

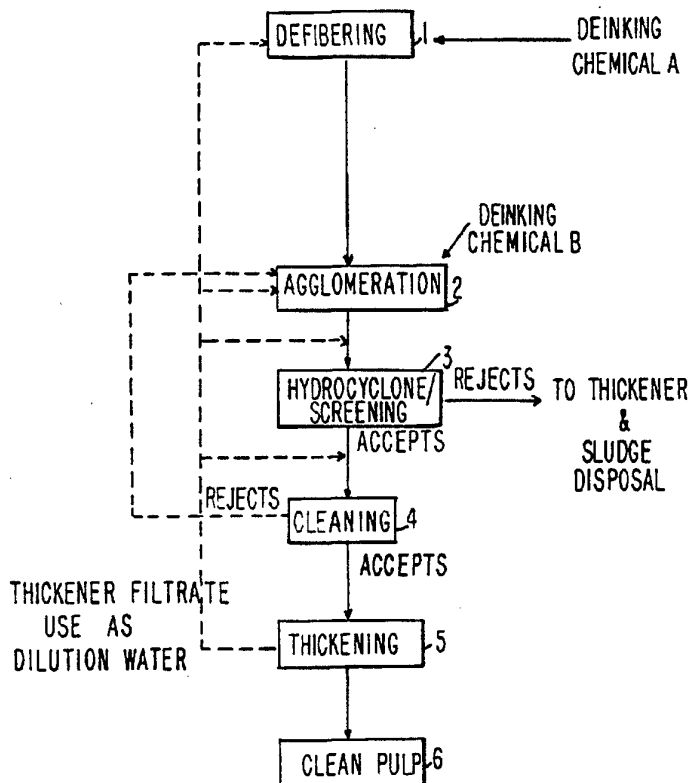


## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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| <b>(21) International Application Number:</b> PCT/US94/08774<br><b>(22) International Filing Date:</b> 2 August 1994 (02.08.94)<br><b>(30) Priority Data:</b><br>08/100,886      2 August 1993 (02.08.93)      US<br><b>(71) Applicant:</b> INTERNATIONAL PAPER COMPANY [US/US];<br>Two Manhattanville Road, Purchase, NY 10577 (US).<br><b>(72) Inventors:</b> SRIVASTA, Narendra, R.; Apartment #71, 450<br>Island Road, Ramsey, NJ 07446 (US). MARKHAM, Larry,<br>D.; 5821 Blue Ridge Drive South, Mobile, AL 36693<br>(US). WESOLOWSKI, Richard, R.; P.O. Box 164, Pulaski<br>Highway, Pine Island, NY 10969 (US).<br><b>(74) Agent:</b> ONOFRIO, Dara, L.; Ostrager, Chong & Flaherty, 300<br>Park Avenue, New York, NY 10022-7499 (US). |           | <b>(81) Designated States:</b> AT, AU, BB, BG, BR, BY, CA, CH, CZ,<br>DE, DK, ES, FI, GB, HU, JP, KP, KR, KZ, LK, LU, LV,<br>MG, MN, MW, NL, NO, NZ, PL, PT, RO, RU, SD, SE,<br>SK, UA, UZ, VN, European patent (AT, BE, CH, DE, DK,<br>ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI<br>patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE,<br>SN, TD, TG).<br><br><b>Published</b><br><i>With international search report.</i> |

**(54) Title:** IMPROVED DEINKING METHOD USING INK AGGLOMERATION**(57) Abstract**

Deinking of printed paper is accomplished by defibering printed wastepaper (1) to produce a pulp medium and contacting the pulp medium with an aqueous medium containing a deinking chemical (2) wherein the presence of the deinking chemical causes agglomeration of ink particles to produce an ink pulp medium. Agglomerated ink particles are removed from the ink pulp medium by size (3) and density (4) separation procedures to produce a substantially ink-free pulp medium. The invention provides improved deinking processes with high ink removal from printed paper by more effective agglomeration of ink particles through separation of defibering and agglomeration actions which are subject to different chemical and physical requirements.



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IMPROVED DEINKING METHOD USING INK AGGLOMERATIONField of Invention

5        This invention generally relates to an improved  
agglomeration process to deink printed paper. More  
particularly, it concerns a process which provides more  
effective agglomeration of ink particles by separating  
the defibering and agglomeration steps which are subject  
10 to different chemical and physical requirements.  
Agglomerated ink particles are removed by size and  
density separation procedures to produce an ink free pulp  
medium used to make recycled paper and board products.

Background Art

15        In the past paper was printed with primarily water  
or oil based inks which were satisfactorily removed by  
conventional deinking procedures. In conventional  
deinking procedures, the paper is mechanically pulped and  
20 contacted with an aqueous medium containing a deinking  
chemical. The pulping and presence of the deinking  
chemical results in a separation of the ink from the pulp  
fibers and the dispersed ink is then separated from the  
pulp fibers by washing or flotation processes.

25        Today, increasing amounts of printed paper are  
generated from electrophotographic processes such as  
xerography and non-impact printing processes such as  
laser and ink-jet printing. Deinking processes capable  
of deinking these types of printed paper are very complex  
30 and are capital intensive. In addition, multiple steps  
are required for debris removal and actual ink removal.  
Generally, ink removal procedures involve washing,  
flotation, forward cleaning and high consistency  
dispersion to reach the level of speck removal and  
35 brightness required in the deinked pulp to produce  
recycled paper.

-2-

U.S. Patent No. 4,561,933 to Wood, discloses a process for deinking xerographically printed wastepaper. Repulped printed wastepaper is treated with a deinking agent consisting of a mixture of alkanols and alcohol ethoxylates to produce a suspension of ink particles. The suspended ink particles are separated from the resulting pulp-medium by washing and flotation process steps. The deinking chemical and process in Wood, however, is limited to deinking only xerographic waste and utilizes multiple process steps to separate the ink.

To surmount these limitations, and as an alternative to conventional deinking procedures, the prior art has shown use of agglomeration deinking processes. Agglomeration chemicals consisting of polymeric or surfactant systems are employed to aid in the ink agglomeration process. In deinking paper through agglomeration processes the waste paper is repulped and then deinked through chemical treatment to provide a slurry of pulp and ink agglomerates. The ink agglomerates are removed from the pulp by sedimentation and separation procedures.

However, the polymeric systems used in these agglomeration processes are specific to certain types of inks. See U.S. Patent No. 4,820,379 to Darlington, in which the deinking chemical used is specific for agglomeration of electrostatic inks, and U.S. Patent No. 4,076,578 to Puddington et al. in which the deinking chemical used is specific for agglomeration of newspaper inks. Alternatively, U.S. Patent Nos. 5,141,598 and 5,200,034 to Richmann et al. discusses surfactant systems including alcohol, glycol and petroleum distillate components, specific for agglomerating electrostatic inks.

These patents disclose agglomeration processes in which the repulping and agglomeration actions are carried

-3-

out in the hydropulper or similar device under the same conditions. There is no distinction made as to the different chemical and physical requirements for the defibering and agglomeration actions. A major constraint  
5 in simultaneous repulping and agglomeration is that the action of the hydropulper breaks the ink into smaller particles at the same time that agglomeration is forming larger particles. By separating the repulping and agglomeration steps agglomeration will proceed without  
10 any particle breakdown thereby producing larger agglomerates which are easier to remove from the pulp.

Therefore, known deinking processes are not entirely satisfactory in that the chemicals used are selective as to the type of ink. Also high concentrations of  
15 expensive chemicals are necessary to obtain effective deinking results and adequate pulp cleanliness is not necessarily achieved. Such processes are cost inefficient as well. Thus the present practice, employing known deinking processes and agglomeration  
20 chemicals have limitations to specific inks and require complex and expensive procedures to obtain recycled grade paper.

There is a need in the art for deinking processes which are less complex and cause agglomeration of all  
25 types of inks, both impact and nonimpact, for all grades of paper. This invention is directed to the provision of such processes which have a wide range of applications in creating recycled grade paper. It would be appreciated that advantage over conventional deinking procedures  
30 would be obtained by providing an effective and efficient deinking method applicable to all types of printed paper.

Accordingly, it is a broad object of the invention to provide an improved deinking process producing more effective agglomeration of ink particles through  
35 separation of the defibering and agglomeration steps

-4-

which are subject to different chemical and physical requirements.

A more specific object of the invention is to provide a deinking process and related apparatus which  
5 utilizes a two-component deinking chemical, including alkanol and nonionic surfactant based systems, for the agglomeration of all types of inks, both impact and nonimpact, from wood containing and wood free grades of paper, in which the components are added separately  
10 during the deinking process to improve agglomeration of ink particles.

Another object of the invention is to provide a low cost agglomeration deinking process that effectively and efficiently removes ink without using a high  
15 concentration of expensive agglomeration chemicals.

A further specific object of the invention is to provide an agglomeration deinking method which is less complex than the prior art ink removal procedures.

Another object of the invention is to provide a  
20 recycled paper product having a high level of cleanliness made by the agglomeration deinking method of the invention from printed wastepaper.

A further specific object of the invention is to increase the pulp brightness by recirculating the  
25 substantially ink free pulp medium and reject streams to cause additional agglomeration of ink particles, wherein removal of the reagglomerated ink particles produces a cleaner pulp medium.

### 30 Disclosure of Invention

In the present invention, these purposes, as well as others which will be apparent, are achieved generally by providing an improved agglomeration process for deinking printed paper by separating the defibering and  
35 agglomeration steps which are subject to different

-5-

chemical and physical requirements. Size and density separation procedures are employed to remove agglomerated ink particles and produce an ink free pulp medium for use in the fabrication of recycled paper and board products.

5       The general deinking process comprises the steps of defibering the printed paper to produce a pulp medium and contacting with an aqueous medium containing an agglomeration deinking chemical. The presence of the deinking chemical causes agglomeration of ink particles  
10 to produce an ink pulp medium. The defibering and agglomeration are done separately in a defibering device and mixing vessel, respectively. The ink pulp medium is passed through a pressure screen and/or a centrifugal cleaner to remove ink particles and to produce a  
15 substantially ink free pulp medium. The substantially ink free pulp medium is thickened and then made into a recycled paper product through conventional papermaking processes.

      In an alternate embodiment of the process of the  
20 invention the substantially ink free pulp medium is recirculated to the mixing vessel to cause additional agglomeration of ink particles. After reagglomeration, the agglomerated ink particles are removed to produce a cleaner pulp medium. Additionally, reject streams from  
25 the production of the substantially ink free pulp medium are recirculated to the mixing vessel to again cause additional agglomeration of ink particles. After reagglomeration, the agglomerated ink particles are removed to increase pulp medium yield and ink removal  
30 efficiency.

      The pulp medium may be subjected to screening and cleaning procedures to remove contaminants, such as staples, glass, paper clips, plastic and stickies prior to agglomeration in the mixing vessel.

35       The deinking chemicals used in the invention are

-6-

generally a blend of chemicals having at least two different component functionalities. The first component, referred to as chemical A, acts to remove ink from fiber, lower the glass transition temperature of polymeric inks and to wet the ink particles. The second component, referred to as chemical B, is a coalescing/liquid bridging agent. The process of the invention utilizes a single deinking agent containing the combined components and functionalities of deinking chemicals A and B. Alternatively, deinking chemical A and deinking chemical B are added as separate components at different points in the invention process. The net effect is to split up the chemical actions of the deinking agent to permit higher concentrations of active agents at the unit operations where they are most effective. Deinking chemical A is typically a nonionic surfactant of the ethoxylate type, whereas deinking chemical B is an alkanol which functions as a coalescing and/or liquid bridging agent.

Process and reaction conditions are controlled in the defibering device and mixing vessel so that effective agglomeration of the ink particles is accomplished. Concentrations of the deinking chemical, as well as the pH and temperature of the aqueous medium are adjusted to yield maximum agglomeration of the ink particles. Coarse and fine ink particles are removed from the ink pulp medium by size and density separation procedures. The resulting ink free pulp medium has a speck removal and brightness level sufficient to produce high-grade recycled paper.

Preferred applications of the method of the invention include use in deinking printed paper to produce high-grade recycled printing and writing paper, or other products such as tissue and towelling, bag grades or board products. Advantageously, the deinking

-7-

method of the invention provides agglomeration processes that are less complex and expensive and more efficient than known agglomeration processes.

5 The invention also provides an apparatus for deinking printed paper utilizing a repulping means, an agglomeration means, a separation means for coarse ink and fine ink and a production means to produce recycled paper.

10 Other objects, features and advantages of the present invention will be apparent when the detailed description of the preferred embodiments of the invention are considered in conjunction with the drawings, which should be construed in an illustrative and not limiting sense as follows:

15

#### Brief Description of the Drawings

FIG. 1A is a diagrammatic view of the general process steps of the invention for deinking of printed paper;

20

FIG. 1B is a diagrammatic view of another embodiment of the general process steps of the invention for deinking of printed paper;

25 FIG. 2 is a schematic view of an apparatus for deinking of printed paper in accordance with the process of the present invention; and

FIG. 3 is a schematic view of an apparatus for deinking of printed paper in accordance with another embodiment of the process of the invention.

30

#### DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENT

The present application is specifically directed to a deinking process which provides more effective agglomeration of ink particles by separating the defibering and agglomeration steps. Effective  
35 agglomeration of ink particles is achieved by splitting

-8-

the defibering and agglomeration actions which are subject to different chemical and physical requirements. Agglomerated ink particles are removed by size and density separation procedures to produce an ink free pulp medium used to make recycled paper and board products.

Copending U.S. Patent Application No. 07/771,370 filed October 3, 1991 discloses use of an agglomeration tower to separate the repulping and agglomeration steps. Specific deinking chemicals used in the present invention are disclosed in copending U.S. Patent Application Nos. 07/984,784 filed December 3, 1992 and 08/093,443 filed July 16, 1993, both applications are a continuation-in-part and a continuation, respectively, of U.S. Patent Application No. 07/720,336 filed June 25, 1991, now abandoned. The copending application specifications are incorporated herein by reference.

With further reference to the drawings, FIG. 1A and 1B are diagrammatic views of the general process steps for the deinking of printed paper.

Defibering of the printed paper occurs in a defibering device to produce a pulp medium 1. After defibering the pulp medium is passed to a mixing vessel containing a deinking chemical 2. The presence of the deinking chemical causes agglomeration of ink particles to produce an ink pulp medium. The resulting ink pulp medium is passed through one or more steps of screening 3 or centrifugal cleaning 4, to remove large ink particles and contaminants and to produce a substantially ink free pulp medium 6.

As shown in FIG. 1B, prior to reaching the mixing vessel 2 the repulped fibers may be subjected to screening and cleaning procedures 1A to remove contaminants, such as staples, glass, paper clips, plastic and stickies prior to agglomeration.

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-9-

In an alternate embodiment of the process of the invention, the substantially ink free pulp medium is recirculated to the mixing vessel 2 to cause additional agglomeration of ink particles, wherein reagglomerated ink particles are removed to produce a cleaner pulp medium. Additionally, reject streams from the production of the substantially ink free pulp medium are recirculated to the mixing vessel 2 to cause additional agglomeration of ink particles, wherein reagglomerated ink particles are removed to increase pulp medium yield.

In any of the embodiments the substantially ink free pulp medium can then be made into a recycled paper product through conventional papermaking techniques.

The critical aspect of the present invention process is in the separation of the defibering and agglomeration steps. The mechanical and chemical actions required for the defibering and agglomeration steps are different. The key differences are summarized in Table I below.

**TABLE I**  
**COMPARISON OF DEFIBERING AND AGGLOMERATION ACTIONS**

**MECHANICAL/PHYSICAL REQUIREMENTS**

| <b>DEFIBERING</b>    | <b>AGGLOMERATION</b>  |
|----------------------|-----------------------|
| High attrition/shear | Low attrition/shear   |
| Moderate mixing      | Very good mixing      |
| Batch/continuous     | Batch/semi-continuous |

**CHEMICAL REQUIREMENTS**

| <b>DEFIBERING</b>         | <b>AGGLOMERATION</b>                    |
|---------------------------|-----------------------------------------|
| Any pH                    | Preferably pH 7 to 11                   |
| Wetting agents (optional) | Coalescing and/or liquid bridging agent |
| Low temperature           | Normally elevated temperature           |

-10-

Generally, the defibering step is carried out in a high shear device such as a hydropulper. Other devices providing similar action can also be employed. The defibering step is carried out preferably at low  
5 temperature in the range of 25 to 45°C with or without alkali to cause separation of the paper fibers. Preferably defibering is carried out for 10 to 20 minutes.

For effective agglomeration to occur, low shear  
10 mixing is preferred which may be achieved in a mixing vessel which is a device other than a pulper. A pulper is generally used for the defibering step and not the agglomeration step. Processes in which the repulping and agglomeration actions are carried out in a hydropulper or  
15 a similar device result in the hydropulper breaking the ink into smaller particles at the same time that the agglomeration action is forming larger particles. By separating the repulping and agglomeration steps agglomeration will proceed without any particle breakdown  
20 thereby producing larger agglomerates which are easier to remove from the pulp. This increases the utilization of pulper capacity, which can be a serious limitation in some commercial operations. The invention process therefore provides for increased production through  
25 better pulper utilization and increased yield of clean pulp.

In addition to agglomeration the mixing vessel can also be used for bleaching the pulp since temperature and pulp consistency conditions are suitable. Multiple stage  
30 bleaching could be accomplished in the vessel because of its design for continuous gentle agitation.

After defibering, the pulp medium is passed to the mixing vessel where the deinking chemicals are added.

Agglomeration deinking chemicals used in the  
35 invention are a blend of chemicals having at least two

-11-

different component functionalities. The first component, referred to as chemical A, acts to remove ink from fiber, lower the glass transition temperature of polymeric inks and to wet the ink particles. The second  
5 component, referred to as chemical B, is a coalescing/liquid bridging agent. The process of the invention utilizes a single deinking agent containing the combined components and functionalities of deinking chemicals A and B. Alternatively, deinking chemical A  
10 and deinking chemical B are added as separate components at different points in the invention process. The net effect is to split up the chemical actions of the deinking agent to permit higher concentrations of active agents at the unit operations where they are most  
15 effective.

Typically, Chemical A, which removes ink from the paper fiber and reduces the glass transition temperature of inks, is preferably an ethoxylated nonionic surfactant, but any other nonionic surfactant may be  
20 used. Chemical B is an alkanol, preferably a mixture of C<sub>5</sub> to C<sub>20</sub> alkanols, but any liquid bridging or coalescing agent for ink may be used.

Any deinking agent as known in the art can be used in the process of the invention. Many types of deinking  
25 agents which may be used have been proposed in copending U.S. Patent Application Nos. 08/093,443 and 07/984,784. These applications disclose deinking compositions including a mixture of C<sub>5</sub> to C<sub>20</sub> alkanols and nonionic surfactants. Alkanols with a carbon number range from 8  
30 to 16 having a straight chain or a slightly branched structure are most preferred. Alternatively, secondary or tertiary alcohols or any chemical agent capable of strong hydrogen bonding and forming liquid bridges between ink particles causing them to agglomerate, are  
35 used in the invention.

-12-

The prior applications also disclose a wide variety of nonionic surfactants which may be used in the invention including polyakyleneoxy ether, polyoxyalkylether, polyoxyethylenephenol ether, oxyethylene-oxypropylene block copolymer, polyoxyethylenealkylamine, sorbitan fatty acid ester, polyoxyethylenesorbitan fatty acid ester, polyethylene glycol esters and diesters and any other nonionic surface active agents with wetting power to reduce the surface tension of water molecules. Particularly preferred nonionic surfactants are of the polyakyleneoxy ether type or ethoxylate derivatives of the  $C_5$  to  $C_{20}$  alkanols, having an ethoxylate content sufficient to provide detergency or wetting. Typically, the ethoxy (EO) group to alcohol, mole/mole average ratio of the deinking compositions range from 0.001 to 12. Hydrophobicity of the compositions as measured hydrophobic-hydrophilic balance (HLB) values range from 0.5 to 12. The cloud point of the compositions are less than 200 °F and hydroxyl values (expressed as eq./100 g) are greater than 0.0001.

Further embodiments of the deinking compositions have HLB values ranging from 0.5 to 6, ethoxy group to alcohol, mole/mole avg. ratio ranging from 0.1 to 5, cloud points in the range of 5 to 100 °F and a hydroxyl value between 0.1 and 1.

Other deinking agents which may be used in the invention include a mixture of one or more  $C_5$  -  $C_{20}$  alkanols and alcohol ethoxylates in the ratio of 4:1 to 1:1.5 of alkanols to alcohol ethoxylates. Specifically, where for every 10 parts by weight of alkanol and alcohol ethoxylates there are between 3.0 and 8.0 parts alkanol and between 2.0 and 7.0 parts alcohol ethoxylates.

FIGS. 2 and 3 illustrate embodiments of the invention process. The net effect of the invention

-13-

process is to separate the defibering and agglomeration actions. In FIG. 2, a single deinking agent containing the combined components and functionalities of deinking chemicals A and B are added to the mixing vessel, 14. Alternately, in FIGS. 2 and 3, the deinking chemical components A and B are split and added at different points in the invention process. Deinking chemical A is added to the pulper 12, and deinking chemical B is added to the mixing vessel 14. This latter embodiment splits the chemical actions of the deinking agent to permit higher concentrations of active agents at the unit operations where they are most effective.

FIG. 2 is a schematic view of an apparatus, generally designated 10, for practicing the deinking process of the invention. The process of the invention entails defibering the wastepaper, causing agglomeration of the ink particles with the addition of a deinking chemical in aqueous medium, and removal of the agglomerated ink by screening and forward cleaning.

As shown in FIG. 2, the printed paper is repulped in an aqueous medium at hydropulper 12 to produce a pulp medium. The defibering of the printed paper is generally at temperatures in the range of 25 to 45° and carried out for a time long enough to cause separation of the paper fibers. The aqueous medium in the mixing vessel is maintained at a pH in the range of 7 - 11.5 and at temperatures in the range of 40 - 90 °C.

Any type of printed paper may be used in this invention including computer printout paper, writing paper, fine paper, coated and uncoated magazine paper, newsprint and packaging board. This list is merely representative of the different types of printed paper and is not considered to be inclusive of all the possible types of printed paper which may be used in the invention.

-14-

**TABLE II**  
**TYPES OF PRINTED PAPER**

|    |                           |
|----|---------------------------|
| 5  | COMPUTER PRINTOUT PAPER   |
|    | WRITING PAPER             |
|    | FINE PAPER                |
| 10 | COATED/UNCOATED MAGAZINE  |
|    | COATED PUBLICATION GRADES |
|    | MANILA FILE FOLDERS       |
| 15 | NEWSPRINT                 |
|    | PACKAGING BOARD           |

20

The sorted printed waste paper is slushed at consistency range between 3 and 30%, in the hydropulper. The repulping is preferably done in an alkali aqueous medium, with sodium hydroxide added to speed the repulping and to aid in the separation of ink particles from the pulp fibers. Ambient temperature is preferred when using highly contaminated waste in the hydropulper since at higher temperatures contaminants, such as stickies, would break down making them difficult to remove. The pulp medium may optionally be passed to a detrashing unit (not shown), which removes large contaminants such as large pieces of plastic or metal.

After defibering, the pulp medium is then pumped to a mixing vessel 14 where the addition of the deinking chemical causes agglomeration of ink particles to produce an ink pulp medium. For effective agglomeration to occur, low shear mixing is preferred and is achieved in devices other than a pulper. Typically, the pulp medium is passed to the mixing vessel for at least 10 minutes, preferably from 20 to 60 minutes, and maintained at a pH in the range of 7 - 11.5 at temperatures in the range of 40 to 90°C. The deinking chemical is present preferably

-15-

at a dosage range between 0.1 to 3% by weight, calculated on the dry weight of the pulp used.

The ink pulp medium is passed through dump chest 16 and a liquid cyclone 18, for removal of heavy contaminants, such as paperclips, staples and glass. After removal of the heavy debris the ink pulp medium is passed through a series of slotted screens 20, 22. Preferably, the ink pulp medium, at consistencies ranging from 0.5 to 6.0%, and temperatures from 25-55°C, is passed through slotted pressurized screens to remove coarse contaminants and large ink particles. The coarse screens 20, typically have hole sizes in the range of 0.040" to 0.062", and the fine screens 22, have slots of width between 0.006" to 0.012". Preferably, secondary and tertiary screening stages are often used for both coarse and fine screening to reduce the loss of good fiber from the system.

The slotted screens 22, remove a large percentage of the stickies from the system, including adhesives from self-stick envelopes and labels. Removal of the stickies is critical during the deinking process. High efficiency stickies removal by the fine screens is achieved in the system by maintaining the screening temperature at close to ambient temperature, so that the stickies do not become softened and more deformable for extrusion through the slots into the pulp accepts stream.

The ink pulp medium is diluted to 1% consistency or less at dilution tank 24, for pumping through a set of forward cleaners (centrifugal cleaners) 26, 28, for removal of the agglomerated ink particles denser than water to produce a substantially ink free pulp medium.

The large ink particles from the screening and cleaning steps are easily separated into the cleaner rejects, and are removed from the system for disposal by landfilling or burning or for other uses.

-16-

Optionally, the substantially ink free pulp medium may be recirculated to the mixing vessel 14. The presence of the deinking agent B causes additional agglomeration of ink particles which are subsequently removed by screening and cleaning procedures. In addition, reject streams from the production of the substantially ink free pulp medium may be recirculated to the mixing vessel 14 where the presence of the deinking agent causes additional agglomeration of ink particles. The reagglomerated ink particles are removed by screening and cleaning procedures. The further steps of recirculating the substantially ink free pulp medium and reject streams result in increased pulp medium yield and produce a cleaner pulp medium.

The substantially ink free pulp is thickened 30, and used to make recycled paper by conventional papermaking techniques.

FIG. 3 is a modification of the process of the invention in which the deinking chemical is separated into chemical components A and B which are added at different points in the invention process. This embodiment splits the chemical actions of the deinking agent to permit higher concentrations of active agents at the unit operations where they are most effective.

In general, the printed paper is defibered in a hydropulper 12 and passed to a mixing vessel 14, where deinking chemical A is added. Deinking chemical A acts to remove ink from the pulp fiber, lower the glass transition temperature of polymeric inks and wet the ink particles. Preferably, deinking chemical A is an ethoxylated nonionic surfactant, though many others may be used as previously indicated. Contaminant removal is achieved by passing the pulp medium through dump chest 16, liquid cyclone 18, slotted screens 20, 22. After passing through dilution tank 24 and forward cleaning

-17-

stations 26, 28 the reject streams are passed to chest 34 where deinking chemical B is added. Deinking chemical B is a coagulating/liquid bridging agent and is preferably an alkanol. The presence of deinking chemicals A and B causes agglomeration of ink particles to produce an ink pulp medium. This ink pulp medium is recirculated to the dump chest 14, and passed through screening and centrifugal cleaners to remove ink particles from the ink pulp medium to produce a substantially ink free pulp medium.

Preferably chemical A at a dosage of 0.2 to 0.4% by weight of pulp is added in the mixing vessel 14. This addition helps reduce the pulping time to that sufficient for defibering alone, 10 minutes instead of the normal 30 minutes, and allows a lower temperature to be used in the pulper. This change would also provide for higher throughput in the pulper.

Chemical B is added at a dosage of 0.2 to 0.5% by weight of pulp. This leads to a 50% or more cost savings in chemical costs and is more effective than conventional processes.

Numerous alternative embodiments to the process of the invention are possible. Another alternative embodiment, is the replacement of the first set of forward cleaners 26 with pressure screens having fine slots, between 0.006" to 0.012" slot width, for removal of the large agglomerated ink particles.

In yet another alternative embodiment, additional sets of forward cleaners 26, 28 can be added in a 2°, 3°, or 4° arrangement for further improvement in the cleanliness and yield of the pulp.

A comparison of conventional agglomeration deinking methods and the method of the invention is shown in Example I below. In the conventional deinking process printed wastepaper was defibered and contacted with a

-18-

deinking chemical in the same vessel, a hydropulper, to produce an ink pulp medium. Alternatively, utilizing the process steps of the invention the printed paper was defibered in the hydropulper and then passed to a mixing vessel where a deinking agent was added. The deinking agent used in Example I is a mixture of one or more C<sub>5</sub> - C<sub>20</sub> alkanols and nonionic surfactants. Example II illustrates the process of the invention in which the deinking chemical is separated into chemical components A and B which are added at different points in during the deinking process. This embodiment splits the chemical actions of the deinking agent to permit higher concentrations of active agents at the unit operations where they are most effective.

These examples are merely representative and are not inclusive of all the possible embodiments of the invention.

## EXAMPLE I

Conventional Agglomeration Deinking Method

Laser computer printout (CPO) wastepaper was repulped at 5% consistency in a Black Clawson Hydropulper at pH 10 simultaneously with 0.8% by weight of an agglomeration deinking agent, which is a mixture of one or more C<sub>5</sub> - C<sub>20</sub> alkanols and nonionic surfactants. The agglomeration was carried out for 30 minutes at 70°C in the pulper. Samples of the ink pulp medium were taken after the agglomeration and labelled "Conventional". Brightness and dirt/speck count measurements were taken. The results are listed below in Table III.

Control

In a comparative experiment, the laser CPO was repulped at pH 10 with no deinking agent for 10 minutes. A sample of the ink pulp medium was taken and labelled "Repulped" and brightness/dirt/speck measurements were carried out.

-19-

Invention Agglomeration Deinking Method

The Repulped sample, from the control experiment, at 5% consistency was transferred to a 5 gallon vessel. The same agglomeration deinking agent as used in the Conventional method was added at 0.8% by weight of pulp. The temperature was maintained at 60°C with continuous stirring using a "Lonar" stirrer to provide sufficient mixing for 30 minutes. Brightness/dirt/speck measurements were taken on this ink pulp medium and labelled "Modified". The results are tabulated below in Table III.

EXAMPLE I  
TABLE III

| SAMPLE       | BRIGHTNESS<br>(G.E.) | DIRT COUNT<br>(ppm) | % DIRT<br>REDUCTION |
|--------------|----------------------|---------------------|---------------------|
| REPULPED     | 74.02 (0.41)         | 10194               | 0                   |
| CONVENTIONAL | 75.98 (0.32)         | 8476                | 16.9                |
| MODIFIED     | 77.48 (0.37)         | 7800                | 23.5                |

Note: Numbers in parenthesis are the standard deviation in the sample.

The results clearly demonstrate the invention process increases the % dirt reduction over conventional agglomeration deinking processes. The separation of the defibering and agglomeration actions produce a pulp of higher brightness and lower dirt count. Thus the invention process increases the agglomeration action which results in cleaner pulp and higher yield. The additional advantages of the invention process are higher throughput, no pulper capacity limitation and lower temperatures for agglomeration.

-20-

## EXAMPLE II

Twenty five pounds (25 lbs) of sorted white ledger (SWL) was added to 35 gallons of water at 70°C and repulped for 5 minutes. A sample, labelled A, was taken  
5 after repulping and measured for brightness and dirt count. The results are listed below in Table IV. The remaining repulped sample was split into sample B and C and treated with a deinking chemical comprised of a mixture of C<sub>12-16</sub> alcohol ethoxylates and C<sub>10-12</sub> alkanols by  
10 the following processes.

To sample B, 0.8% by weight of a mixture of 1.25:1 blend of Alfol™1012 to Alfonic™1216-22 was added. Alfol™1012 is a blend of C<sub>10-12</sub> alkanols and Alfonic™1216-22 is C<sub>12-16</sub> alcohol ethoxylates, both chemicals are from  
15 Vista Chemicals, Houston, Texas. Sample B was repulped with the deinking agent for 30 minutes at 70°C. Brightness and dirt count measurements were taken and listed below in Table IV.

In sample C, the same deinking chemical and  
20 component proportions were used, however, the components were added during separate process steps. First the same amount of Alfonic™1216-22 used in sample B was added to sample C and repulped for 10 minutes. Five gallons of the Alfonic™1216-22 treated sample was transferred to a  
25 vessel where Alfol™1012 was added at the same percent weight based on oven dry pulp as in sample B. The mixture was stirred for 30 minutes at 70°C. Brightness and dirt count measurements were taken and listed in Table IV.

-21-

EXAMPLE II  
TABLE IV

| SAMPLE | BRIGHTNESS<br>(% G.E.) | DIRT COUNT<br>(mm <sup>2</sup> /m <sup>2</sup> of<br>sheet) | SPECK COUNT<br>(#/sq.m) |
|--------|------------------------|-------------------------------------------------------------|-------------------------|
| A      | 74.4                   | 10,879                                                      | 156,905                 |
| B      | 74.3                   | 5,300                                                       | 88,921                  |
| C      | 77.1                   | 5,310                                                       | 83,381                  |

The brightness data in Table IV shows an increase in brightness of 2.7% GE when the chemicals are added separately (sample C). This is due to improved agglomeration, i.e. fewer small ink specks. The average particle size at the end of the conventional treatment (adding both components together- sample B) is 0.059 sq.mm. On the other hand when we split the components and add them as necessary, then we increase the average particle size to 0.064 sq. mm (sample C). This demonstrates that split chemical addition provides for improved agglomeration.

The simplicity of the equipment used and the high amount of ink removal make the agglomeration deinking process of the invention advantageous over prior art practice.

Advantageously, the method of this invention for deinking printed paper is less complex than conventional deinking processes involving washing and flotation procedures and known agglomeration procedures.

It will be recognized by those skilled in the art that the invention has wide application in deinking a variety of printed paper to produce recycled paper.

There are numerous advantages to separate the defibering and agglomeration actions over conventional

-22-

agglomeration deinking processes in which the actions occur simultaneously under the same conditions. Overall improved effectiveness in agglomeration deinking, lower chemical costs, fewer screening/cleaning steps are  
5 generally required and improved hydropulper utilization are among some of the advantages to the process of the invention.

Specifically, advantages of the process of this invention include the increased utilization of pulper  
10 capacity which can be a serious limitation in commercial operations. Through better pulp and deinking chemical utilization more effective agglomeration occurs leading to better ink removal and increased yield of brighter, cleaner pulp. The higher effectiveness of the  
15 agglomeration step leads to lesser equipment requirements and decreased capital costs. The invention process also adds deinking chemicals at points where they would be most efficient and could lead to a lower dosage of chemicals used.

20 Numerous modifications are possible in light of the above disclosure such as application of alternative agglomeration deinking chemicals chosen according to the wastepaper treated. In addition, alternative process parameters may be employed in the invention, which  
25 include using the deinking agent with no pH adjustment to the wastepaper; using the deinking agent in an alkali pH range; or using the deinking agent in the presence of

-23-

other chemicals suitably employed in a deinking and/or papermaking process such as bleaching agents, defoamers, sizing agents, brighteners, water quality processing agents among others.

5           Therefore, although the invention has been described with reference to certain preferred embodiments, it will be appreciated that other composite structures and processes for their fabrication may be devised, which are nevertheless within the scope and spirit of the invention  
10 as defined in the claims appended hereto.

-24-

Claims

1. A method to deink printed paper comprising the steps of:

defibering the printed paper to produce a pulp  
5 medium;

contacting said pulp medium with an aqueous medium containing a deinking chemical, wherein the presence of said deinking chemical causes agglomeration of ink particles to produce an ink pulp medium; and

10 passing said ink pulp medium through a screen and/or centrifugal cleaner to remove ink particles from said ink pulp medium to produce a substantially ink free pulp medium.

15 2. The method as defined in claim 1, wherein said defibering of the printed paper is at temperatures in the range of 25 to 45°C and carried out for a time long enough to cause separation of the paper fibers.

20 3. The method as defined in claim 1, wherein said aqueous medium is maintained at a pH in the range of 7 - 11.5 and at temperatures in the range of 40 - 90 °C.

25 4. The method as defined in claim 1, wherein said deinking chemical is present at a dosage ranging from between 0.1 -3.0% by weight, calculated on the dry weight of the pulp used.

-25-

5. The method as defined in claim 1, wherein said pulp medium is passed to a mixing vessel, for at least 10 minutes maintained at a pH in the range of 7 - 11.5 and at temperatures in the range of 40 - 90 °C, containing  
5 said deinking chemical to produce said ink pulp medium.

6. The method as defined in claim 1, wherein said deinking chemical is a mixture of alkanols and nonionic surfactants selected from the group comprising of  
10 polyakyleneoxy ether, polyoxyalkylether, poloxyethylenephenol ether, oxyethylene-oxypropylene block copolymer, polyoxyethylenealkylamine, sorbitan fatty acid ester, polyoxyethylenesorbitan fatty acid ester, polyethylene glycol esters and diesters or any  
15 other nonionic surface active agents with wetting power to reduce the surface tension of water molecules

7. The method as defined in claim 1, wherein said deinking chemical is a mixture of alkanols and alcohol  
20 ethoxylates.

8. The method as defined in claim 7, wherein said deinking chemical is a mixture of one or more C<sub>5</sub> - C<sub>20</sub> alkanols and alcohol ethoxylates.

-26-

9. The method as defined in claim 8, wherein said deinking chemical is for every 10 parts by weight of alkanol and alcohol ethoxylates between 3.0 and 8.0 parts alkanol and between 2.0 and 7.0 parts alcohol  
5 ethoxylates.

10. The method as defined in claim 1, comprising the further step of passing said pulp medium through screening and/or cleaning procedures to remove coarse  
10 contaminants.

11. The method as defined in claim 5, comprising the further step of recirculating said substantially ink free pulp medium to said mixing vessel to cause  
15 additional agglomeration of ink particles, wherein reagglomerated ink particles are removed to produce a cleaner pulp medium.

12. The method as defined in claim 5, comprising  
20 the further step of recirculating a reject stream from the production of said substantially ink free pulp medium to said mixing vessel to cause additional agglomeration of ink particles, wherein reagglomerated ink particles are removed to increase pulp medium yield.

25

-27-

13. The method as defined in claim 1, comprising the further step of passing said ink pulp medium, at consistencies ranging from 0.5 - 6.0%, and temperatures from 25 -55 °C through slotted pressurized screens to  
5 remove coarse contaminants and large ink particles.

14. The method as defined in claim 1, wherein said ink pulp medium is passed through a centrifugal cleaner to remove ink particles denser than water to produce said  
10 substantially ink free pulp medium.

15. The method as defined in claim 1, comprising the further step of producing a paper product from said deinked pulp medium by conventional papermaking  
15 techniques.

16. A method to deink printed paper comprising the steps of:

defibering the printed paper to produce a pulp  
20 medium;

contacting said pulp medium with an aqueous medium containing deinking chemical A, which acts as a wetting agent, to produce an ink pulp medium;

passing said ink pulp medium to a mixing vessel  
25 containing deinking chemical B, which acts as a coalescing/liquid bridging agent, to cause agglomeration of ink particles; and

-28-

passing said ink pulp medium through a screen and/or centrifugal cleaner to remove ink particles from said ink pulp medium to produce a substantially ink free pulp medium.

5

17. The method as defined in claim 16, wherein said defibering of the printed paper is at temperatures in the range of 25 to 45°C and carried out for a time long enough to cause separation of the paper fibers.

10

18. The method as defined in claim 16, wherein said aqueous medium is maintained at a pH in the range of 7 - 11.5 and at temperatures in the range of 40 - 90 °C.

15

19. The method as defined in claim 16, wherein said deinking chemical A is present at a dosage ranging from between 0.1 -1.0% by weight, calculated on the dry weight of the pulp used and said deinking chemical B is present at a dosage ranging from between 0.1 -3.0% by weight, calculated on the dry weight of the pulp used.

20

20. The method as defined in claim 16, wherein said ink pulp medium is passed to said mixing vessel for at least 10 minutes maintained at a pH in the range of 7 - 11.5 and at temperatures in the range of 40 - 90 °C.

25

-29-

21. The method as defined in claim 16, wherein said deinking chemical A is a nonionic surfactant selected from the group comprising of polyakyleneoxy ether, polyoxyalkylether, poloxyethylenephenol ether, oxyethylene-oxypropylene block copolymer, polyoxyethylenealkylamine, sorbitan fatty acid ester, polyoxyethylenesorbitan fatty acid ester, polyethylene glycol esters and diesters or any other nonionic surface active agents with wetting power to reduce the surface tension of water molecules

22. The method as defined in claim 16, wherein said deinking chemical A is an alcohol ethoxylate or a nonionic surfactant and said deinking chemical B is an alkanol.

23. The method as defined in claim 22, wherein said deinking chemical B is a mixture of one or more  $C_5 - C_{20}$  alkanols.

20

24. The method as defined in claim 22, wherein for every 10 parts by weight of alkanol (chemical B) and alcohol ethoxylates (chemical A) there is between 3.0 and 8.0 parts alkanol and between 2.0 and 7.0 parts alcohol ethoxylates.

-30-

25. The method as defined in claim 16, comprising the further step of passing said pulp medium through screening and/or cleaning procedures to remove coarse contaminants.

5

26. The method as defined in claim 25, comprising the further step of recirculating said substantially ink free pulp medium to said mixing vessel to cause additional agglomeration of ink particles, wherein  
10 reagglomerated ink particles are removed to produce a cleaner pulp medium.

27. The method as defined in claim 25, comprising the further step of recirculating a reject stream from  
15 the production of said substantially ink free pulp medium to said mixing vessel to cause additional agglomeration of ink particles, wherein reagglomerated ink particles are removed to increase pulp medium yield.

20 28. The method as defined in claim 16, comprising the further step of passing said ink pulp medium, at consistencies ranging from 0.5 - 6.0%, and temperatures from 25 -55 °C through slotted pressurized screens to remove coarse contaminants and large ink particles.

25

29. The method as defined in claim 16, wherein said ink pulp medium is passed through a centrifugal cleaner

-31-

to remove ink particles denser than water to produce said substantially ink free pulp medium.

30. The method as defined in claim 16, comprising  
5 the further step of producing a paper product from said deinked pulp medium by conventional papermaking techniques.

31. A recycled paper product made from a  
10 substantially ink free pulp medium, wherein said substantially ink free pulp medium is produced by defibering printed wastepaper to form a pulp medium and contacting said pulp medium with a deinking chemical to cause agglomeration of ink particles to form an ink pulp  
15 medium and removal of said agglomerated ink particles from said ink pulp medium to produce said substantially ink free pulp medium.

32. The recycled paper product as defined in claim  
20 31, wherein said printed wastepaper is cellulosic material including both wood containing and wood free grades of paper.

33. The recycled paper product as defined in claim  
25 31, wherein said substantially ink free pulp medium has a speck removal and brightness level sufficient to produce a high-grade recycled paper.

-32-

34. The recycled paper product as defined in claim 31, wherein said ink pulp medium is passed through slotted screens to remove coarse contaminants and large ink particles.

5

35. The recycled paper product as defined in claim 31, wherein said ink pulp medium is passed through a centrifugal cleaner to remove ink particles denser than water to produce a fine ink particle stream.

10

36. The recycled paper product as defined in claim 31, wherein said ink free pulp medium is formed into a paper product by conventional papermaking techniques.

15

37. The recycled paper product as defined in claim 31, wherein said deinking chemical is a mixture of alkanols and nonionic surfactants.

38. An apparatus for deinking impact or nonimpact printed paper to produce recycled grade paper comprising:

defibering means for defibering the printed paper into a pulp medium;

agglomerating means for separating ink particles from the printed paper and agglomerating the ink particles to produce an ink pulp medium;

25

separating means for separating the agglomerated ink particles from said ink pulp medium by size and density

-33-

separation to produce a substantially ink free pulp medium; and

production means for producing recycled grade paper from said substantially ink free pulp medium.

5

39. The apparatus as defined in claim 38, wherein said repulping means is a hydropulper, kneader or other similar apparatus which is sufficient to defiber the printed wastepaper.

10

40. The apparatus as defined in claim 38, wherein said agglomeration means is a deinking chemical which is a mixture of C<sub>5</sub> - C<sub>20</sub> alkanols and nonionic surfactants.

15

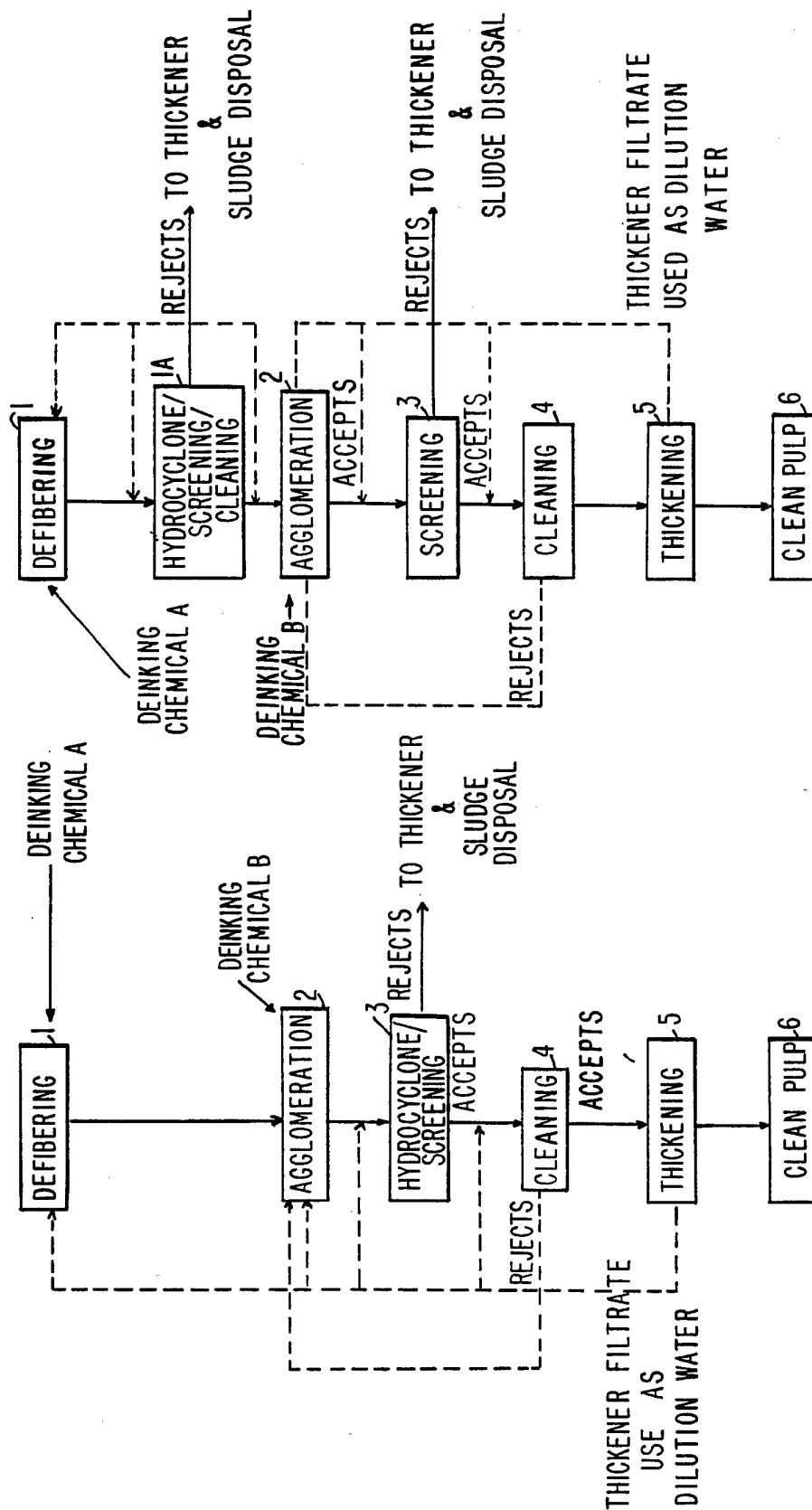
41. The apparatus as defined in claim 38, wherein said separating means is a slotted or pressurized screen to remove coarse contaminants.

20

42. The apparatus as defined in claim 38, wherein said separating means is a centrifugal cleaner to remove ink particles denser than water to produce said substantially ink free pulp medium.

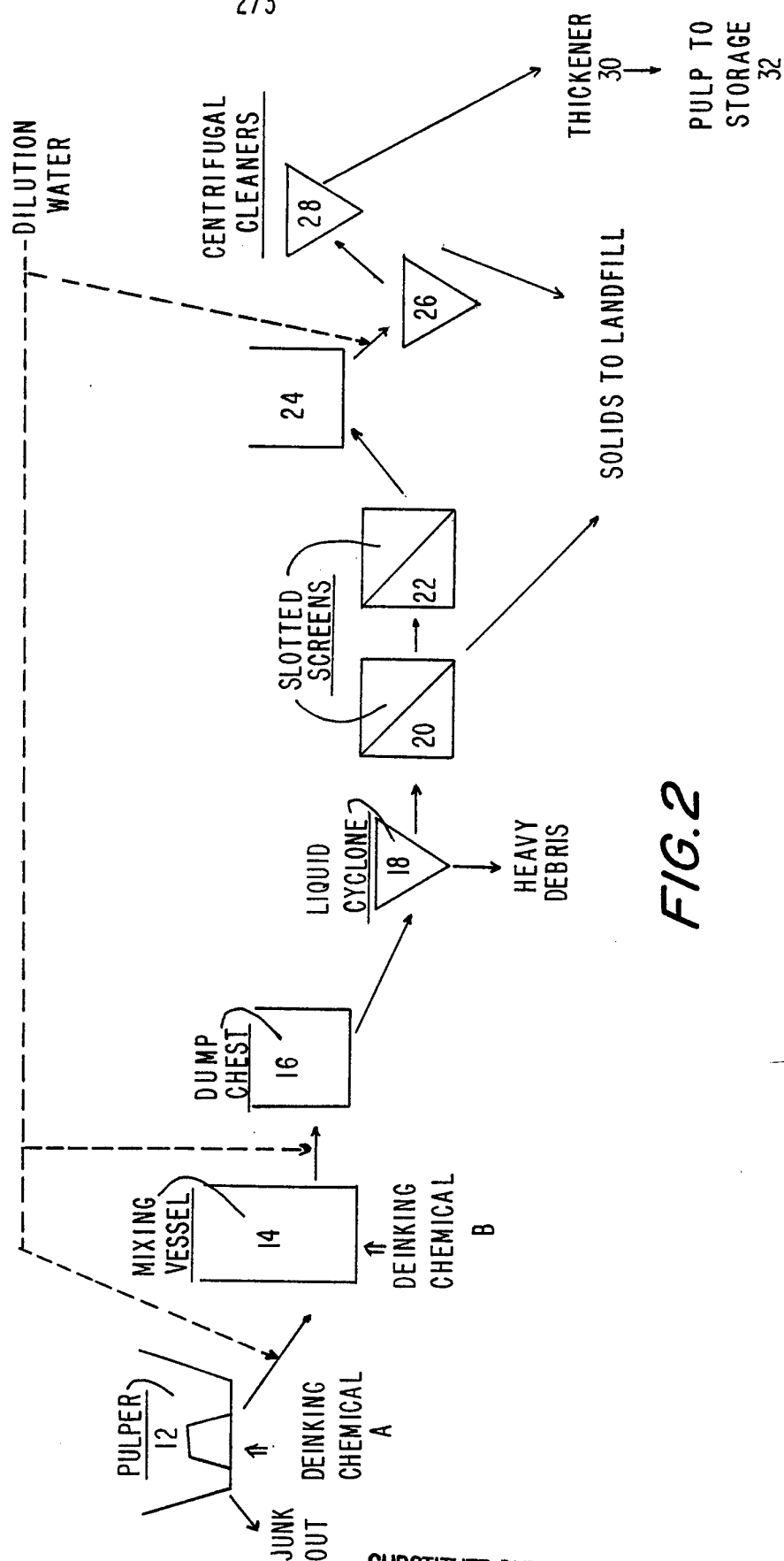
25

43. The apparatus as defined in claim 38, wherein said production means is conventional paper production apparatus to produce high-grade recycled paper.



**FIG. 1B**

**FIG. 1A**



**FIG. 2**

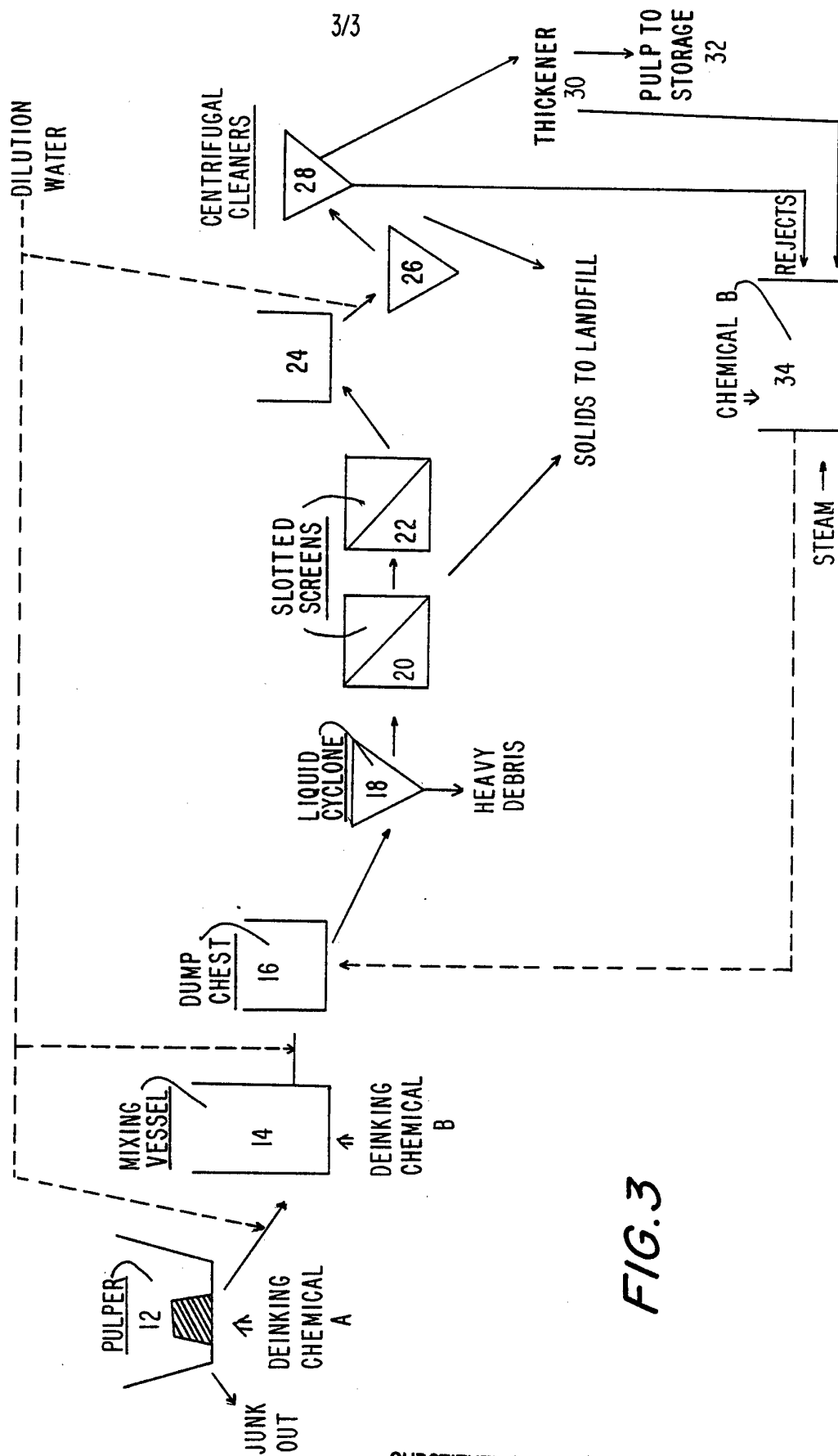


FIG. 3

## INTERNATIONAL SEARCH REPORT

International application No.  
PCT/US94/08774

## A. CLASSIFICATION OF SUBJECT MATTER

IPC(5) :D21C 5/02

US CL :162/5

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 162/5, 4

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

| Category*    | Citation of document, with indication, where appropriate, of the relevant passages | Relevant to claim No.                                        |
|--------------|------------------------------------------------------------------------------------|--------------------------------------------------------------|
| X<br>--<br>Y | US, A, 4,561,933 (WOOD ET AL.) 31 December 1985, column 3, lines 60-67.            | 1-9, 11-24, 29-31, 35-37<br>----- 10-13, 16-25, 29-30, 32-34 |
| Y            | US, A, 4,895,622 (BARNETT ET AL.) 23 January 1990, column 4, lines 1-6.            | 16-24, 29-30                                                 |
| Y            | US, A, 4,820,379 (DARLINGTON ET AL.) 11 APRIL 1989, column 7, lines 6-26.          | 25, 34, 10-13                                                |

☐ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

|                                                                                                                                                                         |                                                                                                                                                                                                                                                  |
|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| * Special categories of cited documents:                                                                                                                                | "T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention                                              |
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| "P" document published prior to the international filing date but later than the priority date claimed                                                                  |                                                                                                                                                                                                                                                  |

Date of the actual completion of the international search

05 OCTOBER 1994

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