet Web	\$) 2)	Yes		No
Tear		Yes		Yes

Deinked Pulp + 40% Fresh Stock

TEST	WASH		FLOAT
Opacity	No	54 2	No
Brightness	Yes		Yes
Mullen	Yes		Yes
Tensile	No		No
Wet Web	No		No
Tear	No	÷.	No

TABLE II

9		MEAN	PROBABILITY	SIGNIFICANT DIFFERENCE
OPACITY	Base* Wash Float	83.2 80.9 84.6	.0000	Yes
BRIGHTNESS	Base* Wash Float	77.1 82.9 70.8	.0000	Yes
TENSILE	Base* Wash Float	6.5 4.4 5.5	.0256	Yes
WET WEB	Base* Wash Float	277.0 212.9 201.1	.1281	No
MULLEN	Base* Wash Float	18.9 10.4 16.1	.0024	Yes
TEAR	Base* Wash Float	19.3 19.47 18.07	.0412	Yes
Deinked Stock	k + 15% Base			
OPACITY	Base* Wash Float	83.2 80.66 85.78	.0000	Yes
BRIGHTNESS	Base* Wash Float	77.1 81.12 72.28	.0000	Yes
TENSILE	Base* Wash Float	6.5 5.08 6.62	.0020	Yes
WET WEB	Base* Wash Float	277.0 251.6 239.0	. 5314	No
MULLEN	Base* Wash Float	18.9 13.6 18.1	.0000	Yes
TEAR	Base* Wash Float	19.3 18.1 18.8	.3272	No

\*BASE STOCK appears for reference only. Not included in probability.

TABLE II (Continued)

(H)		MEAN	PROBABILITY	SIGNIFICANT DIFFERENCE
Deinked Stock + 25% Base				
OPACITY	Base <b>*</b> Wash Float	83.2 80.82 84.50	.0012	Yes
BRIGHTNESS	Base <b>*</b> Wash Float	77.1 83.25 73.18	.0000	Yes
TENSILE	Base <b>*</b> Wash Float	6.5 5.0 6.125	.0388	Yes
WET WEB	Base <b>*</b> Wash Float	277.0 293.5 257.5	.1950	No
MULLEN	Base* Wash Float	18.9 13.75 17.05	.0000	Yes
TEAR	Base <b>*</b> Wash Float	19.3 18.75 19.13	.7080	No
Deinked Stoo	ck + 40% Base			
OPACITY	Base <b>*</b> Wash Float	83.2 80.6 83.9	.0002	Yes
BRIGHTNESS	Base* Wash Float	77.1 83.38 74.90	.0000	Yes
TENSILE	Base* Wash Float	6.5 5.24 6.5	.0034	Yes
WET WEB	Base <b>*</b> Wash Float	277.0 293.8 282.5	.1138	No
MULLEN	Base* Wash Float	18.9 14.75 16.50	.0003	Yes
TEAR	Base* Wash Float	19.3 19.13 18.88	.6540	No

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\* BASE STOCK appears for reference only. Not included in probability.

Data:		
WASH		FLOATATION
360		325
78%		86%
\$70/ton		\$70/ton
\$415/ton		\$41 <i>5/</i> ton
\$89.74/ton		\$81.39/ton
\$166		\$166
\$53.84/ton		\$48.83/ton
\$219.84/ton		\$214.83/ton
\$ .80/ton	@2,0%	\$5.52/ton
\$ .80/ton	@3.0%	\$4.82/ton
	e.3%	\$6.71/ton
\$7.70/ton \$219.84/ton		\$17.05/ton \$214.83/ton
\$227.54		\$231.88
	Data: <u>WASH</u> 360 78% \$70/ton \$415/ton \$415/ton \$89.74/ton \$166 \$53.84/ton \$219.84/ton \$ .80/ton \$ .80/ton \$ .80/ton \$ .80/ton \$ .80/ton \$ .80/ton \$ .80/ton \$ .80/ton \$ .80/ton	Data: <u>WASH</u> 360 78% \$70/ton \$415/ton \$415/ton \$89.74/ton \$166 \$53.84/ton \$219.84/ton \$ .80/ton @2.0% \$ .80/ton @3.0% @.3% \$7.70/ton \$219.84/ton \$ .3%

\*Walden's Fiber and Board Report, November, 27,1979

GRAPH I

#### FIBER CLASSIFICATIONS - CLARK

X









**Opacity Points** 



Brightness



GRAPH IV

Tensile





Wet Web



## GRAPH VI

Mullen



MATTN

5



Tear



#### DISCUSSION OF RESULTS

Table I presents the results of the analysis of variance between the basestock values and the deinked pulps with the addition of fresh pulp. The analysis of variance was computed using Western Michigan University's "STATPAK" program. All data observations were entered for each test (i.e. opacity, brightness, etc.) The decision of significance is based on  $\propto = .05$ . This means that a "yes" response assures us of at least a 95% chance that a significant difference exists between the basestock value and the value of the stock compared to it.

The first table compares the basestock values of opacity, brightness, mullen, tensile, wet web and tear to the test values obtained for a combination of 75% deinked stock and 25% fresh pulp. The proportion of "yes" responses to "no" responses indicates that the deinked pulps plus 25% fresh stock are significantly different than basestock values.

The second table compares basestock test values to deinked pulp plus 40% fresh pulp. Here, the proportion of values that are not significantly different would indicate that the pulps appear statistically the same. Except for brightness and mullen, we can assume that floatation and wash deinked stocks plus 40% fresh pulp are equivalent to the basestock.

The wash plus 40% fresh pulp brightness value appears higher than the basestock value. This is due to color difference. Brightness meters will respond with higher values at the blue end of the spectrum than the yellow end. The wash deinked stock retained a blueish tint which accounts for the higher values. Because of this, the brightness value for wash deinked stock has been discounted.

TABLE #2 compares Wash to Floatation deinked stock at 0, 15, 25 and 40 per cent fresh pulp additions. The significant difference responses for each test remain fairly consistent throughout the fresh stock addition ranges. Significant differences between wash and floatation occur for the opacity, brightness, tensile, and mullen test values. This indicates that differences between wash and floatation do exist, even at the 40% fresh pulp addition level, where table 1 showed that both pulps were equal to the basestock values.

TABLE #3 is an analysis of the pulp materials cost for floatation and wash deinking. These figures are based on a 60% deinked stock and 40% fresh virgin pulp combination. Note that 1200 pounds of deinked stock and 800 pounds of virgin pulp is required to produce a ton of pulp. Chemical costs for a ton of finished product is only calculated for 1200 lbs., the amount of deinked stock needed in the furnish. Results of this table show a slightly higher cost for the floatation deinked pulp.

Graph #1 is a bar graph comparing fiber length of basestock, wash deink, and floatation deinked stocks. The fiber classification was performed in accordance to TAPPI standard T-233. The percentage values are based on weight of fibers in each classification out of a five gram sample. The most outstanding result is the low percent of fines in the wash deinked sample as compared to the basestock and floatation pulps. This indicates a loss of fines through the sidehill screen. The floatation and basestock pulps appear very similar in fiber length proportions.

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The remaining graphs are included to convey the idea of floatation and deinked stocks approaching basestock values. At times, it may appear that deinked pulp values greatly exceed basestock values. However, the analysis of variance of the mean values for the test observations, more accurately describe the difference between the acutal mean values.

#### CONCLUSIONS

A 40% addition of fresh pulp to wash and floatation deinked pulps was sufficient to produce a sheet of similar characteristics as one made from unprinted recycled paper. The only significant difference between the deinked and unprinted sheets was a slight loss of burst strength.

However, a significant difference in strength and opacity characteristics was found when comparing floatation to wash deinked pulps. Mullen, tensile and opacity were higher for floatation deinked pulps. This is due to the fines loss in the wash deinking process.

The cost of floatation deinking was found to be slightly higher than wash deinking. However, the yield of 86% for the floatation process in this procedure is lower than yields generally found in industrial practice. The use of a secondary floatation cell to recapture lost fiber may have increased the yield, thus reducing the cost.

It should be noted that the floatation process is very sensitive to upset. Variations in chemical addition, temperature and pH to name a few, greatly affect the performance of the process. The wash deinking process has fewer process limiting factors.

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