12-1976

Comparative Study of Flotation and Sidehill Washing Stages in Deinking Coated Magazine Stock

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COMPARATIVE STUDY OF FLOTATION AND SIDEHILL WASHING STAGES IN DEINKING COATED MAGAZINE STOCK

By

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

Western Michigan University
Kalamazoo, Michigan
December, 1976

ABSTRACT

This is a study comparing the effectiveness of removing ink particles, consumption of water, and the retention of coating and/or filler pigments in deinked pulp between the flotation washing method and the counter-current sidehill washing method. Coated magazine waste paper was deinked using 5.3% Sodium Hydroxide at 190°F. The washing stage of both methods was carried out at a .9% consistency and a temperature of 30°C. The results of the study indicate that the flotation method of washing deinked pulp removed the majority of ink particles but did not remove the coating and filler pigments. The sidehill on the other hand, removed most of the coating and filler pigments while retaining more ink particles than the flotation method. The flotation method consumed less water than did the counter-current washing method.

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INTRODUCTION

The objective of this study is to compare the retention of coating pigment and mineral fillers of coated magazine stock in the washing stage of a deinking operation using the flotation and sidehill washing systems. This will be accomplished by ashing the waste paper before deinking and then comparing these results to each of the ashed deinked pulps and handsheets made after the flotation and sidehill washings.

Comparison of the two washing systems as to their ability to remove ink from the pulp is also of concern. The results of this comparison will be reported as G.E. brightness of handsheets made from the deinked pulp.

Evaluation of pulp yield will also be studied. This will be accomplished by collecting the pulp after each type of washing stage, filtration in a buchner funnel, followed by oven drying of the pulp, and finally ashing to obtain the ash-free yield.

A comparison will also be conducted, as to the amount of water consumed by each washing system. This will be reported as liters of water consumed per ton of deinked pulp obtained from each system. Examination of the effluent obtained from each system will also be made as to its concentration of pigments and ink particles.

LITERATURE REVIEW

Historical Background

Deinking of waste paper is no new process. It has been around for many years. The first recorded attempt to repulp a printed sheet of paper occurred in 1695, at the mill owned by George Balthasan Illy of Denmark (20). The first patent was issued to Matthias Koops of Great Britain on April 28, 1800, for his process of deinking old printed papers (20). Since all paper made previous to this time was hand made, there was little need to reuse old papers. After the 1850's machine made paper was increasing in popularity. By now, however, wood was beginning to be used as a base for paper, so attention was drawn away from deinking and was focused on newer methods of pulping wood, and also on improvements upon the newly developed fourdrinier papermaking machine.

Deinking in the United States became of major importance when the pulping mills began to shut down. The major papermaking areas in the mid-west (Kalamazoo) was able to import their pulps from Canada and also Europe via the Great Lakes water system. When this became too expensive, the paper industry began supplementing virgin pulp, with fibers obtained through deinking old printed papers. These deinked fibers however were (at first) restricted to rag or chemical pulps. Groundwood or mechanical pulps of any kind were not allowed into the deinking system, because of difficulty encountered when deinked. This meant that sorting of papers used for recycling must be done, thus increasing the cost of the operation.

The economics of the deinking operation have played a dramatic role in determining the use of virgin pulp or deinked fibers. When the cost of virgin pulp increased, the amount of deinking increased; conversely, when the price of virgin pulp decreased, the amount of deinking also declined. This trend is clearly obvious when a mill located on the Mississippi River in 1922 experienced a lowering of the water line to such an extent, that groundwood pulp produced up the river could not be brought down to the paper mill. Instead of closing the mill, deinking was done on old papers, using bentonite. The mill was able to supplement the deinked fiber for the groundwood pulp until the river rose at which time groundwood was available again at a lower cost than the deinking operation could be run.

In 1931, at the Fox River Valley of Wisconsin, a deinking system was devised for reusing discarded telephone directories made primarily from groundwood pulp. This was the first time that groundwood printed papers were deinked in a commercial process. This process was applicable only for directories printed with ink which contained a pigment made from the iron lake of logwood. This pigment could be solubilized by treating the books in a solution of sulfur dioxide dissolved in water, and then washing the pulp in diffuse washers. The process required fifty pounds of sulfur per ton of pulp treated (20). A small amount of bleaching could also be accomplished at the same time. The process was very successful, but as World War II broke out, the availability of the logwood pigment became scarce.

The next improvements to the deinking process were initiated to improve the brightness of the deinked pulp. Bleaching of the deinked pulp began in the late 1930's and continued through the 40's. The first chemical used for bleaching was sodium peroxide, buffered with the proper amount of magnesium sulfate. Other chemicals used in bleaching deinked stock are hydrogen peroxide and sodium and calcium hypochlorite.

The next major development in deinking did not occur until 1961. In this year, the first 100% recycled paper newsprint producing mill went into operation at Garfield New Jersey. This mill, the Garden State Paper Company, even today does not disclose publicly the chemicals used in their process.

A fairly recent development in the deinking area is a system designed to separate the ink from the waste water after deinking has occurred. The process is called flotation separation and will be discussed later in this paper.

Statistics

The United States, although ranking among the highest producers and consumers of paper in the world, ranks near the bottom in comparison with other countries in the deinking and recycling of usable discarded printed papers. In the 1970 production year, the United States recycled 21% of its total production (17) while West Germany recycled 31% (21), Japan 38% (21), and Sweden 25% (9).

Let us take a closer look at the amount and uses of paper produced in 1970. The total production in that year was 57 million tons of paper, with only 12 million tons being recycled. An argument may arise that some of that paper could not be used for deinking or recycling. This is a ligitimate argument, but actually only 8 million tons of the 45 million tons not recycled, were used in permanent applications, (hardcover books. or documents)(21). The other 37 million tons of usable paper was buried in the ground or destroyed in other methods. Dividing the paper produced into categories, we see that the corrugated boxboard papers lead recycling with 24% of its total production of 14.5 million tons, while newsprint recycles 23% of its total production of 9.75 million tons, followed by printers trim at 100% of its small 2.2 million tons of total production, and finally the office paper with 4.78% being recycled (21). It is clear from these facts, that the high tonnage categories, newsprint and corrugated boxboard, can and should see increases in their recycling capacities in the future years.

Deinking will become an increasingly important process of obtaining usable fiber in the near future because of its relatively inexpensive source of paper fibers. Also, when looking at the amount of recycled paper used in 1970, it must be understood, that this figure includes not only the deinked paper, but also the recycled paper used in the production of boxboard, which does not require the pulp to be deinked prior to production.

The major reason for the slow acceleration of deinking and recycling in this country is probably due to economics. The major cost involved in deinking is the manual labor of sorting and collecting the reusable materials. Many of the new adhesives, glues, and other binders make deinking impossible because of the difficulty involved in breaking up paper produced from these chemicals.

Another problem associated with sorting, is the increasing amount of paper printed using the offset printing method. The ink involved in this printing process is extremely hard to remove from the paper. This type of printing must be sorted from the reusable paper. Work must also be done in this field to develop inks that can be used in the high speed printing operations but must also have characteristics which will not hinder the deinking processes.

Processes

The deinking systems do follow a rough pattern in as much as the basic steps followed from printed sheet to deinked pulp. The first step in the deinking process, is the defibering of the printed sheet. A large hopper chest is usually used for this purpose. The pulp is dumped off a conveyor belt after it has been sorted and separated. Then deinking chemicals are added to the pulp after it has been sufficiently broken up.

There are a wide range of chemicals used in the deinking process but the most widely used is sodium hydroxide. It is by far the longest used chemical in the history of deinking. It's main purpose in the deinking process is its known ability to swell the paper fibers allowing rapid disintegration of a formed sheet (21). It is also used to maintain an alkaline pH, thus aiding the saponification, breakdown and dispersion of printed inks and their binders. The pH, however must be controlled below 10.5 or decomposition of the paper fibers will occur (6). This will result in an increase in operating cost and a decrease in the yield. The absence of groundwood becomes important because sodium hydroxide or any other hydroxide will attack this mechanical pulp, causing a formation of brown stock, which is difficult to bleach. In spite of these problems, caustic soda is still the predominately used chemical in deinking processes, accounting for 8-50% of the total amount of chemical used.

Another more commonly used chemical is sodium silicate, usually added a rate of 3%. It's function is to promote clean deinked stock and to buffer the reacting solution to a pH of about 9. Sodium or hydrogen peroxides are also added to some processes in order to break down caseins, starches, adhesives or glues, and also to perform a one step bleaching effect on groundwood and chemical pulps.

Organic soaps of different kinds are added in some mills because of claims that better ink removal can be achieved. Other chemicals such as bentonite, diatomateous earth, phosphates, and ammonia have been added by different mills deinking different types of printed paper.

The G A F Corporation has recently been exploring the use of cyclohexylpyrrolidone. This chemical is found to be soluble in cold H_2O and insoluble in medium to warm water. It has claims to help the deinking process of specially difficult paper and can easily be recovered because it is not consumed in the reaction (19).

Recently, a process of bleaching has been incorporated into a mill that will deink the pulp, and bleach it all in one step. The bleaching agent can be either sodium hypochlorite or a combination of sodium hypochlorite and zinc hydrosulfate. This type of operation not only saves time it also can save some waste disposal problems. Instead of deinking, washing, diluting, bleaching and washing again, the new process will only use the washing step one time.

The next aspect of deinking to be discussed are the other variables involved in the process. The pH as mentioned previously must be maintained between 9 and 10.5. This range is optimum because deinking will only take place in an alkaline solution. The reason for this is the average inks used for printing are a varnish linseed oil base. To decompose this, an alkaline solution must be used. The higher limit is needed because if the pH increases to 11 or higher, degradation will occur. The normal yields for deinking in an alkaline solution is 78-86% (6).

The temperature at which the deinking process takes place is not as critical as pH. A higher temperature is favorable because the reaction will occur at a faster rate. On the other hand, higher temperatures require larger amounts of energy, thus raising the cost of the operation. Also, if peroxides are used in the deinking process, they will start to decompose at 50°C and higher (122°F). The reaction temperature varies widely with temperatures any where from room temperature to 160°F depending upon the specific mill and process used.

The concentration of pulp does not demand any set conditions in the deinking process. It ususally will depend upon the type of equipment used and also the type of paper being deinked.

The next step in the deinking process, is removal of the dissolved ink, impurities, and contaminates with the use of screens or cleaners. The centrifugal is the most popular cleaner used for separating the large heavier contaminates from the deinked pulp. Dewatering may be done now to recover some of the chemicals by using skrew type thickeners. The stock can now be screened using pressurized screens. This series of steps seems to be done by the majority of deinking mills.

By now, the majority of impurities have been removed from the pulp, and it can now be washed. One of the most efficient techniques for removing deinking chemicals is by using a series of side hill washers. This washer requires no power to run because it takes advantage of gravity to propel the pulp along its screen. The side hill looks like a large screen made from a non-corrosive metal with wooden supports. The screen is raised at one end to the specified angle of 45° with respect to the horizontal plane. The stock is pumped to the top of the washer and allowed to tumble down to the bottom of the screen, loosing water, chemicals and dispersed ink as it tumbles. The stock is collected at the bottom of the screen, diluted again and pumped to the next washer. The usual amount of screens incorporated into the process will depend upon, space available, amount of production, and degree of washing desired. The water obtained from the pulp is counter flowed in order to reduce the amount of water consumed.

Another type of ink removal system very popular in Europe is the flotation process. The process is based upon the principle that fatty acids in the presence of calcium salts will form soaps (7). When these soaps exist in the deinking pulp, air can be bubbled up from the bottom. The soap will form foam in the pulp and rise slowly to the top of the pulp. As it rises, it will come in contact with the dispersed ink pigments and carry them also to the top. The foam can then be scrapped off of the surface of the pulp thereby also removing the ink pigments from the cleaned deinked pulp. This process has the advantage of using no water dilution as compared with the side hill washers. There is one problem associated with this process however, the chemicals must somehow now be removed. The process removes ink but not some expensive chemicals.

The pulp is now clean, and it can be used directly in papermaking applications, or dewatered and baled ready for shipment, or it can be bleached further to obtain a higher brightness.

Advantages-Disadvantages

Besides the obvious economic advantages of deinked pulp; low cost (depending upon the price of virgin pulp) less pollution and lower cost of deinking equipment when compared with kraft pulping, it seems that some very definite strength and optical properties advantages can be obtained with a deinked pulp. The pulp, having been refined once before, has a lower Canadian Standard Freeness than virgin pulp. During deinking and bleaching, the fibrils and side chains formed by refining are degraded from the fibers by the strong chemicals. If the deinked stock is refined, the resulting fibers will contain fibrils again, and the strength of the pulp will be increased. The burst resistance is actually higher after deinking, then all other virgin pulps except bleached softwood kraft (10). The theory involved here is that since the refibrillization has occurred, the shorter fibers can form stronger bonds because of reduction in unbonded areas.

The real advantage of deinked pulp is the increase in opacity (10). The explanation for this fact is that the deinked pulp contains many fines as well as short fibers. These fibers form a more closely formed sheet with the fiber fines plugging up the holes, thus increasing the opacity value.

The deinked stock is very sensitive to refining. The beater curves show a drastic reduction in freeness as the refining time is increased. An example of the sensitivity deinked stock has toward refining, it has been experienced that if deinked pulp is allowed to agitate for long periods of time, the strength of the pulp starts to drop (10).

Newsprint cannot be economically deinked using sodium hydroxide and then bleached to a high brightness. When a newsprint, or a similar groundwood sheet, is deinked in the presence of caustic soda, the resulting pulp will change to a brown color. Bleaching this stock requires large amounts of chemicals thus raising the cost of the deinking operation.

If sodium hydroxide is used in deinking coated papers containing calcium carbonate, the composition of the adhesives must be known, or the pH of the system may mysteriously drop off. Any adhesive incorporating casein or α-protein, when in the presence of sodium hydroxide will form calcium caseinates and proteinates. The casein and α-protein will hydrolize when heated in an alkaline solution forming ammonia and carbon dioxide. When these compounds react with sodium hydroxide and water, they form sodium bicarbonate which will lower the pH of the deinking solution. This results in an expense because make up sodium hydroxide must be added in order to stabilize the pH.

One of the major problems associated with the deinking process is the disposal of the waste water. Usually the water and the ink pigment along with some chemicals are used in land fill operations. The amount of water shipped can add a considerable cost to the disposing method. Clearly the water must be removed and possibly the ink pigments could be oxidized in a line kiln or other high temperature furnaces. This process could in addition to remove the waste, add energy that could be used to heat water or other substances. The flotation system of ink removal can reduce this problem considerably.

EXPERIMENTAL SECTION

Deinking Stage Design

The type of stock chosen for this study is Time Magazine because of its relative abundance and also because it is printed on coated base paper. Preliminary ashing of this stock revealed an ash content of 23.23% with a moisture content of 4.25%. The stock was then cut into 1-2 inch square samples and stored in a polyethylene bag.

The variables in this deinking stage were chosen at approximately the average conditions used in the paper industry. The consistency of the cook was 2.3% based on bone dry and ash free fiber weight. A temperature of 190° F was achieved by the direct injection of live steam into the stock slurry. Sodium Hydroxide was used as the deinking chemical, at a concentration of 5.3% (based again upon B.D. and a free fiber wt.). A suitable dispersing agent was added at a 1% concentration (B.D. and A-F fiber wt.). The total cooking time from when the stock is mixed in with the deinking solution until dilution occurs is approximately 27-30 minutes. The time is proportioned as follows: 1-2 minutes are allowed for the stock samples to disintegrate; the stock slurry is heated to 190°F over a five minute period; reaction of the caustic with the stock at 190°F with increased mixing is done over another five-minute period; and finally cooling of the stock is done in a water bath for 15 minutes. The pulp is now diluted to the desired consistency and ready for the washing stage.

The use of a one-gallon size laboratory Waring blender with a transformer connected to it in series is used in this stage. The transformer acts as a resistor, in that it will only allow a certain percent of full current to pass through to the blender.

Deinking Procedure

The procedure starts by pouring 2.8 liters (allow 200 ml of water to condense from the steam to get a total of 3 l) of hot tap water into the blender. Next. 3.75g of NaOH (5.3%) pellets are added to the water, and allowed to dissolve with the blender selector on low, and the transformer allowing only 20% of full current to flow to the blender. Once the pellets are dissolved, approximately .73 g of the dispersant is added. The stock samples (95.6/g) are now slowly added to the blender. The required time is allowed for desentigration, while the steam line is made ready. Steam is directly injected into the side of the reaction container while mild mixing is taking place. Once the stock has reached 190° F, the steam line is withdrawn, the top of the blender is clamped into place, and the current is increased to 50% of full current. After the five-minute cooking time has expired, the power is shut off, the top unclamped and removed, and the whole mixing container is placed in a cold water bath for fifteen minutes.

Sidehill Washing Stage Design.

The variables affecting the sidehill washer are very similar to those that affect the flotation cell. Consistency is the major variable which dictates the ultimate cleanliness of the pulp. That is, the lower the consistency of the pulp, the cleaner the washed pulp will be. however, the economics of the amount of water consumed, as well as the cost of concentrating the effluent are also important. Therefore a consistency of approximately .9% (based upon O.D. and A.F. fiber wt.) is about the average value used in the paper industry, and so it is also used in this study.

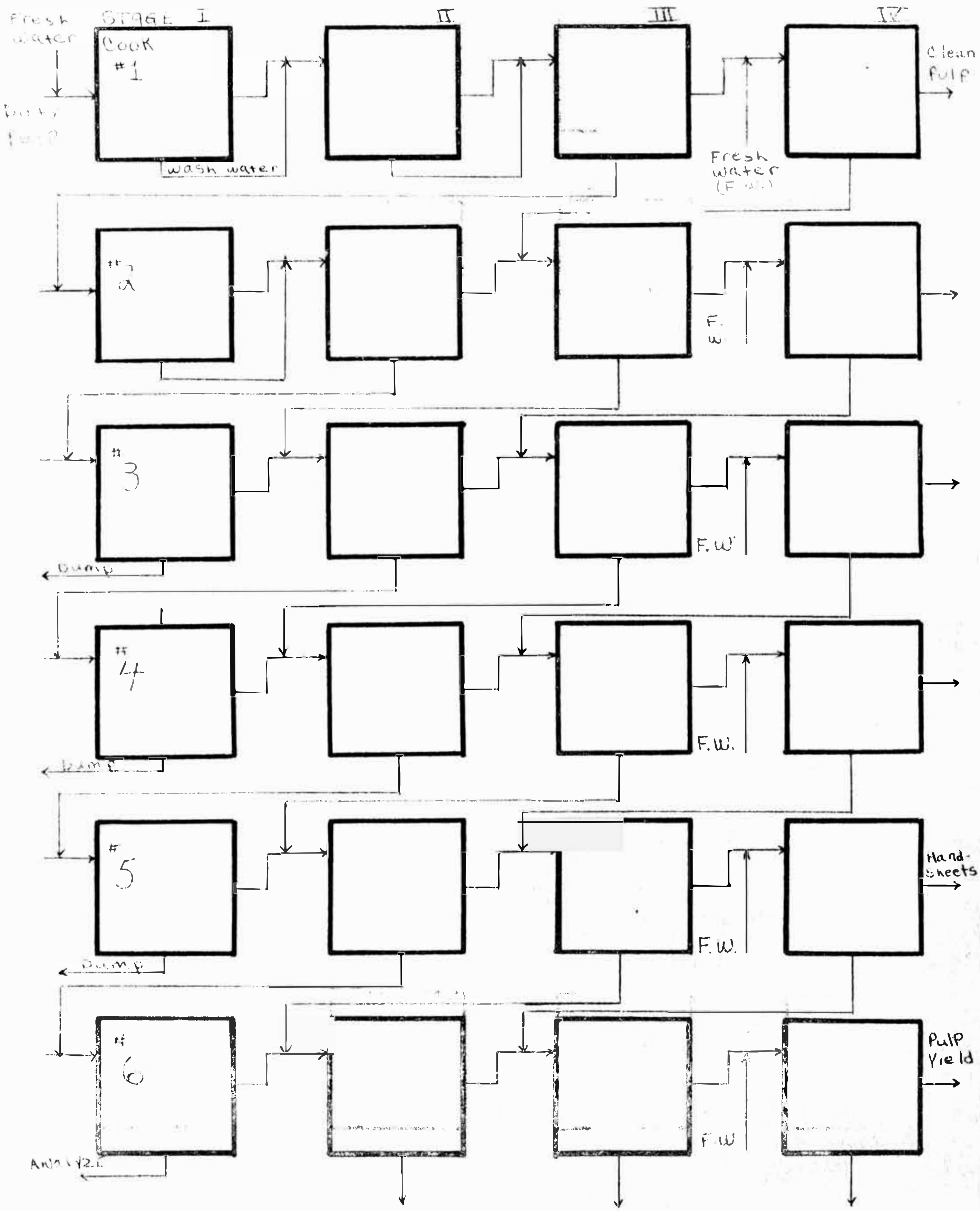
The temperature is a secondary variable which can effect the results of the deinking process. A temperature of about 30°C seems to be a good value, as this gives the best results in the flotation cell. The sidehill washer is not greatly effected by temperature, so 30°C is also used in that washing system.

The sidehill incorporated in this study, is a small laboratory washer specially made for Western Michigan University by the Kalamazoo Tank and Silo Company. Reference to a thesis submitted in 1958 is made here in regards to the actual dimensions of the sidehill (4).

Sidehill Washing Procedure

A four stage washing system was employed in this study using counter-current flow of the wash water. Since only one washer was available, four preliminary deinking cooks were required to set up the counter-current wash waters (see figure 1). The fifth and sixth cooks were then used for analyzation. The pulp collected from the fourth stage in the fifth cook was filtered and oven dried to obtain a moisture free weight. A small portion of the pulp was used to determine an ash content. Knowing these two values, a pulp yield was determined for the process. Handsheets were made from the clean pulp from the sixth cook. The water collected from the first

Figure 1


Fresh water
STAGE I
II
III
IV
Cook #1
Clean Pulp
wash water
Fresh water (F.W.)
#2
F. W.
#3
F.W.
Dump
#4
F.W.
Dump
#5
Hand-sheets
F.W.
Dump
#6
Pulp Yield
F.W.
Analyze

stage of the sixth cook was analyzed, as this is the effluent from the process. The volume of water collected was measured, as well as the ash and fiber content.

Flotation Washing Stage Design

The only procedural difference encountered in the flotation washing system was the fact that two cooks were needed to obtain a large enough sample slurry to use the flotation cell. The first cook was made according to the original deinking procedure stated previously. The second cook also followed the specified variables. However, the amount of stock sample was increased from 95.61 g to 100.2 g. The amount of chemicals and of water were also increased, but in proportion to the increase in fiber content. Therefore, all of the conditions and variables mentioned previously still pertain.

The Voith laboratory flotation cell was used in this study. The size of the model is described as being a 15 L capacity flotation cell.

Flotation Washing Procedure

A suitable frothing agent was added to the pulp slurry at a concentration of 1% (based upon 144 g, O.D. and A.F. weight). The concentration of the pulp was held at .9% (O.D. and A.F. fiber wt.) and the temperature at the time of flotation was 30°C. The use of an external air supply was found to be of no added value in this study. However, the valve which allows the external air hose to be connected to the flotation cell was left open. Air was observed to enter the cell through this valve and produce a suitable froth. The recirculation valve was adjusted so that enough pulp would recirculate to minimize the amount of air entering the cell at this point.

The flotation process was allowed to proceed for 15 minutes. During this time, the froth was skimmed off the top of the pulp slurry with a plastic spatula, and collected in a container. Next the pulp was dewatered in a 100 mesh screen, followed by a 150 mesh screen. The water obtained in this step was analyzed to determine its fiber and ash content. The froth collected during the process was also analyzed for its ash and fiber content. No secondary flotation of the froth was done in this study.

The pulp obtained from the first flotation washing was used to make handsheets. The pulp from a second flotation washing was then oven dried and weighed. A portion of this pulp was analyzed to determine its ash content, which was used to calculate the pulp yield obtained from the flotation washing method.

TABLE 1

	Sidehill Washing	Flotation Ashing
Magazine stock ash	23.2%	23.2%
Pulp ash after deinking	3.8%	11.4%
Handsheet ash	2.0%	5.6%
Brightness of Handsheets (G.E.)	58.3	59.2
Pulp Yield	75.0%	82.0%
Water Consumption l/ton	93,100 l/ton	75,000 l/ton
Water consumed in whole process l/ton	114,500 l/ton	122,500 l/ton
Percent solids in effluent	.6 - .7%	1.4 - 1.5%

DISCUSSION OF DATA

The general data collected in this study is found in Table 1. It can be seen, that although the sidehill wash removed far more solids than did the flotation wash, handsheets produced from the sidehill washed pulp registered a one point lower G. E. brightness than the flotation handsheets. The brightness of the handsheets was measured instead of the pulp. Although, this may not necessarily give an idea of the brightness of the pulp the flotated handsheets did contain more than double the ash content of the sidehill handsheets. The reason for this is not clearly explained by Table 1. A visual inspection of both sets of handsheets with the naked eye revealed that the sidehill handsheets contained small black specks, while no such specks are observed in the flotation handsheets. An assumption is then made, that the solids of the sidehill washed pulp contain a large amount of ink particles while the solids of the flotation washed pulp contain very little ink particles. The very high solids content of the flotated pulp is due to the large amount of coating and filler pigment in the pulp. If this assumption is true, then the coating and filler pigments also are responsible for the low brightness of the flotated handsheets. With removal of these pigments, the brightness of the pulp will increase to a more suitable value. Recovery of the pigments could also become possible through settling or some other method.

The low brightness of the sidehill pulp is due to the large amount of ink particles in addition to some coating and filler pigments. The ability of the sidehill to remove ink particles is assumed to be quite low. The sidehill washer does however remove solids caused by coating and filler pigments.

The pulp yields presented in this study were calculated by performing material balances around the two washing systems. The material balance for the four stage sidehill washing system was performed around the sixth cook (see figure 1). Here, the effluent water from the first stage as well as the pulp from the fourth stage were filtered and ashed. The values obtained from the balance showed a 75% yield of pulp, containing an 11% ash yield.

The balance around the flotation cell was carried out in a similar method. The pulp and froth were both filtered and ashed. The yield value presented is obtained from primary flotation only. Secondary flotation of the froth could not be accomplished because of the small amount of sample. The fiber content in the froth was calculated at approximately 62% of the total froth collected. This could surely be reduced by flotating the froth collected from approximately eight cells. If the 82% yield is again obtained, the fiber recovery would amount to about half of the 62% lost in the froth. The pulp yield for the flotation method could then theoretically be raised to 87-90%. The only increase in cost of operation would be one extra flotation cell for every eight primary cells. The other losses involved in the flotation method were a result of a dewatering stage mentioned previously.

The water consumption figures are based upon a deinked ton of paper collected from each process. This would seem that the only variable effecting the water consumption is the pulp yield. Actually if the yields had come out the same, the water consumption of the sidehill would be lowered by about 12,000 liters. This, however, would still be a higher consumption rate than the flotation washing system at its present yield. The reason for the large discrepency between the two systems is the fact that, the sidehill pulp leaves the last stage at a consistency of about 8-9%. Since the systems were operating at the same dilution consistency, the water usage increased in the sidehill because of this water loss in the pulp.

The amount of effluent is another concern when deinking. The effluent must be concentrated enough, so it can be disposed of economically. The flotation washing system produced the froth at about a 1.4-1.5% consistency. The sidehill washing system on the other hand produced effluent at about .6-.7% consistency. This amounts to about an extra 1000 liters of water which needs to be concentrated using the sidehill deinking method.

CONCLUSIONS

The flotation method of washing coated magazine grade stock after deinking produces a better pulp yield, consumes less fresh water and removes ink particles better than the counter-current sidehill washing system. Also, the effluent produced from the flotation cell, can be disposed of at a lower cost because of its higher solids concentration.

The counter-current sidehill washing system removed more of the coating and filler pigments and consumes less energy than does the flotation method.

RECOMMENDATIONS

The washing system most suitable for deinking coated magazine grade waste paper will depend upon the end use of the deinked pulp. If an increase in coating and filler pigments will not produce any problems in a specific operation, then the flotation washing system is recommended. However, if this increase in pigments cannot be tolerated, then the sidehill washing system can be used.

Further work can be done with the Voith flotation cell. A secondary flotation of the primary froth should be investigaed as to its fiber recovery potential. The possible theoretical pulp yield presented in this study for the flotation method should be proven experimentally.

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