# Semiannual Patents Review July 2002 – December 2002

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**KEYWORDS**

Contaminant, Deinking chemical, Enzyme, Patent, Reclaimed fibers, Recycling, Review, Stickies, Wastepaper

## INTRODUCTION

This review summarizes patents related to paper recycling that were issued during the last six months of 2002. 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 postconsumer paper. Only brief summaries of individual inventions are included in this review. Because many of the patents are not readily available in English, much ofthe information was extracted from abstracts that were translated to English. For more complete information, readers will need to access the full text of a specific patent. Three excellent on-line sources for further review are located at:

- http://www.uspto.gov ;
- http://ep.espacenet.com.;
- http://www.mayallj.freeserve.co.uk.

As an indication of the amount of patent activity in the papermaking field, the 48 patents reviewed here were selected from 215 patent abstracts obtained from the initial search. Selection was based on what was considered to be the specific application of the patented technology to paper recycling as compared to papermaking in general.

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## PROCESSES AND EQUIPMENT

Enhanced removal of pressure sensitive adhesives (PSA) from wastepaper can be accomplished by omitting a surfactant during repulping and using fine slotted pressure screens after fiberization according to a Japanese patent by Matsushita et al. [1]. The inventors indicate that by omitting a surfactant, the size of PSA contaminants remains large and the contaminants can easily be removed by pressure screens with 0.10- to 0.20-mm slots. The ink particles can be removed after screening by steps such as kneading, dispersion, and flotation. The process is claimed to work on wastepaper containing up to 5% PSA. By being able to process such high PSA loading levels, lower-grade and lower-cost wastepaper can be utilized. The patent provides examples and results from laboratory experiments.

The separation of paper fibers from aluminum foil in composite laminated papers can be accomplished by means presented in a Chinese patent granted to Chen [2]. The method treats the foil laminate in an acid or alkali bath, which allows the aluminum component to go into solution. The paper fibers are then removed and the remaining solution is cooled and concentrated to recover the resulting aluminum salts.

The properties of a super-calendered paper produced from secondary fiber can be improved through precise refiner control strategy according to a patent by Jocham et al. [3]. The refiner rotational speed is controlled relative to the paper polish at the super calender. The pressure at the super calender is held below 100 $N/mm^2$ so as not to exceed a "permissible black satinizing value." Refining intensity is increased until the desired paper polish is obtained.

A method for repulping polymeric-coated wastepaper is disclosed in a U.S. patent by Bouchette et al. [4]. The method utilizes an inclined, pressurized drum-type pulper into which the wastepaper and water are added. Steam is

injected into the pulper along with pulping aids as needed. The material is tumbled in the drum by paddles and the fiber and coatings are separated. After an appropriate time, the steam is vented and a vacuum is applied to reduce the temperature and remove excess water. The material is then transferred to a high-solids vibrating coarse screen that separates the fiber from the contaminants. This step is followed by dilution of the fiber and processing through conventional pressure screens. It is claimed that during the process, the separated polymeric material tends to form into large balls and to collect detached inks. The resulting fibers are suitably cleaned for use as a feedstock for paper tissue and towel production. Results of tests performed with the pulping system are given in the patent.

A U.S. patent application by Freeland [5] describes techniques for the repulping of waxed old corrugated containers (OCC). The process entails blending waxed OCC with OCC at a ratio of 1/9 in the pulper and adding an agglomeration chemical to the pulper or to the resulting slurry. The chemical addition causes the removed wax to agglomerate so that it is easily removed by screening following pulping. The agglomeration chemicals, alone or in combination, can include sodium sulfate, potassium sulfate, lauryl sulfate, sodium silicate, sodium metasilicate, d-limonene, and propylene carbonate, but preferably are a mixture of sodium metasilicate and d-limonene. Ideally, sodium hydroxide is also used during pulping. The preferred pulping consistency is 8% at a temperature of 125°F. The resulting pulp is easily processed into paper because of its sufficiently low wax content. The process is also claimed to effectively remove Stickies. Results of several pulping trials using the chemicals and combinations, including a common household cleaner, are presented in the disclosure.

Lastly, a patent by Doelle and Matz [6] describes a novel high consistency pulper that removes sufficient quantities of contaminants from wastepaper so that a dispersion unit is not required. The desire of the inventors is to provide a higher fiber throughput compared with that from a system consisting of a standard pulper and a dispersion unit. The pulper is designed to operate between 15% and 40% consistency. The pulper consists of a rectangular tank with a plurality of conical augers perpendicular to the fiber flow through the tank. The conical augers are arranged such that the small end of one auger is adjacent to the large end of the next auger. Pulp bales are added at one end of the system where water sprays are located. As the bale is pulled into the pulper, the wastepaper and water are mixed to form a slurry. The slurry transferred between augers is transported toward the outlet end of the pulper. The continuous action of the slurry against itself and the pulper components fiberizes the pulp and removes contaminants from the wastepaper. At the outlet of the pulper, the slurry is passed over a vibratory screen to separate large contaminants from the fiber. The fiber is washed through the screen with water sprays for further preparation for papermaking.

## DEINKING AGENTS

Numerous patents address improved de-inking agents for use in paper recycling. The first of these, by Lafon [7], describes a new liquid de-inking agent that is a surfactant of polyalkoxylated terpenic origin. The new agent shows improved sheet brightness relative to a currently used de-inking composition. The new agent is added during wastepaper re-pulping. The agent is intended for recycling systems that use flotation or flotation and washing.

Many of the new de-inking compositions are patented in Japan. One of these, by Edo et al. [8], uses alkylene oxides combined with an amine compound. The composition is said to help in the removal of adhesives as well as ink from the recycled fiber. A patent granted to Goto et al. [9] describes combining a de-inking agent with a second compound to change the cloud point of the de-inking agent. The desired cloud point is 0°C to 10°C below the temperature of the flotation or washing step. Another patent, also to Goto et al. [10], addresses micronizing difficult-to-remove inks, such as toners, and dispersing the ink particles for removal in flotation or washing. The method utilizes surfactants whose surface tensions differ by 0 to -3 mN/m from the surface energy of the ink particles.

The de-inking of pulps can also be accomplished by the use of an agent containing a pentavalent phosphorous compound, with three different possible formulations, according to a patent by Tanaka et al. [11]. The agent is said to provide excellent ink coagulation and bubble adhesion during flotation while limiting the entrapment of fiber in the foam.

Brightness and ERIC values of secondary fiber can be improved with a recently developed styrene copolymer. The composition and process of using the material are described by Nguyen and Hengst in U.S. patents 6488808 [12] and 6482292 [13], respectively. The water-soluble styrene-methacrylic acid copolymer with a molecular weight of 1,000 to 100,000 is used at dosages of 0.01% to 5%, ideally with a surfactant. The copolymer is claimed to be more hydrophyllic and have higher surface activity than previous copolymers, thus keeping the released ink particles dispersed and preventing re-deposition on the fibers. After treatment with the copolymer and surfactant during pulping, the fiber can be processed by flotation or washing. Results of laboratory experiments are detailed in the patents and show the synergistic effects of the copolymer and surfactant.

Another de-inking agent, based on a water-soluble amphoteric acrylamide based polymer, is used in the flotation process according to a Japanese patent granted to Murano [14]. The agent is claimed to effectively remove inks, ash, and pitch from the recycled pulp. The compound has an average molecular weight of 500,000 to 5,000,000 and is comprised of defined percentages of quaternized methyl chloride, carboxylic-acid-containing monomers, and acrylamide units.

The last de-inking agent, from a Japanese patent by Edo et al. [15], is formulated to work under normal and abnormal conditions. The compound maintains adequate frothing and ink removal under conditions of excessively degraded wastepaper or high consistency in the flotation stage. The compound consists of alkyl groups, oxyethylene groups, and oxypropylene groups.

## ENZYMES

The development of more useful enzymes for recycling continues to be an active area. Numerous patents have been granted recently for modified enzymes and methods for producing them. The first of many patents in this area, granted to Patkar et al. [16], discusses lipolytic enzymes and the development of improved properties by methods of gene shuffling. The expressed proteins from the gene shuffling are screened for lipolytic enzymes with improved properties. The variants of the enzymes are envisioned for multiple uses, including pulp and paper. Another patent, by Baba et al. [17], describes endoglucanases that lack a cellulose-binding domain and are of Zygomycetes origin. The endoglucanases are said to be useful for de-inking pulps, increasing freeness, removing or altering fiber colors, and reducing fluff, A related patent, by the same inventors [18], describes the use of a cellulase preparation combined with a non-ionic surfactant. The cellulase preparation contains endoglucanases of transconjugant origin. The mixture can be used to improve ink removal from wastepaper when combined with other de-inking agents.

Two enzyme-related patents suggest using enzymes for color removal or bleaching of fibers. The first, by van Gastel et al. [19], describes phenol-oxidizing enzyme variants that are selected for neutral or high pH activity. The inventors suggest that the variants can be used for pulp bleaching. The patent gives detailed descriptions of the variants and method for acquiring them. The second patent, by Rodriguez et al. [20], discusses deriving enzymes with peroxidase activity from artichokes. The enzyme has many potential industrial uses, including the bleaching of recycled fiber.

The quantity, size, or tackiness of organic contaminants, or "stickies," can be reduced in recycled pulps and papers through the use of enzyme compositions according to Glover et al. in a recently granted U.S. patent [21]. The enzyme compositions contain esterases and/or lipases. The compositions are said to reduce the size of the contaminants, allowing the particles 20 stay more dispersed. In some cases, the contaminants are even converted to compounds that do not interfere with the papermaking process. The compositions can be applied at any of numerous points in the recycling process, but ideally are used after a flotation stage and prior to the headbox. The compositions can be applied at rates of 0.005 to 4 lb per ton of dry fiber and are most effective when in contact with the fiber for 30 to 120 minutes. The enzyme composition is said to work well on old newspaper (ONP), OCC, and MOW. The patent gives results of experiments conducted with. the enzyme compositions and shows the reduction of contaminants versus no treatment. The compositions that can be used include commercially available products.

Several U.S. patent applications have been published for the use of enzymes in paper recycling. Although they have not currently been granted patent status, they are presented to give the reader a sense of the activity in this area.

The first, by Neal and Page [22], provides a method for improved de-inking of starch-containing wastepaper. The method uses a starch-degrading enzyme, preferably an amylase, combined with a pectase lyase. The enzyme combinations are used with conventional de-inking chemicals and are selected for activity in the 6 to 10 pH range. The enzyme treatment can occur during or after wastepaper pulping. Ideal enzyme reaction times are 10 to 180 minutes prior to ink removal. Laboratory results show improved brightness and decreased residual ink. Another disclosure by the same inventors [23] reports the use of a deinking composition that is used during the repulping of wastepaper. Repulping is conducted at a pH range of 4 to 8.5. The composition consists of a lipase and a fatty acid ester. The process is especially useful for wastepaper containing old newspapers and old magazines because the pulping is conducted at neutral pH, preventing yellowing of the fibers. The fatty acid ester plays a key role in the process as it provides a substrate for the lipase. During the process, the lipase is converted to a fatty acid and a corresponding alcohol. This allows it to function as both a dispersant during ink removal and as an ink collector during subsequent flotation.

The third patent application, by Miettinen–Oinonen et al. [24], describes novel neutral cellulases identified by the inventors. A multitude of fungal organisms were screened for alkaline or neutral cellulases that would prove useful in the textile, detergent, and paper industries. The disclosure mentions three cellulases in particular and compositions made from these.

The last, by Yang et al. [25], discloses a composition for enzymatic de-inking. The composition contains microbial cellulases with high beta-glucosidase to filter paper unit activity. The patented method of using these enzymes was previously described in U.S. patent 6426200 by the same inventors.

## BLEACHING

Peroxide bleaching of recycled pulps could be improved with a new auxiliary bleaching compound. According to a Japanese patent by Okada et al. [26], the compound, a quaternary ammonium organic salt, inhibits the decomposition of the peroxide, giving higher brightness, while allowing the dosage reduction of peroxide and de-inking agents. Further, the chemical oxygen demand of wastewater is reduced as are scaling problems.

A Canadian patent granted to Wong [27] also addresses hydrogen peroxide bleaching of wastepaper. The method treats fiberized pulp with a chelant followed by peroxymonosulfuric acid prior to the application of hydrogen peroxide. The process is said to be effective enough to allow the use of OCC as furnish for making fine papers and can be performed under atmospheric pressure conditions.

## PAPERMAKING ADDITIVES

Improved retention and drainage in papermaking systems can be achieved with a new retention aid based on dendrimeric polymers, as described in a recent U.S. patent by Allen et al. [28]. The polymer was developed to help capture the many anionic materials in the papermaking slurries, including fines, fillers, resins, and Stickies, and thus reduce the loading of closed loop whitewater systems. The polymer can potentially be used with any type of pulp, including recycled. Dendrimeric polymers are a new class of highly branched macromolecules that extend from a central core, with end groups that can be easily tailored to specific end uses. The dendrimeric polymers in this invention are specifically developed so that the end groups have a positive charge at the pH range of the papermaking system. The polymer is ideally applied prior to the headbox but after all high turbulence operations, such as fan pumps or pressure screens. Results of laboratory and pilot-scale experiments conducted of conventional retention aids and two versions of the dendrimeric polymer are given in the patent. Under many conditions, the new polymer showed improved first pass retention and drainage rates along with lower whitewater turbidity.

Matshumita et al. [29] have developed a papermaking additive for enhancing the strength of both virgin and recycled paper. The additive, a water-soluble polysaccharide with an anion group and an acrylamide group, is claimed to adhere to the pulp better than previous additives. The increased retention improves burst properties while reducing the loading of whitewater and wastewater systems and also prevents paper machine contamination. The patent describes in detail the methods of making the additive and methods for using it.

## HOT MELTS AND COATINGS

Numerous methods have been used to produce moisture-proof corrugated board, but many produce materials that are not easily recycled. A recently granted U.S. patent by Muise and Neogi [30] claims methods to improve functionality and recyclability over previous methods. The new process fully encapsulates corrugated container blanks between two layers of thermoplastic film. The film is slightly larger than the blank and covers the edges of the blank. The film and blank are heated to seal the blank edges as well as to bond the film to the blank. The blank is passed through a die-cutting machine to remove excess film that covers any slits or cut-outs in the blank. The excess film is precisely cut such that the integrity of the sealed edges is not damaged. The invention claims that the film is more easily removed and separated during subsequent recycling processes than is wax or resin impregnated container board.

A recent U.S. patent granted to Kuno et al. [31] describes new hot-melt coatings for paper that are easily removed during wastepaper recycling and overcome some disadvantages of previous recyclable moisture-proofing coatings. The new compounds do not require extensive drying or curing as are required with latex and wax emulsions. They also do not have the tackiness associated with amorphous poly-alpha-olefin hot-melt compositions. The compositions can be produced with polypropylene, polyethylene, or ethylene-vinyl acetate in various proportions with a tackifier. The compositions can be adjusted to provide the correct hardness for the desired waterproofing properties, and the melt flow rate of the compositions can be adjusted to allow use in conventional coaters. Details of the formulations and tests conducted on the treated paper are presented.

Kaminaga et al. [32] have developed an easy-to-use silane compound for providing water resistance to paper. The patented water-soluble compound is added directly to paper stock with no need for pH adjustment to the paper. Because the chemical is finely distributed in the paper stock, the inventors claim that there is little interference with fiber bonding. Further, there is good retention so dosages can exceed 2% while paper machine fouling is limited. Test results presented in the patent show wet tensile strengths are improved compared to previous compounds.

The compound does not interfere with future recycling. Methods and formulations for manufacturing the compound are described.

Reducing the amount of silicone used in producing a release liner coating while maintaining adequate functionality is the goal of Kachi et al. in their Japanese patent [33]. The water-soluble formulation utilizes specific silicone particles that are smaller than 400 nm and that make up only 5% ofthe emulsion. Because the particles are small and because of surface energy differences between the silicone and the carrier, the silicone tends to stay near the top of the coating when applied to the paper substrate. Many possible chemical formulations are presented in this patent. Since small amounts of silicone are used in the coating, it is inferred that recycling problems will be reduced.

## TONERS

One recent Japanese patent by Matsuoka et al. [34] describes a resin system for toners that allows for easier toner removal during conventional wastepaper recycling. The resin system contains a poly alpha-hydroxycarboxylic acid that is more easily broken down during repulping with an alkali than is the more commonly used styrene binder. The new resin system is claimed to have a longer shelf life and better application properties than previous materials used to replace the styrene binder. The patent provides the chemical and processing details for producing the resin system.

A unique approach to recycling copy paper is to remove the toner without disrupting the paper substrate, which could be done at the point of use. A recent patent granted to Kimura and Matsui [35] details an invention to achieve this goal. The process utilizes paper that is surface treated with a carboxymethyl cellulose polymer. The printed image is applied to this surface using conventional xerography techniques. The paper to be recycled is fed to a machine that applies less than 1 g of water to an A4 size printed surface. The paper is then heated to loosen the toner layer, which is subsequently peeled from the paper by a belt or drum apparatus. The cleaned paper can be reused.

Two related patents assigned to Minolta Co. Ltd. attempt to achieve this same goal. A patent by Yoshie and Yamaoka [36] describes possible formulations that can be applied to sheet substrates, including paper. The coatings are selected to provide swelling of the printed surface when exposed to water, thus loosening the toner. The patent by Yoshida [37] describes a toner removal apparatus to be used with specially coated substrates.

## RECYCLED PRODUCTS

An apparatus and process for remanufacturing and reusing paper winding cores is addressed in a U.S. patent granted to Ostroff [38]. Currently, reuse of paper cores is limited by the out-of-roundness that develops in the core during its initial use, forcing the core to be discarded or repulped into fiber. In the first step of the patented process, the steel end caps are removed from the recovered cores and the crimped ends are cut off. The ends of the core are then machined to form either a male or female socket joint, which can be mated to a second paper core with similar joints. Two or more cores can be joined end-to-end by gluing the socket joints. Next, the joined cores are ground to a constant outside diameter, which is slightly smaller than the industry standard. A properly sized sheet of paper is then glued and wrapped around the core to bring it to the proper outside diameter. The resulting core is subsequently cut to the proper length.

A U.S. patent application by Boney [39] is also concerned with the disposal of paper winding cores. This disclosure shows a method and apparatus for converting used paper winding cores into end caps for use on new paper winding rolls. The final product can be used in place of wood composite end caps that are currently used. Used cores are shredded to a defined size and then compression molded into the end caps. No additional adhesive is required during the molding process due to the large amount of adhesive originally used in the core. Typically, the only required water is already present in the previously shredded core.

## WASTEWATER TREATMENT AND SLUDGE

One ofthe many problems with the disposal of paper sludge is the large quantity of water remaining with the waste. A recent patent granted to Ohara and Kubota [40] describes a flocculant that dehydrates the sludge, allowing more effective dewatering and thus lowering disposal costs. The patent describes the dehydrating agent, a saltwater solution of a first-class amino group polymer, and a method of manufacturing it. Another recent patent, by Weir et al. [41], pertains to improving the dewatering of sludges through the sequential use of linear polymer and a structured polymer. The method claims faster dewatering and improved solids retention.

Disinfection and improved dewatering of waste-activated sludge can be achieved by the application of a pulsed high voltage electric field, according to a U.S. patent by Held and Chauhan [42]. The waste-activated sludge is initially dewatered by conventional means and then passed through an electroprorating station. This in-line tubular device contains electrodes powered by a high voltage source that generates an electric field of 15 to 100 KV/cm. The pulsed

electric field disrupts the cellular membrane and allows the internal fluids to be released from the cellular material. In a second filtering step. the free liquids and dissolved organic materials are removed from the bulk of the sludge and returned to the primary digestion tank as additional nutrients. The second device, a filtration module, utilizes a porous tapered cone inside a porous cylinder and is arranged to produce a decreasing cross-sectional area that squeezes the sludge as it moves through the filter. The system is claimed to remove an additional 25% of water from the sludge, greatly decreasing the disposal cost.

Another recently patented wastewater treatment system, invented by Mimura et al. [43], also uses pulsed high voltage electric fields. The invention is intended to treat high molecular weight and aromatic compounds rapidly. The wastewater is aerated with oxygen prior to passing through the electrodes of the treatment cell. It is claimed that the electric field converts the dissolved oxygen to hydrogen peroxide or hydroxy radicals, which then cleave the aromatic and high molecular weight pollutants. Details of experiments conducted with the electrocatalysis process are given in the patent.

A new composition for maintaining the proper nitrogen and phosphorous levels in wastewater biological treatment ponds has been patented by Hahn [44]. The composition, meant to replace currently used anhydrous ammonia and phosphoric acid, is a solution of urea ammonium nitrate, ammonium phosphate, free urea, and water. The solution is claimed to be nontoxic and nonhazardous. Also, the solution is not harmful to the bacteria. Successful results from trials conducted at a paper mill with the solution are presented in the patent.

## MISCELLANEOUS

Kuzuu [45] has patented the novel use of wallpaper to indicate room atmospheric conditions. The wallpaper would contain compounds that change color with temperature or humidity and quickly and easily display the room conditions. It is also suggested that other color-changing compounds could be used to detect various gases, such as carbon dioxide and carbon monoxide. No suggested compounds are given.

Polyethylene-coated wastepaper can be disintegrated and mixed with bitumen-containing materials to improve product performance [46]. The shredded material acts as a stabilizer to enhance the deformation resistance, needle penetration, and run-off value of the bitumen product. A somewhat related patent by Gamza [47] describes shredding wastepaper and polyethylene, mixing them, and hot pressing to form thermal insulation panels.

A rather unique use for industrial wastes, including paper mill sludge, is the production of artificial fishing reefs. A U.S. patented concept by Kim [48] suggests combining the waste materials with a plasticizer, such as clay. and a mineralizer, such as limestone. This mixture is pug-milled and then pressure molded into large objects. The resulting object is dried or calcined at 700°C to 1,000°C and subsequently sintered at 1,000°C to 1,300°C. The process is claimed to make the materials environmentally inert. The resulting reef material is also claimed to be more resistant to the seawater environment than are currently used concrete or steel materials.

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