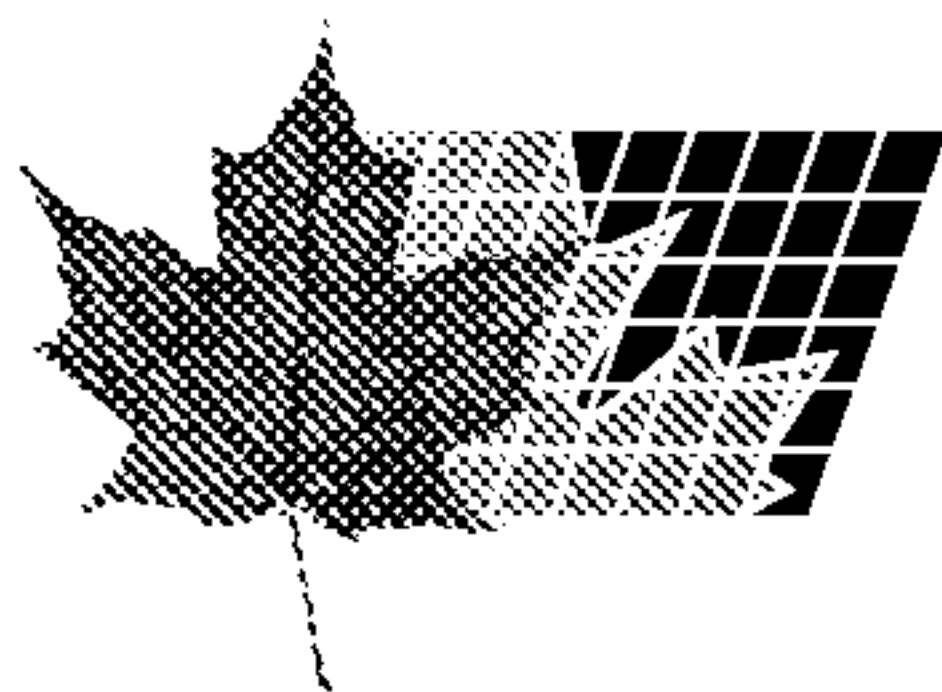




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(54) **PROCEDE BIOLOGIQUE DE DESENCRAGE**

(54) **BIOLOGICAL METHOD OF DEINKING PRINTED
WASTEPAPER**

(57) A biological method of deinking printed wastepaper consisting of the steps of disintegrating of printed wastepaper in hot water containing cellulase or pectase and removing ink particles from fibers by froth flotation or washing.

ABSTRACT OF THE DISCLOSURE

A biological method of deinking printed wastepaper consisting of the steps of disintegrating of printed wastepaper in hot water containing cellulase or pectase and removing ink particles from fibers by froth flotation or washing.

The present invention relates to a method of deinking printed wastepaper.

BACKGROUND OF THE INVENTION

5 Deinking of pulp fibre is essentially a laundering or cleaning process in which ink is considered to be the dirt.

Chemicals along with heat and mechanical energy, are used to dislodge the ink particles from fibers and to
10 disperse them in the aqueous medium. The ink particulars are then separated from the pulp fibers, either by washing or flotation or by using a modern hybrid process that combines the two elements.

15 The chemicals used for the conventional deinking process are surfactants which function as detergents to remove ink from fiber, keep the ink particles dispersed to prevent redispersion on the fibers, and provide a foaming action for the froth flotation of ink particles.

20 A typical surfactant is a long chain molecule with a hydrophobic part to the one end and a hydrophilic part to the other end. The hydrophobic part may be consist of fatty acid, fatty alcohol, alkylphenols or other oil soluble
25 surfactants.

The hydrophilic part of the deinking surfactant usually consists of anion molecules such as carboxyl acid salts or sulfonic acid salts and nonionic molecules such as
30 polyoxyethylenated chains.

The typical surfactants commonly used in the washing and froth flotation deinking process are; sodium and potassium salts of strait chain fatty acid (soap), linear
35 alkylbenzenesulfonate (LAS), olefine sulfonate, long chain

fatty alcohol, polyoxyethylenated alkylphenols, alkylphenoethoxylates, and polyoxyethylenated strait chain alcohols.

5 The Major disadvantage of using these surfactants in the deinking process is excess foaming in the subsequent pulp stock flow and paper making process lines. In addition, some of the above surfactants are resistant to biodegradation in the effluent treatment stages causing a serious environmental
10 problem.

In the froth flotation deinking process, a collector is added to agglomerate ink into large particles and attach them to the air bubbles. Collectors are required for effective
15 flotation and are usually anionic long-chain fatty acid soap. Fatty acid collectors are precipitated with calcium ions to form larger, insoluble ink particles and collector particles. With injection of air in the flotation cells, the agglomerated ink particles adhere to the bubbles, rise to the
20 surface and are skimmed off from the system.

Major disadvantages of the flotation method using the fatty acid collector is a pitch deposition and calcium scaling problems in the subsequent stock lines and paper
25 making equipment. Besides the surfactants, other chemicals are caustic soda, sodium silicate, metal ion chelating agents and hydrogen peroxide.

The hydrogen peroxide bleaching agent has to be added
30 in order to prevent a pulp colour yellowing caused by the additions of caustic soda and to improve brightness of pulp fibers.

With the advance in the modern printing and
35 photocopying technology, conventional deinking with the aid

of surfactants encounters serious problems with the wastepaper printed with the use of heavily coated, highly polymerized, or nonimpact inks, such as ultraviolet, heatset, Xerox, laser and ink jet. These inks usually contain cured
5 polymer resins which bind ink particles so strongly on the fiber surface that it is impossible to dislodge the ink completely during the wastepaper defiberizing (pulping) stage with the conventional deinking chemicals. Excess heat and mechanical energy are also required along with the
10 ineffective conventional chemicals.

In the conventional flotation deinking process for newsprint wastepaper a major technical problem is experienced with fine ink particles embedded in the fibre bundles and
15 between fibrils which are almost impossible to remove from the fibers by a washing and/or flotation process.

SUMMARY OF THE INVENTION

This invention provides a new and much improved method
20 of deinking printed wastepaper. This method is effective in newsprint deinking, as well as the deinking of wood free printed wastepaper such as whiteledger, laser printed, xerographic copypaper and computer printout wastepaper.

25 This invented deinking method is to remove ink particles with the use of biological activity of enzyme on the cellulose fiber surface and a dispersing function of enzyme protein on ink particles.

30 In contrast to the conventional method no alkali and deinking surfactants are required although some surfactants can be used along with the enzyme to enhance the deinking efficiency. In the froth flotation process the fatty acid collectors are not required. Since caustic soda is not used

in the newsprint deinking, hydrogen peroxide bleaching agent is not also required for yellowing prevention.

5 The elimination of the fatty acid collector in this biological deinking process will solve the persistent pitch and scale deposition problem associated with the conventional flotation process using the fatty acid type soap and calcium salts and silicates.

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DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENT

The method will now be described.

15 Printed Wastepaper, such as old newsprint or printed wood free wastepaper, is disintegrated in a conventional pulper (consistency 4-7%) or in a high consistency pulper, 12-15%, at the water temperature ranging from room temperature up to 60 C. The addition level of enzyme is 0.005% to 5.0% based on dry weight of wastepaper, pH of the
20 stock slurry is adjusted in the range of 3.0 to 8.0. As compared to the conventional pulping process using caustic and surfactants in pulping, the process using enzymes can be completed in a relatively short period and ink particles are completely separated from the fiber surface and dispersed
25 well. The dispersed inks are removed out of pulp fibers by conventional washing process equipment such as a vibration screen and a drum washer without the aid of detergent surfactants. The ink particles dispersed with the action of enzyme protein can be also selectively removed out of the
30 diluted pulp slurry with conventional flotation equipments in which air is injected into the pulp to provide bubbles to pick up the particles. No fatty acid collector is required in the case of waste newsprint. A small amount of fatty acid collector may be added to enhance the ink removal efficiency
35 in the case of laser-printed wastepaper.

This biological deinking process is to lower pulping energy to a large extent since the addition of enzyme results in a reduction in pulping time, as compared to the pulping in the absence enzyme, of almost 50% reduction. The observed
5 faster and easier pulping in the presence of enzyme may be attributed to an unique biological activity of enzyme which is effective to debond the fiber bonding and dislodge the inks bonded on the fiber surface as well as within the fibrils. A partial enzymatic hydrolysis of cellulose within
10 micro structure of fiber surface may occur during the pulping stage. This biological activity of enzyme takes out fine ink particles embedded within fiber bundles, fibrils and fines which have been impossible to be take out by conventional deinking chemicals.

15

According to this biological deinking method of old newsprint the addition of hydrogen peroxide to prevent the fiber yellowing is not required, which will result in a substantial reduction of deinking chemical cost as compared
20 to the conventional deinking process using caustic soda, hydrogen peroxide, chelating agent and sodium silicates.

It should be pointed out that the physical strength properties of the resulting pulp fiber prepared by this
25 invented method are found to be higher than those of the corresponding pulp prepared by the conventional method, in addition to the much higher resulting pulp brightness. The enzyme addition does not appear to degrade the fiber strength, instead improving the fiber strength for reasons
30 that are as yet unknown.

Example 1.

Deinking of old newsprint with a cellulolytic enzyme.

10 A sample of old newsprint wastepaper was added to a pulper which was filled with 40 C water at the consistency of 4% and a cellulase was dissolved at the dosage level of 0.1% based on oven dry weight of wastepaper. The wastepaper was soaked for 10 minutes and then disintegrated for 5 minutes. After a complete disintegration of wastepaper, one half of the pulp slurry was diluted to a 1% consistency.

15 The diluted pulp slurry was moved to an air flotation cell and then the dispersed ink particles were removed out of the pulp slurry by skimming off a froth containing ink particles out of the cell while injecting air through a porous plate. The flotation time for the complete removal of the ink froth was one minute.

The other half of the pulp slurry was washed on a laboratory vibration screen to remove the dispersed ink particles.

25 The resulting recycled pulp fibers obtained by the flotation and the washing step were evaluated for pulp brightness and mechanical strength properties. To compare this enzyme treated deinking pulp to the conventional deinking pulp, the same sample of wastepaper was treated in
30 the pulper with the addition of 1.0% NaOH, 0.3% H₂O₂, 3% sodium silicate solution (water glass), 0.8% of SERFAX MT-90 (Trademark) (fatty acid soap) and 0.2% IGEPAL (Trademark) -660 based on oven dry weight of wastepaper. The pulping time was 10 minutes for a complete
35 disintegration. After diluting to 1% consistency,

the dispersed ink particles were removed by the flotation method with the laboratory flotation cell as described above.

As shown in Table 1, the brightness of the pulp deinked with enzyme was much higher than that of the pulp deinked with the conventional chemicals and the mechanical strength of the enzyme-deinked pulp was also superior to that pulp deinked with the fatty acid collector and the dispersant (IGEPAL 660). The microscopic observation revealed that the pulp prepared by the present invention contained more long fiber fractions and has smoother fiber surface and looked less mechanically damaged.

Table 1. Comparison of properties of recycled pulp by method of present invention and the conventional method.

		brightness (%)		tensile index (N.m/g)		tear index (mN.m /g)	
		KONP	AONP	KONP	AONP	KONP	AONP
present method	flotation	47.1	45.2	28.9	32.4	11.7	13.6
	washing	50.3	48.6	29.3	32.9	11.8	14.1
SERFAX MT-90		45.1	38.4	30.1	32.8	10.8	13.1

KONP: Korean old newspaper
AONP: American old newspaper

The enzyme treated pulp gave cleaner and brighter pulp with the washing as compared to the flotation ink removal.

The enzyme addition appeared to accelerate the wastepaper disintegration to a large extent. When the old newspaper was disintegrated in the conventional pulper at the 4% consistency, the addition of 0.5% enzyme reduced the pulping time from 5 minutes (no enzyme addition) to 30 seconds for a complete disintegration as shown in Table 2.

Table 2. Relation between enzyme addition and disintegration time.

Enzyme (%)	0.5	0.1	0
disintegration time (sec)	30>	60-120	300<

Example 2.

Deinking of laser CPO (computer printout) with cellulolytic enzyme.

It is almost impossible achieve a complete removal of laser beam cured ink particles from laser CPO wastepaper with the conventional deinking chemicals, because the ink particles are so strongly adhered to the fiber surface that alkali and general deinking surfactants in the conventional deinking chemicals are not able to dislodge and disperse in the pulp water slurry.

A sample of laser CPO wastepaper was added to water in a laboratory high consistency pulper at the consistency of 12.5% and a cellulase was added to the water at the dosage level of 0.2% based on the dry weight of paper. At stock water temperature of 20-35 C, the pulping was carried out for

20 minutes. The completely disintegrated pulp slurry was diluted to 0.5% and then dispersed ink particles were removed out of the pulp slurry using the laboratory flotation cell as explained in Example 1. In this case, to increase the ink removal efficiency and selectivity a small amount of the conventional fatty acid collector, SERFAX MT-90, of 0.3% based on dry weight of wastepaper was added prior to the air flotation and the flotation time was 3 minutes. To compare to enzyme deinked pulp, the conventional deinked pulp was prepared by the same way but the different chemical conditions as follow:

1% NaOH on dry weight of wastepaper
0.1% IGEPAL 660 dispersant
0.8% SERFAX MT-90
pulping temperature : 50 C
pulping time : 30 minutes
calcium salt addition to the flotation cell: 200 ppm
flotation time : 3 minutes

The brightness and the strength properties of the resulting pulp samples were compared in Table 3.

As shown in the table, the image analysis of the paper samples indicates that the number of the residual ink particles was much less, about 10 times, for the pulp deinked with the enzyme and the tensile strength was also higher as compared to the pulp prepared with the conventional chemicals.

A recycled chemical pulp of high quality in terms of dirt count and fiber strength properties can be obtained with the use of enzyme in a combination of a small amount of fatty acid collector by the flotation method.

Table 3. Comparison of pulp properties recycled by the method of present invention and conventional method.

	brightness (%)	dirt amount (count/area)	tensile index (N.m/g)
5 enzyme:MT-90-(0.3%)	79.0	450	34.3
MT-90 (90%)	80.6	4,330	26.3

10 Example 3.

Deinking of waste newsprint by pectinolytic enzyme.

15 As the same method to example 1, the waste newsprint containing 0.1% of pectase was soaked for 10 minutes at 40C and disintegrated for 5 minutes. Diluting the disintegrated pulp to 1%, the ink particles are removed by flotation for 1 minute.

20 As shown in Table 4, the brightness and tensile strength of paper sheet prepared by the method of the present invention are improved.

Table 4. Comparison the method of using pectinolytic enzyme with conventional method.

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	brightness (%)	tensile index (N.m/g)
30 present method	44.2%	33.3%
MT-90 (0.8%)	38.4%	32.8%

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1. A method of de-inking waste printed paper, comprising
 - a) pulping waste printed paper with an enzyme capable of dislodging ink particles from the waste printed paper in an aqueous medium at a pH resulting from not having added alkali prior to or during the de-inking process, and wherein the ink is dislodged from the waste printed paper by action of said enzyme; and
 - b) removing dislodged ink particles from the resulting pulp containing medium.
2. The method of Claim 1 wherein dislodged ink particles are removed by flotation.
3. The method of Claim 1 wherein dislodged ink particles are removed by washing.
4. The method of Claim 1 wherein the amount of enzyme used is in the range of about 0.005 to about 5 percent-by-weight based on the dry weight of the wastepaper.
5. The method of Claim 1 wherein said enzyme is selected from the class consisting of cellulase, hemicellulase, pectinase, other carbohydrases and mixtures thereof.
6. The method of Claim 1 wherein said enzyme is a cellulase selected from the group consisting of cellulases derived from *Trichoderma viride*, *Aspergillus niger* and mixtures thereof.

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7. The method of Claim 1 including controlling the pH of said aqueous medium from about 3 to about 8.
8. The method of Claim 1 wherein the pulping occurs at a high consistency of pulp.
9. The method of Claim 1 wherein the pulping occurs for a period of less than 1 hour.
10. The method of Claim 1 wherein the paper is pulped at a temperature in the range of about 20 degrees Celsius up to about 60 degrees Celsius.
11. The method of Claim 1 including controlling the pH of said aqueous medium from about 3 to about 7.
12. A method of recycling waste printed paper, comprising
 - a) pulping waste printed paper;
 - b) contacting waste printed paper with an enzyme capable of dislodging ink particles from the waste printed paper in an aqueous medium at a pH resulting from not having added alkali prior to or during the de-inking process, and wherein the ink is dislodged from the waste printed paper by action of said enzyme; and
 - c) removing dislodged ink particles from the resulting pulp containing medium.
13. The method of Claim 12 wherein dislodged ink particles are removed by flotation.

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14. The method of Claim 12 wherein dislodged ink particles are removed by washing.
15. The method of Claim 12 wherein the amount of enzyme used is in the range of about 0.005 to about 5 percent-by-weight based on the dry weight of the wastepaper.
16. The method of Claim 12 wherein said enzyme is selected from the class consisting of cellulase, hemicellulase, pectinase, other carbohydrases and mixtures thereof.
17. The method of Claim 12 wherein said enzyme is a cellulase selected from the group consisting of cellulases derived from *Trichoderma viride*, *Aspergillus niger* and mixtures thereof.
18. The method of Claim 12 including controlling the pH of said aqueous medium from about 3 to about 7.
19. The method of Claim 12 wherein the pulping occurs at a high consistency of pulp.
20. The method of Claim 12 wherein the pulping occurs for a period of less than 1 hour.
21. The method of Claim 12 wherein the paper is pulped at a temperature in the range of about 20 degrees Celsius up to about 60 degrees Celsius.
22. The method of Claim 1 including controlling the pH of said aqueous medium from about 3 to about 7.