Semiannual Patents Review July—December 1999

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KEYWORDS

Chemicals, Deinking, Enzyme, Patent, Recycling, Review, Stickies, Waste paper.

INTRODUCTION

This review summarizes patents related to paper recycling that were issued during the last six months of 1999. Two on-line databases, Claims/U.S. Patents Abstracts and Derwent World Patents Index, were searched for this review. This semiannual feature is intended to inform readers about recent developments in equipment design, chemicals and process technology for recycling paper, and alternative products derived from post consumer paper. Only brief summaries of individual inventions comprise this review. For more complete information, readers will need to access the full text of a specific patent.

We organized the patents into the following broad categories of recycling: equipment-processes, recycled products, chemicals, and efficient recycling. The global potential for recycled paper is reflected by the predominance of world patents cited in this review.

EQUIPMENT-PROCESSES

Paper Preparation

All recycling starts with separating the selected paper from wastes and preparing the paper to facilitate repulping.

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Office waste materials can be segregated from the paper component, shredded, and compacted with equipment described in a world patent (1). Compacted paper is baled, and waste material (cans, plastic, etc.) is baled separately and ejected from the compactor. The machine, small enough to be used in an office environment, reduces both the volume and disposal cost of waste materials.

Equipment and the accompanying process for more efficient wetting of baled shredded paper prior to fiberization is covered by another world patent (2). The process uses vacuum to thoroughly wet wastepaper to shorten repulping time and minimize fiber damage during repulping.

Flotation

Various approaches For improving flotation equipment and stages were the topic of several recently issued patents.

Extremely small contaminants, such as flexographic ink particles, are difficult to remove by conventional deinking methods and equipment. Specialized flotation equipment and a related deinking process have been developed and the patent rights assigned to Voith Sulzer (3). Flotation incorporates process water from the thickener to dilute the pulp slurry. Fine hydrophobic contaminant particles act as nuclei that attach to microbubbles (0.1-0.3 mm). Chemically assisted contaminant separation successfully separates pulp fibers by flotation.

Pressurized flotation developed by Beloit Technologies (4) uses a module based upon circulation through multiple baffles to promote stock mixing and divert stock through various outlets to a cyclone separator. A detailed schematic of the module is included in the patent. The

advantage of this system is to produce a higher brightness pulp.

A flotation process incorporating an inverted conical vessel with air bubbles fed faster than the pulp feed uses a water spray at the top of the vessel to separate pulp fibers from foam. Ink-containing foam is removed from the top of the vessel. This novel flotation system and corresponding deinking process (5) claims an improved fiber yield.

Oji Paper's (6) approach to flotation introduces air into the lower portion of the treatment tank. The liquid from the upper portion of the tank is transferred to a vapor liquid separation tank in which ink-containing froth is ejected by overflow. The claimed advantage is reduced air bubbles intermingled in the pulp slurry compared with conventional flotation.

Lamort's (7) improvement to flotation efficiency directs the pulp slurry from the top to the bottom of the chamber, whereas air bubbles introduced in the lower part move upward. Pumps are located in various intake offices and baffles facilitate recirculation of the slurry through consecutive flotation stages.

Screening-Cleaning

A comprehensive cleaning, pressure screening, flotation system designed by Voith (8) targets stickie contaminants by adding a dispersion stage followed by additional flotation and screening. The initial flotation stage is done in the absence of flotation chemicals and primarily removes ink particles. Fibers cleaned in the initial stages are treated chemically to activate contaminant surfaces prior to dispersion and final flotation and screening.

A multipurpose agitating drum provides recovered paper cleaning, screening, fiberizing, and pulping in a single unit. At least a portion of the drum has a polygonal-shaped wall that enables some of the contents to avoid the region of maximum agitation. Compaction, kneading, and pulsation are an integral part of this equipment and process developed by Voith (9) for processing recycled paper.

A patent for a novel hydrocyclone system (10) that combines stock cleaning and thickening was assigned to Thermo Black Clawson. This method employs forward cleaning for heavy contaminant removal and reverse through-flow cleaning for lightweight contaminants. Both subsystems operate at low consistency, but the consistency of the output of the through-flow cleaner is double that of the input, thereby serving as a stock thickener.

A truncated conical screen is the unique feature in Bolton's stock washer that combines washing, thickening, and classifying solids (11). High impact wash sprayer assemblies rotate about the conical screen and direct pulp stock onto the walls of the conical screen. Contaminants and fines pass through the screen and are discharged. Cleaned pulp exits through the apex of the screen for subscquent processing.

A stock thickener (12) especially useful for deinked pulp incorporates a ceramic surface to prevent accumulation and clogging by adhesive contaminants. Accumulated clean pulp is efficiently removed with a plastic scraper.

Bleaching-Color Removal

The approach for color removal and bleaching taken by Voith Sulzer (13) introduces intense mechanical dispersion of the pulp suspension containing bleach chemicals. The system is pressurized, includes oxygen, and is run above 40 °C for at least 10 minutes. The process can be followed by flotation and washing to optimize the whitening. Another bleaching method patented by Voith (14) uses pulp containing peroxide and oxygen at high consistency and temperature to achieve improved brightness and streak-free recycled paper.

Fluorescence originating in papermaking additives continues to be a problem for recyclers. Peracetic acid is used to reduce or eliminate fluorescent material in both recycled fibers and sludge in Air Liquide's recent patent (15). The acid can be introduced in one of several places during processing including thickening and dispersion stages or in the bleach tower.

Novo Nordisk has patented an improved method for bleaching lignin-containing pulps by activating peroxidases (16). This multipurpose invention describes methods of oxidizing a substrate with a source of hydrogen peroxide in the presence of a peroxidase enzyme, treating wastewater from pulp manufacturing, modifying lignin or lignin-containing material, and bleaching solubilized dyes.

A more environmentally sound bleach process is described in Solvay's (17) bleaching method. A biodegradable chelating compound based on aspartic acid is used under alkaline conditions to selectively remove transition metals that decompose peroxy

type bleaching agents. Alkaline earth metals that stabilize peroxide are retained in the pulp.

Nontraditional Deinking

Fuji Xerox Co. (18) targets toner removal with an image-peeling belt that has a meltable resin layer heated by halogen lamps in pressure rollers. Softened toner and inks are transferred to the belt and separated from paper surfaces onto an efficient collection roller.

Other nontraditional deinking patents appearing in this search include a patent (19) assigned to Toshiba that describes removal of colored inks from copy paper using a laser beam. Toner or ink is fragmented and peeled off individual sheets of paper by a pulsed laser.

Ricoh holds two related patents for ink removal from individual sheets of paper. One approach (20) introduces a liquid to reduce adhesion of the ink to the paper and subsequently transfers the image to a heated separating roll. The cleaned sheet is dried for reuse and the image is cleaned from the separating roll. Another (21) describes ink-peeling rollers with axially arranged protrusions corresponding to the image pattern and rollers without protrusions to flatten the paper, assuring a wrinkle-free regenerated sheet.

A third Ricoh patent is for preparing the paper for easy regeneration (22). The paper is coated with a thermoplastic or meltable material. An aqueous solution containing a surfactant is then impregnated into the paper creating a hydrophilic surface that enables the printed image to be peeled off when heat is applied.

Decopier Technologies' recent patent (23) also removes toners from a single sheet of paper with a nontoxic deinking solution comprised of a surfactant, swelling agent, and several water-soluble solvents. After softened toner is removed, the sheet is heated to vaporize the deinking solution and the paper is calendared to restore the sheet for immediate reuse.

RECYCLED PRODUCTS

Composites

Numerous products based on recovered paper or mill sludge and the specialized equipment and method of processing these materials also appeared in this search. One process is based on pelletizing recovered paper and waste synthetic thermoplastic resin articles (24). Recovered paper is heated with granulated resin and water is removed. The raw material is extruded and cut into pellets for molding synthetic resin articles. This process obviates the need for cross-linking agents, uses waste material, and generates no sludge or wastewater due to dry processing.

A low density cushioning material (25) made from recycled paper fibers is the topic of Oji Paper's claim. A water repellency or a hardening treatment is applied to paper fibers. Alow density product (0.02-0.30 g/cm^3) is obtained when this slurry is dewatered, dried, and molded. The resulting biodegradable product provides superior cushioning.

A different approach for using recovered paper and plastics is described in the Konishi patent (26) for a molded article. This process fiberizes and fluffs these starting materials, mixes and interlaces the fluffs, and finally thermopressure molds the mixture into a desired shape. The molded articles are sufficiently strong, economical to produce, and do not require fine classification of the starting waste materials,

Several Japanese patents that use waste paper have been recently awarded. One patent for a sleeping mat combines recovered paper (27) with reinforcing fibers. Corrugated sheets are made and laminated together with adhesive to form an inexpensive, multilayered mat. Another paper-based mat combines recovered paper with paper mill sludge (28). The recycled fiber slurry is dehydrated, crushed, and mixed with sludge. Reinforcement paper is impregnated with the dried sludge-fiber mixture and adhesive and is compression-cast to form a mat.

Hokkai Nisshoku's claim (29) deals with manufacture of a fiber base for turf seeds suitable for sodding a golf course or stadium using recovered paper kneaded with an adhesive. This biodegradable fiber mat can be made to a predetermined thickness and vegetative material can be added to the base layer. In another Japanese patent, pulverized scrap paper is mixed with a synthetic resin, adhesive, and fine ceramic powder to form a composite (30) suitable for growing plants. Holes are punched into the composite, and pesticides, plant regulators, and disinfectant are sprayed into it providing a superior growing medium for plants.

Absorbent granular material made from recycling rejects is described in Marcal Paper Mills' patent (31). Fillers and other solids are chemically flocked

and skimmed from the flotation clarifier, dewatered, and blended with dried rejects producing coarse particles with approximately 50% solids. These particles are highly absorbent, dust-free, and inexpensive to produce.

Buoyant, oleophilic, absorbent cellulosic granules capable of mopping up oil spills on water is the topic of a German patent assigned to the Bergakademie Freiberg (32). Moist fibrous material with resin and/or wax is comminuted, compressed into a plug, and pressurized to 60 bars at 75 to 100 °C. When pressure is released, the freshly exposed surfaces are pressure-coated with softened hydrophobic material. Subsequent processing removes moisture to form the lightweight, granular material. These granules could also be used for fibrous reinforcement in composites or for oily water filtration. Although not mentioned in this patent, wax-contaminated scrap paper could potentially be used as a starting material.

A method for manufacturing a fire-resistant, waterproof paperboard suitable as a building material from recycled paper is described in a Japanese patent (33). Paper is ground into a fine powder and mixed with a fire-resistant adhesive, an antimold agent, and a waterproofing agent before being pressed into a board under high pressure.

Recycled fiber is used to reinforce plastic molding for decorative purposes. The resulting composite has a stone-like appearance (34). Composites useful as a wood substitute can be made from hard-to-recycle, poly-coated papers and a thermoplastic resin (35). The paper is sheared until internal fibers are exposed and combined with the resin. The resulting product uses discarded containers and produces a useful product, such as pallets. Another U.S. patent (36) centers on lightweight, heavy duty, moisture resistant recyclable pallets made from corrugated fiberboard. Another type of fiberboard is manufactured using entangled recycled fiber mixed with adhesives as a surface layer and finely ground pieces for the core (37).

Other Paper Products

Recycled newspapers are converted into soft paper products in Kimberly Clark's recent patent (38). Newspapers are repulped with a surfactant at a low temperature (100 °F) to retain oil from the newsprint which softens the sanitary paper produced.

Oji Paper Co. has developed a coated paper for double-sided printing that uses deinked pulp as the base (39). Both sides of the base paper are coated with a pigment layer containing at least 3% titanium dioxide and an adhesive. Fluorescent whitening dye is incorporated into the deinked base paper. The processed paper can be printed on both sides and has good permanence.

Recycled fibers are used for a wide array of paper products ranging from paper towels, napkins, and juice cartons to currency and coffee filters. These products can be made with Hercules' process described in a recent patent (40). Wet strength resin comprising the reaction product of epihalohydrin with a polyamide is incorporated into slurried recycled fibers. The advantage of these products is their wet strength and easy repulpability without compromising wet and dry strength properties of the paper produced.

Fuel

Solid fuel is an alternate option for recovered paper use. A novel approach mixes scrap paper and plastics with other organic refuse, compressing the waste and fermenting the organic material with the aid of microorganisms (41). The residue is dried, crushed, and pelletized for use as a fuel. Nonflammable materials, such as iron, can be recovered.

Another invention describes furnace-ready fuel production from scrap paper and paper composite reprocessing (42). The method involves separating the residues into fractions of different sizes by screening. A large fraction with low chlorine content can be sorted out for use in the cement industry and fluidized bed furnaces.

CHEMICALS

Newly developed or modified deinking chemicals also appeared in this patent search. Several chemicals addressed special needs of recycled fibers, such as drainage enhancement and maintaining pulp brightness.

Buckman has developed a stabilized enzymatic preparation (43) useful for deinking newsprint. A polyamide oligomer is added to improve the stability and shelf life of liquid enzymes as well as improving the dispensability of the preparation at either high and low temperatures.

A chemical developed by Akzo Nobel (44) controls cellulosic fines and fillers in the process water of

closed mills. Effective for both improved pulp drainage and fines retention, this chemical permits a more efficient, faster papermaking operation, improves the dry strength of the resulting paper, and decreases the amount of additive required.

Morton International has patented a deinking collector chemical (45) comprised of a surfactant derived from lanolin. The collector is used in the neutral pH range.

An agglomeration chemical suitable for recycling waxed old corrugated containers is described in a patent assigned to Weyerhaeuser (46). The method uses screening and cleaning to remove agglomerated wax resulting in a pulp furnish suitable for use on a paper machine.

A deinking agent for recycling all types of recovered paper and printing techniques has been developed by Daiichi Kogyo Seiyaku Co. (47). The patent claims to achieve high quality recycled fiber with enhanced brightness and yield.

Other deinking chemicals appearing in the search are described in a patent (48) that replaces peroxide with sodium hypochlorite to prevent alkaline darkening and adds a solvent at low temperature to produce fluffy pulp inexpensively. Another (49) covers a polypropylene glycol diether and monoether composition for superior deinking without excess foaming. Kao Corp. has several new deinking chemicals (50, 51) especially useful in deinking newspapers, OMG, and telephone directories. Rhodia has been awarded a world patent (52) for a surfactant based on a polyalkoxylated terpene that is equally effective for ink removal by flotation or flotation-washing.

EFFICIENT RECYCLING

A U.S. patent (53) awarded to Avery Dennison describes a PSA product and associated process for improving contaminant removal by screening and cleaning. The tackified rubber-hot melt PSA deinking composition is added directly to a slurry of recovered printed paper or as a component of PSA-coated paper or label stock in the recovered paper. The deinking composition contains SB and SIS rubbers tackified with both resin- and rosin-based tackifiers.

Another patent (54) focuses on printing inks and claims improved deinkability. The anion-based aqueous composition contains finely dispersed pigments and a synthetic polymer binder suitable for flexo or intaglio printing. This special formulation facilitates ink removal by flotation.

The Eastman Chemical Co. was recently awarded a U.S. patent (55) for a water dispersible adhesive useful for making paper articles that can be recycled in either neutral or alkaline media. The adhesive is a hot melt adhesive with low molecular weight, branched copolyester containing a sulfomonomer, and it exhibits broad compatibility with a variety of adhesive additives.

A patent for a water-soluble release liner for PSAs has been awarded to Nippon Synthetic Chemical Ind. (56). This composition applied to the base paper is a blend of a polyvinyl alcohol-based resin and a fluorine-based compound. The release paper coating displays good barrier characteristics but dissolves during recycling, permitting recovery of the base paper,

Several patents are aimed at using recycling rejects as fully as possible. Ahlstrom has an environmentally sound paper recycling process and apparatus (57) for reclaiming both the fiber accepts and the fillers from the vortex cleaning rejects. Because the concentrated mineral fraction is cleaned and returned to the paper making process, the rejects discharged from the recycling process are significantly reduced. Coated broke is pulped and fed into a fractionating device to separate the coating flakes. The accepted fiber fraction is recycled back to the mixing tank of the paper machine. Mineral content is recovered separately, concentrated, and fed into the pigment dosing system. A related patent (58) extends the process to recycling filler-containing papers. Similarly, Clement's patent for minimizing waste products from recycling has been reissued (59). This process reclaims both the deinked fibers and the mineral fillers for pulp and board making. Solids-free water from the pulp slurry is used as the wash liquid for subsequent ink separation, thus closing the water circuits. Both chemical and thermomechanical treatments are included in this process.

A unique patent assigned to Penn State Research Foundation (60) describes a new class of proteins, expansins, which could offer an effective and environmentally benign method to recycle paper. Expansins are naturally occurring "wall-loosening" enzymes capable of disrupting hydrogen bonding. These proteins offer possibilities in the pulp and paper industry and, for our special interest, potential for recycling a wide range of papers. The patent covers isolation and application of expansins.

An enzyme, lactase, also plays an important role in Buchert, Kruus, and Viikari's innovation (61). The oxidative enzyme serves as an agent for modifying cellulose and can be used on all types of cellulosic fibers. Laccase can replace toxic or hazardous chemical oxidants. Paper produced from the patented process has improved technical properties, water retention value, and flexibility.

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Key to Abbreviations

CN = Canadian patent
DE = German patent
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JP = Japanese patent
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COVER DESIGN

Our cover features one of the works of Marilyn Sward. The performance/installation, "Directional Energy," was at the exhibition Glass Layers at Columbia College. It is comprised of 70' x 50' x 20' hand-formed paper, pigment, gold leaf and photographic emulsion. The *Paper Art* article "Floor Drains", starting on page 9, is written by Ms. Sward.