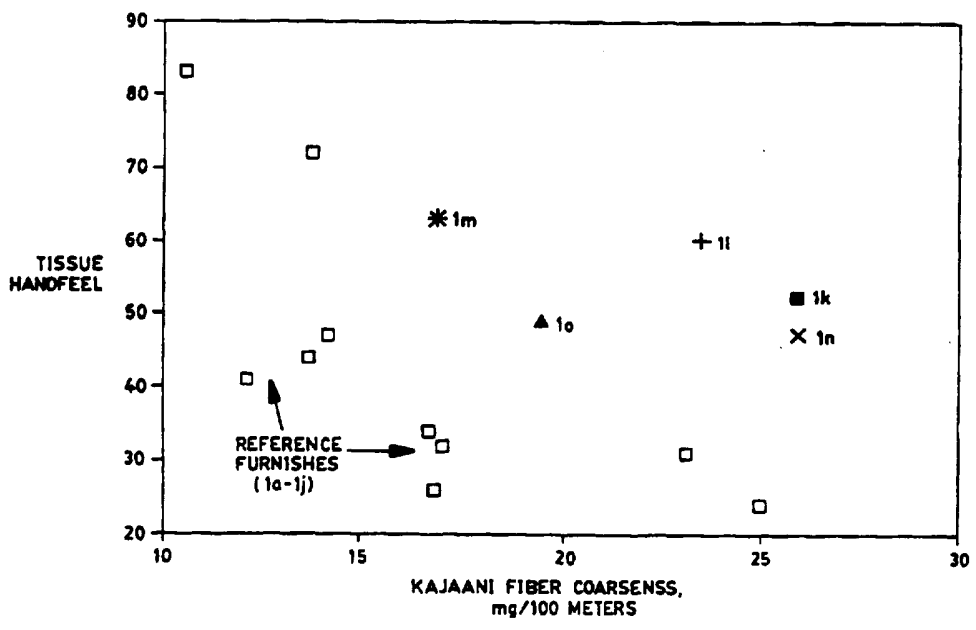




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(54) Title: PRODUCTION OF SOFT PAPER PRODUCTS FROM HIGH AND LOW COARSENESS FIBERS



(57) Abstract
Paper products such as bathroom tissue, facial tissue, napkins and paper towels are made from high coarseness fibers such as recycled newsprint fibers, CTMP, TMP, and groundwood. These products exhibit premium level qualities in terms of softness, bulk, and flexibility. The novel process selectively treats these high coarseness fibers in a way that makes the fibers feel softer and enhances papermachine operation with this type of furnish, thereby permitting higher product quality levels than previously possible with high coarseness fibers.

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PRODUCTION OF SOFT PAPER PRODUCTS
FROM HIGH AND LOW COARSENESS FIBERS

CROSS REFERENCE TO RELATED APPLICATION:

This is a continuation-in-part of U.S. Patent
5 application Serial Number 08/268,232, filed on June 29, 1994.

BACKGROUND OF THE INVENTION

For each paper making process a correlation exists
between fiber coarseness and product quality in terms of
product softness or handfeel. Expensive high quality fibers
10 such as bleached northern kraft softwood fibers are fine,
flexible, and produce high quality tissue products. In
contrast, mechanical pulping of softwoods produces coarse,
stiff fibers typically used in making newsprint. Newspapers
contain a preponderance of coarse, high yield fibers, typically
15 stone groundwood (SGW), thermomechanical pulp (TMP), and/or
chemithermomechanical pulp (CTMP) fibers. Such coarse
newsprint fibers are usually highly refined to cause fractures
and fibrillations which aid in imparting strength to the
resulting newsprint. Such refining changes the "freeness" of
20 the coarse fibers from "high" freeness fibers to "low" freeness
fibers. If such refined, coarse mechanical fibers are used in
a tissue making process the resulting sheet is not soft, and
therefore has poor tissue properties. A recent thorough
discussion of the relationship between tissue softness and
25 fiber coarseness is contained in Canadian Patent No. 2,076,615.

Attempts to produce soft tissue or towel type sanitary paper products from a majority of high yield fibers such as CTMP, TMP or SGW pulp have not been successful. Likewise, producing soft tissue and towel products by recycling
5 old newspapers has not been very successful partially because the predominant fiber in newsprint or in old newspapers are low freeness, coarse, high yield fibers.

Other complicating factors in producing soft tissue and towel products by recycling old newspapers are difficulties
10 with the paper machine operation caused by poor drainage associated with low freeness fibers, and problems caused by high amounts of fines and other substances, which separate from the fibers and accumulate in the paper machine water system (white water). These materials make it difficult to crepe the
15 tissue sheet from the Yankee drying cylinder, and therefore necessitate operating the paper machine at conditions which do not promote maximum softness.

The present invention solves these difficulties by enzymatically modifying the fibers and by adding oils of the
20 type used in newspaper ink to the high coarseness fibers, thereby softening the fibers and giving them release properties which aid in the creping step on the paper machine. For recycled newsprint, the fibers are enzymatically modified, and oils of the types used in newsprint ink are added, and a
25 portion of the printing oils in the fibers are retained, thereby softening the fibers, and making the fibers more flexible which aids in the creping step on the paper machine. Consequently, previously unachievable levels of tissue and towel softness are possible with the present invention using
30 high coarseness fibers or recycled newsprint fibers.

Methods to improve softness of tissue products using low coarseness fibers by lotionizing the surface of the tissue product have been developed. These methods, however, are applied to the tissue product prior to it being converted into
35 the final product. It would be desirable to provide a method

of achieving these softness gains without the cost and complexity of the current methods.

Conventional recycling of old newspapers to obtain fibers comparable to the type of fibers used to originally make the newsprint is known in the art as "deinking" and typically involves pulping, washing (usually with surfactants), screening, centrifugal cleaning, solubilizing insoluble contaminants (usually by strong caustic treatments), washing and bleaching of the fibers to counteract the yellowing effects of caustic treatments.

The first step in conventional recycling of old newspapers is to separate the paper into individual fibers in water to form a pulp slurry. Surfactants and caustic are added to facilitate the solubilization and separation of contaminants from the fibers. This is followed by removing inks and contaminants from the fibers by a combination of various process steps such as screening, centrifugal cleaning, washing, flotation and the like. The screening and centrifugal cleaning steps remove large contaminants such as paper clips, staples, plastics, etc. The primary purpose of washing and flotation steps is to suspend contaminants in the water and to remove the contaminants from the fibers. When caustic is used to facilitate contaminant removal, some yellowing of the fibers unfortunately occurs due to the caustic treatment. After or during caustic treatment and washing, the fibers are usually bleached (e.g. - with hydrogen peroxide) to counteract the yellowing effect of caustic or to produce better fibers having higher brightness than the fibers in the original waste paper. Cleaned, decontaminated, and bleached fibers are usually blended with virgin fibers and then used in a paper making process for which the fibers properties are suitable. Because the starting fibers are newsprint type fibers, i.e., coarse, low freeness and low brightness fibers, such recycled fibers are most often reused for making blank newsprint. They are generally not suitable because of their high coarseness and low freeness for making soft tissue products unless blended with

a majority of higher quality fibers such as bleached northern softwood kraft pulp.

Conventional pulping of used newspaper to obtain recycled newsprint fiber is usually done in a high attrition pulper at a consistency of 4-8% and at 90°F-160°F for 20 minutes to 60 minutes, depending on the exact type of waste paper being processed. Caustic soda or other alkaline substances such as sodium silicate are commonly used to raise the pH of the pulp slurry to pH 9-10 to aid in separating fibers (defibering) and also to loosen the inks and separate dirt from the fiber. At an alkaline pH vegetable oils in the inks are saponified while mineral oils are emulsified by the combination of alkaline pH, soaps, and surfactants, all of which enhance the removal of oils during washing. A surfactant deinking aid (for higher pH ranges) is usually added to further help separate inks from fiber.

The caustic step in recycling processes of old newsprint to obtain well cleaned quality fibers causes swelling of the fibers, and usually solubilizes many components. In addition to saponifying vegetable based printing oils, caustic also saponifies natural organic acids typically present in old newspapers to produce the corresponding soaps of the saponifiable materials. The saponified vegetable oils and organic acids so formed aid in removal of other contaminants from the fibers, such as non-saponifiable printing oils (mineral oil). These substances are subsequently removed from the fibers by washing and/or flotation after the caustic treatment.

A major recycler of old newspapers, Garden State Paper, in recent journal articles, one entitled "The Big "D": Getting Rid of the Ink in Recycled Fiber appearing in the journal Paper Age, 1991 Recycling Annual, at pages 23 and 50 and the other article entitled "Recycling From the Newsprint Perspective, at pages 9, 12 and 13 of the same 1991 Recycling Annual, (Paper Age, 1991 Recycling Annual) describes its newsprint recycling and deinking process as cleaning and

screening followed by a series of 3 washings facilitated by the addition of chemicals to emulsify the printing oils and resins. Again the aim of this process is to remove printing ink constituents including oils as completely as possible. This
5 is especially important because the recycled newsprint fiber is made into blank newsprint paper which would not have adequate brightness or strength without removing the ink constituents.

A common component of deinking systems for newspaper
10 waste involves separating ink from the fibers and removing the ink typically through washing and flotation steps. While conventional alkaline deinking chemicals are very effective in such deinking, they are known to have the disadvantage of lowering brightness. Recent research has been directed to
15 avoiding alkaline deinking chemicals in deinking systems.

Recent developments in wastepaper deinking (U.K. Patent Application 2,231,595 published 21/11/90 entitled "Deinking Water Printed Paper Using Enzymes" and a North Carolina State University publication entitled "Enzymatic
20 Deinking of Flexographic Printed Newsprint: Black and Colored Inks") deal with the use of enzymes to aid in the detachment and removal of inks from the fibers. These processes describe the use of enzymes such as cellulase, pectinase, xylanase, and hemicellulases to facilitate ink removal without the negative
25 effects of caustic treatment on brightness along with the use of flotation to remove the agglomerated ink particles. Since printing oils are lighter than water, they are readily removed by flotation treatment particularly in view of the chemicals added to aid in separation. While enzymes are used, this
30 thorough removal of ink components is counter to the objective of the present invention which retains the oils for tissue softness. A paper presented at the Fifth International Conference on Biotechnology from May 27 to May 30, 1992 in Kyoto Japan entitled "Enzyme Deinking of Newsprint Waste" by
35 John A. Heitmann, Thomas W. Joyce and D.Y. Prasad described research occurring at the department of Wood and Paper Science,

North Carolina State University, Raleigh, North Carolina. That article described the use of acidic flotation deinking systems in which the only chemicals used were enzymes, calcium chloride and a surfactant. The enzymes were a preparation containing
5 both cellulase and hemicellulose. Increases in freeness and brightness were noted. However, the important distinction is that the acidic flotation deinking system described removes ink along with its associated oils which is contrary to the present invention.

10 More recently, high consistency pulping (13-18%) has been utilized for recycling old newspapers. This type of pulping technology utilizes the additional effect of rubbing/kneading between the fibers/papers at higher consistency to defiber and assist in separating inks from the
15 fibers. Generally the pulping temperature, time, and chemical additions are the same as lower consistency pulping described above.

One aspect of the present invention avoids conventional deinking but instead retains a significant
20 component of the ink, i.e., the printing ink oils. The present invention is based on the discovery that if the oily component of ink is not removed from coarse fibers in old newsprint, surprisingly high quality, soft tissue products can be produced. To accomplish this task, a formulation of enzymes
25 is utilized to loosen a limited amount of the ink constituents for removal and/or redistribution on the fibers. In addition, by avoiding saponification conditions, e.g. alkaline saponification of fatty acid oils such as vegetable oils, and fiber components such as hemicellulose are not allowed to leach
30 out of the fibers into the paper machine water system and cause difficulties with the creping operation.

SUMMARY OF THE INVENTION:

The present invention provides a method for producing soft tissue products by treating the fiber prior to sheet
35 formation. It has been known that fiber coarseness is a contributing factor in the production of soft tissue products.

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The present invention provides a method of modifying high coarseness and low coarseness fibers to improve the softness potential of the fibers.

The softness potential of virgin chemical fibers and recycled fibers can be increased by the addition of vegetable oils, e.g. soya, linseed, castor, safflower, olive, peanut or their fatty acid ester derivatives; mineral oils or lanolin oils and their ethoxylated, acetylated or ester derivatives. The treated fibers are then subjected to appropriated enzyme and disperser treatment.

Virgin, coarse, high yield fibers (e.g. stone groundwood, thermomechanical and chemithermomechanical pulps) can be made suitable for producing soft tissue type products by addition of oils typically found in newspaper inks and subjecting the intentionally oil treated virgin fibers to appropriate enzyme treatment. Novel fibers and sanitary paper products containing a majority of enzyme treated coarse, high yield type fibers having oily materials are produced according to the present invention. Enzyme treatment utilizes one or more enzymes selected from the group consisting of cellulase, hemicellulose, such as xylanase, and lipase.

The method of making sanitary paper products from virgin or recycled cellulosic fibers disclosed herein comprises:

(a) pulping the cellulosic fibers in water with agitation to produce a pulp slurry at a consistency of about 3% to about 18% and a pH below about 8.0;

(b) adding to the slurry an enzyme selected from the group consisting of cellulase, hemicellulose and lipase and maintaining the pulp slurry at a temperature above about 100°F for at least 15 minutes;

(c) dewatering the pulp slurry to a consistency from about 25% to about 35%;

(d) crumbing the dewatered pulp, thereby producing crumbed fibers;

(e) passing the crumbed fibers through a fiber

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disperser while maintaining the fibers at a temperature of about 180°F and mixing either a vegetable, mineral or lanolin oil or their derivatives with the fibers; and

(f) using the enzyme treated fibers as a source of
5 fibers in a papermaking process to produce sanitary paper products.

The novel sanitary paper product is made of cellulosic fibers and has a basis weight of between 7 pounds per ream and 40 pounds per ream, a Normalized Tensile Strength
10 (metric) of between 5.0 and 20.0, and containing from about 0.2 to 5.0% of an oil selected from the group consisting of vegetable, mineral or lanolin oils or their respective derivatives.

The novel method of modifying cellulosic fibers
15 comprises modifying cellulosic fiber to improve their tissue and towel making properties comprising:

(a) adding about 0.2% to about 5.0% of a vegetable, mineral or lanolin or their respective derivatives to the cellulosic fibers at a consistency of about 25% or greater,
20 crumbing the fibers, and passing the crumbed fibers through a fiber disperser while maintaining the fibers at a temperature of about 180°F;

(b) adding to the furnish at a temperature below 140°F, an enzyme selected from the group consisting of
25 cellulase, hemicellulose and lipase and maintaining the pulp in contact with the enzyme and surfactant at a consistency of between about 3% to about 18% and at a temperature of about 100°F and about 140°F for approxiametly 15 minutes.

The improved cellulosic fiber for making sanitary
30 paper products disclosed herein comprises an enzyme modified cellulosic fiber containing bewtween about 0.2% and about 5.0% of an oil selected from the group consisting of a vegetable, mineral or lanolin or their respective derivatives.

BRIEF DESCRIPTION OF THE DRAWINGS

35 Figure 1 graphically shows the relationship between fiber coarseness and tissue softness conventionally obtained

by lightweight, dry crepe tissue making processes and the superior results obtained by the present invention.

Figure 2 is a tabular presentation of the experimental results of examples 1 and 2. In addition, Figure 2 incorporates the experimental results of U.S. Patent application Serial Number 08/268,232, filed on June 29, 1994. Figure 3 is a tabular presentation of the difference in response between the use of a castor oil and a mineral oil. Figure 4 is a tabular presentation of a control sample with and without treatment.

DESCRIPTION OF THE INVENTION AND PREFERRED EMBODIMENT

The present invention is based upon the discovery that high yield type cellulosic fibers (i.e. fibers produced by predominantly mechanical separation of wood fibers and typically containing at least 80% by weight of the source material) can produce very soft tissue type products having product qualities comparable to tissue products made from expensive bleached northern softwood kraft fibers. These cellulosic fibers include high coarseness fibers having a coarseness of greater than 17 mg/100 meters, and low coarseness fibers having a coarseness of less than 18 mg/100 meters. Particularly, soft tissue type products can be produced from these types of cellulosic fibers by adding an oil selected from a group consisting of mineral oils, vegetable oils, lanolin oils, and cosmetic type oils.

Prior to adding these oils the cellulosic fibers are subjected to an enzyme treatment. Soft paper products are then made with the oil treated and enzyme modified fibers. It is critical in the practice of the present invention that a sufficient quantity of oils be on or in the fibers prior to making tissue or other types of sanitary paper products (e.g. towels, napkins and facial tissues) from such fibers.

Soft tissue type products can also be produced from old newspapers (ONP) by retaining certain oil materials typically found in used newspapers, subjecting the used newspaper fibers containing such oils to an enzyme treatment

and making paper with such oil and enzyme modified fibers. It is critical in the practice of the present invention that a sufficient quantity of oils typically found in used newspapers be on or in the fibers prior to making tissue or other types of sanitary paper products (e.g. towels, napkins and facial tissues) from such fibers. Furthermore, if the oil is removed during deinking or not present as with virgin fibers, the oil can be added to the fibers and the fibers containing such oils can then be subjected to an enzyme treatment prior to making sanitary paper products from the oiled and enzyme modified fibers to obtain the benefits of the present invention.

Vegetable oils and mineral oils are typically used in newspaper printing inks and are found in used newspapers, generally as components of the printing inks. In order to retain oil components of used newspapers, conventional repulping and deinking process must be modified. The preferred modification of conventional deinking is to eliminate saponification conditions in which vegetable type oils (or any oil containing an ester group) are converted into soaps. However, if the oils, are removed during deinking, they can be replaced with an oil selected from the group consisting of vegetable, mineral or lanolin oils or their derivatives prior to enzyme treatment.

In one embodiment, the method of the present invention employs virgin cellulosic fibers as a starting material. The virgin cellulosic fibers are pulped to produce a slurry having a consistency between about 3% to about 18% and a pH below about 8. The slurry is then treated with a surfactant and an enzyme such as a cellulase, xylanase or lipase or a combination of such enzymes at a temperature above about 100°F. for at least 15 minutes. After the enzyme is added, the slurry is dewatered to a consistency from about 25% to about 35%. The dewatered slurry is then crumbed, thereby producing a crumbed fiber. The crumbed fiber is then passed through a fiber disperser and mixed with an oil selected from the group consisting of vegetable, mineral or lanolin oils or

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their derivatives up to the point where the final paper product will still be absorbent and aesthetically appealing. Preferably, about 0.2% to about 5.0% of the selected oil is mixed while maintaining the fibers at a temperature of about 5 180°F. The slurried enzyme treated pulp is then used as a source in conventional sanitary paper manufacturing process, preferably a tissue papermaking process. If it is necessary to conduct a screening, cleaning, flotation and/or some washing of the pulp slurry prior to using it as a furnish for making 10 sanitary paper products (e.g. tissue, towel, facial tissues or napkins), it is important that a substantial quantity of the oily contaminants be retained on the pulp after such screening, cleaning, flotation and/or washing steps, or else replaced prior to enzyme treatment and papermaking to achieve the 15 desired softness and aesthetic levels.

The pulping process of the present invention when using either virgin high coarse or low coarse fibers preferably involves pulping the fibers at 6-9% consistency and an elevated temperature, preferably about 120°F-180°F. The fibers are 20 pulped continuously for a time sufficient to defiber the pulp and prepare it for reaction with the enzyme and/or surfactant mixture. Preferably the fiber is pulped for about 15 to 60 minutes. The slurry is then transferred to a holding chest/blend where the pH is adjusted to a temperature and pH 25 level sufficient for reaction with the enzyme and/or surfactant mixture. The preferred temperature conditions is about 120°F-140°F with a pH of between 4 and 7. Surfactant and enzymes are then added to the pulp slurry and allowed to react with the fibers for a reaction period of about 15 minutes to about 30 30 minutes to complete the treatment. The pH is then adjusted to about 7, and the pulp is dewatered through a press, such as a commercially available Andritz press, to a consistency about between 25% to about 35%. The dewatered pulp is then crumbed using a commercially available crumbing apparatus, such as that 35 from Scott Equipment Co., New Prague, MN, to produce a crumbed fiber. The crumbed fiber is then passed through a fiber

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dispenser such as a commercially available Micar, manufactured by The Black Clawson Company, Middletown, Ohio, and mixed with oil selected from the group consisting of vegetable, mineral or lanolin oils or their derivatives while maintaining the fibers at a temperature of about 180°F.

The micar is situated so that it is capable of (1) injecting steam so that the fiber maintains a temperature of about 180°F., (2) and so that the selected oil can be mixed with the crumbed fibers.

The amount of oil added and mixed will vary from about 0.2% to about 5.0% depending on the weight of the dry fiber. The fiber are the maintained at a temperature sufficient to allow the oils to be retained in or on the fiber. The fiber temperature is preferably maintained at about 180°F. by injecting steam when the selected oil is mixed with the fibers.

Additional screening is unnecessary with the virgin fibers, although screening and/or centrifugal cleaning may be practiced to remove any large contaminants to protect the papermachine. Optionally, limited washing of the enzyme treated and oil containing pulp can be done on the papermachine by using the unwashed pulp in a papermaking furnish on a papermachine.

Preferably the slurry and enzyme treatment steps when using virgin cellulosic fibers or old newspapers is the same. This treatment is conducted in several stages beginning with slurring the cellulosic fibers or newsprint at a consistency of between about 3% and 18% with or without surfactant, and preferably at a temperature of the pulp slurry between about 100°F and 180°F and maintaining the elevated temperature for at least about 15 minutes. This is followed by adjusting the pH and reducing the temperature of the pulp slurry to a temperature and pH suitable for maintaining active enzyme conditions. Preferred enzyme treatment conditions are a pH of 4 to 7 and a temperature below about 140°F and preferably above about 100°F. If pulping of the virgin coarse fibers or newspapers is performed under conditions also suitable for

enzyme treatment, pulping and enzyme treatment steps can be combined.

When pulping and enzyme treatment are combined into a single step, the enzyme with or without the addition of a
5 surfactant, can be added to the water either prior to or after addition of the virgin fibers or newspapers for pulping. Optionally, a surfactant of the type typically used in contaminant removal in newsprint recycling processes is added to the pulp slurry. One or more enzymes can be used. Enzymes
10 are preferably selected from the group consisting of cellulase, xylanase and lipase. The pulp is maintained in contact with the enzyme for at least about 15 minutes and preferably about 30 minutes.

When using virgin high coarse or low coarse fibers, newsprint, or old newspapers, a critical component in the above
15 process sequence is having oil selected from the group consisting of vegetable, mineral or lanolin oils or their derivatives in contact with the enzyme treated fibers and retained with (on or in) the fibers during papermaking.
20 Without being bound thereby, our theory by which virgin high coarse and low coarse fibers become very suitable for making soft tissue type sanitary paper products is that some interaction between the fibers, oils and enzymes occurs that is enhanced by the presence of a surfactant. This interaction
25 synergistically improves the tissue making properties of the coarse fibers.

Other oils that may be employed in accordance with the present invention include vegetable oils, e.g. soya, linseed, castor, safflower, olive, peanut or their fatty acid
30 ester derivatives; mineral oils or lanolin oils and their ethoxylated, acetylated or ester derivatives.

DYES

Recycled newsprint fibers of the present invention retain inky contaminants and are therefore a light gray color.
35 Tissue products made with a majority of such fibers are preferably dyed to a more pleasant color. The dyes useful in

this invention must be water soluble and because of the difficulty of uniformity dyeing oily contaminated fibers, the dyes should be substantive to cellulosic fibers. They should also be cationic, i.e. they will form positively - charged colored cations when dissociated in water. These dyes are particularly well suited for dyeing mechanical and unbleached chemical pulps. Such pulp fibers contain a significant number of acid groups, with which the positively-charged cations can react by salt formation. These dyes can be selected from among the basic dyes, a group well known from prior art, in which the basic group is an integral part of the chromophore, or from the newer class of cationic direct dyes, in which the basic group lies outside of the molecules resonance system. The dye is preferably added in amounts ranging from 0.01% to 3%, most usefully, at 0.05 to 0.5% of the weight of air dry fiber.

These dyes can be applied at any normal papermaking pH, either acidic or neutral. Their excellent affinity for unbleached fibers allows them to be added to the papermaking system as late as the inlet to the fan pump, but a longer residence time, e.g., introduction at the suction side of the machine chest transfer pump would be preferred. In either case a thick stock location with good mixing is desirable.

ENZYMES

Suitable enzymes for use in the present invention should be selected from the group consisting of cellulase, hemicellulose (e.g. xylanase), or lipase enzymes and preferably one of each type is used in combination. Each type of enzyme functionally targets different components of used newspaper fibers and/or contaminants usually associated with such fibers. Cellulase enzymes contribute to ink removal by attacking the cellulose component of fibers in the proximity of ink. Xylanase and other hemicellulases attack hemicellulose components of fibers for brightness enhancement while lipase attacks resins in the fibers and in the ink formulations. When all three types of enzymes are used together a synergism results, that achieves better ink removal as well as

eliminating so called "stickies". Stickies are a well known contaminant in used paper resulting from adhesives, pressure sensitive labels, etc. and are known to cause papermachine runability problems. A mixture is preferably selected of
5 enzymes that will attack the printed waste paper in a way that enhances tissue softness and modifies contaminants so that they do not hurt papermachine operation. Also, enzyme treated pulp in accordance with the present invention will improve paper machine running ability and produce a superior product at low
10 costs.

Hemicellulose is a general term describing various types of enzymes each degrading specific types of compounds commonly known as hemicellulose and found in wood and other plant materials. Xylanase is the preferred hemicellulose
15 enzyme because it is active toward the xylan, a common type of hemicellulose. The constituents of hemicellulose differ from plant to plant. The most abundant of the wood hemicelluloses are the xylans, which are polymers of 1,4-linked β -D-xylopyranose units some of which bear short side chains such
20 as 1,3-linked α -l-arabinofuranose units or esterified 1,2-linked α -d-glucuronic acid units. Also important, particularly in softwoods, are 1,4- β -D-glucomannans with randomly distributed glucose and mannose units, bearing side chains such as 1,6-linked α -D-galactopyranose units. Hemicellulose differs
25 from cellulose in three important respects. In the first place they contain several different sugar units whereas cellulose contains only 1,4- β -D-glucopyranose units. Secondly they exhibit a considerable degree of chain branching, whereas cellulose is a strictly linear polymer. Thirdly, the degree
30 of polymerization of native cellulose is ten to one hundred times greater than that of most hemicelluloses. The term "hemicellulose" refers to any specific enzyme class that reacts with a specific hemicellulose and as such, hemicellulose is not a specific enzyme class but a generic term of art for a group
35 of enzyme classes. Xylanase is a specific enzyme class that attacks xylan and therefore xylanase falls within the general

term "hemicellulose."

Many types of enzymes could be used within classes of enzymes known as cellulase, xylanase (or other hemicellulose) and lipase. Cellulase has the most commercial choices available because it comes from many different sources, such as from *Aspergillus niger*, *Trichoderma reesei*, *T. viride*, *T. koningi*, *F. solani*, *Penicillium pinophilum*, *P. funiculosum*. It is preferred to use a cellulase that poses endo-exo glucanase functionality to attack both amorphous and crystalline regions of cellulose so that the enzyme can attack any place on the cellulosic surface where ink is attached.

The preferred cellulase is a product sold under the trademark Celluclast® 1.5 L, by Enzyme Process Division, Bioindustrial Group, Novo Nordisk A/S, Novo Allé, 2880 Bagsvaerd, Denmark. Celluclast 1.5 L is a liquid cellulase preparation made by submerged fermentation of a selected strain of the fungus "*Trichoderma reesei*." The enzyme catalyzes the breakdown of cellulose into glucose, cellobiose and higher glucose polymers. The relative amounts of reaction products formed depend on the reaction conditions. Celluclast 1.5 L has an enzyme activity of 1500 NCU/g and is a brown liquid with a density of approximately 1.2 g/ml. Activity is determined on the basis of Novo Cellulase Units (NCU). One NCU is the amount of enzyme which, under standard conditions, degrades carboxy methylcellulose to reducing carbohydrates with a reduction power corresponding to 1 micromole (umol) glucose per minute. Standard conditions are: Substrate - carboxymethyl-cellulose (CMC Hercules-7LFD); Temperature - 40°C; pH - 4.8; Reaction time - 20 minutes.

Xylanase can be obtained from a source such as *A. pullulans*, or *Streptomyces lividans*, or *Streptomyces roseiscleroticus*. Its purpose is to attack the xylan portion of the lignocellulose fiber which is considered to link the white colored cellulose with the brown colored lignin. Therefore, the attack on xylan hemicellulose enhances the removal of lignin, thus making the fiber brighter. It is not

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necessary that the xylanase be cellulase free or from any particular bio-source. In this respect, mushroom enzyme (multiple enzymes found after mushroom growing) could be used without purification.

5 One preferred xylanase enzyme is Pulpzyme® HA which is a xylanase preparation derived from a selected strain of *Trichoderma reesei* available from Enzyme Process Division, Bioindustrial Group, Novo Nordisk A/S, Novo Allé, 2880 Bagsvaerd, Denmark. Pulpzyme® HA contains endo-1,4-beta-D-
10 xylanase (EC 3.2.1.8) as well as exo-1,4-beta-D-xylanase (EC 3.2.1.37) activities. Pulpzyme® HA has a certain amount of cellulase activity in addition to its xylannase activity.

Pulpzyme® HA is a brown liquid preparation of a xylanase having an activity of 500 XYU/g and containing
15 approximately 300 endo-glucanase activity units (EGU/g). One xylanase activity unit (XYU) is defined as the amount of enzyme which under standard conditions (pH 3.8, 30°C, 20 min. incubation) degrades larchwood xylan to reducing carbohydrates with a reducing power corresponding to 1 umol xylose. One
20 endo-glucanase unit (EGU) is defined as the amount of enzyme which under standard conditions (pH 6.0, 40°C, 30 min. incubation) lowers the viscosity of a carboxymethyl cellulose solution to the same extent as an enzyme standard defining 1 EGU. Pulpzyme® HA has a very low activity towards crystalline
25 cellulose. Another preferred xylanase is Pulpzyme® HB which is a xylanase preparation derived from a selected strain of bacterial origin. It is available from Enzyme Process Division, Bioindustrial Group, Novo Nordisk A/S, Novo Allé, 2880 Bagsvaerd, Denmark. It contains endo-1,4-beta-D-xylanase
30 activity (EC 3.2.1.8), and is virtually free of cellulase activity. Pulpzyme® HB is commercially available as a brown liquid preparation, having an endo-xylanase activity of 600 EXU/g in which one endo-xylanase activity unit (EXU) is defined as the amount of enzyme which, under standard conditions (pH
35 9.0, 50°C, 30 min. incubation), degrades RBB xylan.

Lipase can come from *Pseudomonas fragi*, *Candida*

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cylindricea, Mucor javanicus, Pseudomonas fluorescens, Rhizopus javanicus, Rhizopus delemar, Rhizopus niveus, and various species of Miehei, Myriococuum, Humicola, Aspergillus, Hyphozyma, and Bacillus. These have both lipase and esterase activities, and they are known to degrade triglyceride in wood resin into glycerol and fatty acids. As such the lipase enzymes could attack the vegetable oil component of the ink directly. The glycerol by product of lipase activity could help to make the cellulose softer.

10 Preferred lipase enzyme is Resinase[®] A 2X, which is a liquid lipase preparation for the hydrolysis of ester constituents of wood resin. Resinase[®] A 2X is commercially available from Enzyme Process Division, Bioindustrial Group, Novo Nordisk A/S, Novo Allé, 2880 Bagsvaerd, Denmark as a brown liquid preparation with an activity of 100 KLU/g. The lipase activity is measured in Kilo Lipase Units (KLU). One KLU is the amount of enzyme activity which liberates one millimole butyric acid per minute from an emulsion of tributyrin at a temperature of 30°C and a pH of 7.0. The analysis is made in a pH-stat system in which the liberated acid is titrated continuously by addition of sodium hydroxide. The enzyme is not substrate-limited during the analysis.

25 Other enzymes could also be used in combination with these three types of preferred enzymes. They are ligninase, laccase, pectinase, protease and mannanase. Also, enzymes could be obtained from DNA altered and engineered microorganisms which express more of specific enzymes or more volumes to get better economy.

30 The preferred amount and combination of enzymes is 1.33 kg.cellulase/ton (2,000 lbs) of pulp for cellulase, 0.33 kg. xylanase/ton, and 0.33 kg. lipase/ton. As low as 0.25 kilograms of enzymes per ton of pulp (kg/ton) to as high as 25 kg/ton of pulp can be used as the total amount of all enzymes. However, 1 to 3 kg/ton total of all enzymes is a particularly preferred usage rate. The preferred range for each enzyme is: 35 cellulase, 0.25 to 10, kg/ton; xylanase, 0.05 to 2.5, kg/ton;

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and lipase, 0.05 to 2.5, kg/ton.

Swelling of the fiber structure improves the enzyme action by assisting the penetration of the large enzyme molecules into the fiber. Elevated temperature (e.g. above
5 ambient and below 140°F), use of surfactant, and acid or mild alkaline chemicals can be used in pulping the newsprint to physically open up the lignocellulosic fiber structures so that enzymes can better penetrate the structures and perform their respective functions. If high pulping temperatures are used
10 e.g. above about 140°F, the temperature must be lowered to a temperature suitable for enzyme treatment before the enzymes used are added. For most enzymes, the suitable temperature is less than about 140°F.

SURFACTANT USE WITH ENZYME TREATMENT

A synergistic result is obtained with the combination of a surfactant and an enzyme. The minimum effective amount of surfactant to obtain synergy, is the amount needed to open up the fiber rather than the higher levels used for solubilizing oils by emulsifying the oily contaminants. The preferred amount of surfactant is from about 0.025% to about 0.1% based on the weight of fibers. Nonionic surfactants are preferred for addition to the enzyme treatment step to improve the enzymatic action for a better handfeel improvement. A preferred nonionic surfactant is commercially available as DI600[®] from High Point Chemical Corp. DI6000 is an alkoxyated fatty acid, nonionic surfactant specifically developed for flotation type deinking of newsprint. Other nonionic surfactants well known in the art of deinking could be used, such as: Alkyl phenyl ether of polyethylene glycol, e.g. Union Carbide's Tergitol[®] series of surfactants; alkylphenoethylene oxide condensation products, e.g. Rhone Poulenc, Incorporated's Igepal[®] series of surfactants; aryl alkyl polyether alcohol, e.g. Rohm and Haas's Triton[®] X 400 series of surfactants such as Triton X-100. In some cases an anionic surfactant may be used depending on the contaminants present in the wastepaper. Examples of suitable anionic surfactants are: ammonium or sodium salts of a sulfated ethoxylate derived from a 12 to 14 carbon linear primary alcohol such as Vista's Alfonic[®] 1412A or 1412S; and, sulfonated naphthalene formaldehyde condensates, e.g.. Rohm and Haas's Tamol[®] SN. In some cases, a cationic surfactant can be used, especially when debonding is also desired. Suitable cationic surfactants include imidazole compounds e.g., CIBA-GEIGY's Amasoft[®] 16-7 and Sapamine[®] P quaternary ammonium compounds; Quaker Chemicals' Quaker[®] 2001; and American Cyanamid's Cyanatex[®].

Oil Types

Oils of the type typically used in printing, particularly printing of newspapers and in the formulation of

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ink for such printing, are suitable for practice in the present invention. Mineral oils and vegetable oils are the most common types of oils used in formulating printing inks for newspapers. Mineral oil, also known as white mineral oil, alboline, paraffine, Nujol, Saxol, and lignite oil, is generally classified as CAS #64742-46-7. While historically such oils may have been derived from various sources, commercially they are typically a petroleum distillate fraction with a carbon chain averaging from about 10 to about 14 carbon atoms and usually a mixture of paraffinic hydrocarbons, naphthenic hydrocarbons and alkylated aromatic hydrocarbons. Such oils have a specific gravity of about 0.8 to about 0.85, a viscosity at 100°F of 38-41 SSU (Saybolt Universal Units) and an initial boiling point of about 500°F (260°C). Vegetable oils of the type typically used in formulating printing inks can be derived from various sources. Typical is an oil derived from soy beans known as Soya oil, Chinese bean oil, soy bean oil, or just plain soy oil with a chemical abstract service designation CAS #8001-22-7. Such oils are saponifiable with a saponification value of about 185 to 195, a solidifying point of about 5°F to about 18°F, a melting point of about 70 to about 90°F and an Iodine value of about 135 to 145. Other vegetable sources of oil and other types of oil suitable for use in printing inks can also be used in the practice of the present invention.

25 Oil Content

The amount of oil that should be on the fibers (whether on the surface or within the structure of the cellulosic fibers) should be from about 0.2% to about 2%. When newspaper is being used, then preferably this oil content is obtained by not saponifying or solubilizing oils on used newspapers during pulping and treating the used newspapers and preparing them for use in a papermaking furnish. It is also preferred that a surfactant, if used, be used in moderation so as not to wash off oils while preparing newsprint for use in a papermaking furnish for sanitary paper products. When virgin fiber is being used the oil can be added to virgin fibers by

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either adding oil onto the pulp prior to slurring, by adding the oil into a water slurry of the fibers so that the oil comes in contact with the fibers prior to subjecting the fibers to enzyme treatment in accordance with the disclosure herein, or preferably by injecting or mixing oil with the fibers in the fiber disperser. In its broadest concept, the invention requires the presence of oils on or in the fibers from about 0.2% to about 5.0%.

While the synergistic effect is obtained with oils and enzyme treatment of cellulosic fibers, it is most beneficial to high yield fibers, other cellulosic fibers would have their sanitary qualities enhanced by the process of the present invention so that softer more flexible sanitary paper products could be made from such fibers. Such fibers include both northern and southern softwood and hardwood kraft, both bleached and unbleached, bleached and unbleached sulfite fibers in addition to the bleached and unbleached high yield fibers such as stone groundwood fibers, thermomechanical fibers and chemithermomechanical pulp fibers. Specific examples of such fibers are: bleached softwood chemithermomechanical pulp (SWCTMP); bleached northern softwood kraft (NSWK); bleached recycled fiber (RF); bleached eucalyptus kraft pulp (BEK); bleached southern softwood kraft (SSWK); and bleached hardwood chemithermomechanical pulp (HWCTMP).

The oil containing and enzyme treated fibers of the present invention can be used in conventional papermaking processes for the production of sanitary paper products including toilet tissue grade paper, facial tissue grade paper, paper towels and paper napkins in accordance with any conventional process for the production of such products. The softness and bulk of such products would be improved by the use of oil containing and enzyme treated fibers of the present invention. Because of the bulk improvements, paper towels produced with fibers of the present invention would be enhanced.

In accordance with the present invention, it has been

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discovered that conventional deinking is counterproductive to making of soft tissue products from used newspapers because it removes oil that can be beneficial to softness of tissue and towel products. The present invention is also based on the
5 discovery that oil of this type used in newsprint is beneficial to softness of tissue and towel products. Softness is difficult to measure or quantify for tissue products because softness is perceived by the user by handfeel which is influenced by smoothness and other surface characteristics in
10 addition to sheet puffiness. Handfeel tests have been developed and handfeel data reported herein has been obtained using the following test:

HANDFEEL TEST

SCOPE

15 Several different lightweight, dry crepe tissues for use as standards were produced from commercially available pulp of differing qualities for imparting softness to tissue products and were used to define a numerical softness scale. A numerical value was assigned to the softness of each tissue
20 standard. The softest product was assigned a handfeel value of 86, and was a lightweight, dry crepe tissue produced with 50% Irving northern softwood kraft fibers and 50% Sante Fe Eucalyptus kraft pulp. The harshest product for use as a standard was produced with 100% bleached softwood
25 chemithermomechanical pulp, (SWCTMP) and was assigned a handfeel value of 20 on the scale. Other lightweight, dry crepe tissue samples for use as standards in defining the "Handfeel Softness" scale and having softness qualities between the softest and harshest tissue standards were produced from
30 different pulp or pulp blends and were assigned handfeel softness values between 20 and 86. The pulps used are further described in the following paragraphs. Pulp blends and fiber coarseness of the pulp blends used to produce additional tissue standards are given in Table III along with the tensile
35 strength of each tissue standard. Tissue manufacturing

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processes other than the lightweight, dry crepe process and other pulp fibers than those used to produce the standards are capable of producing tissue products outside of the 20 to 86 handfeel softness scale defined by tissue standards described herein. However, for the purpose of establishing the improvement in softness achievable with the present invention, the above defined handfeel softness range of 20 to 86 for lightweight, dry crepe products is accurate and sufficient for comparative purposes. Recycled newsprint fibers of the present invention could produce tissue products having softness values higher than 86 when used in other tissue making process such as the through-dried process or when blended with other fibers.

PULPS USED TO PRODUCE HANDFEEL STANDARDS

(a) Bleached softwood chemithermomechanical pulp (SWCTMP) (Temcell grade 500/80) having a Canadian Standard Freeness (CSF) of 500 and an ISO brightness of 80 was made from Black spruce and Balsam fir. Pulping was with sodium sulfite pretreatment and pressurized refining followed by alkaline peroxide bleaching to 80°ISO brightness. Kajaani coarseness of the fibers equaled 27.8mg/100 meters and the Kajaani weight average fiber length was 1.7mm.

(b) Bleached northern softwood kraft (NSWK) (Pictou grade 100/0 - 100% softwood) was made from Black spruce and Balsam fir. Pulping was by the kraft process to Kappa# = 28 followed by CE₂DED bleaching to 88°ISO brightness. Kajaani coarseness equaled 14.3mg/100 meters and Kajaani weight average fiber length was 2.2mm.

(c) Bleached recycled fiber (RF) was made from sorted mixed office waste that was pulped, screened, cleaned, and washed to 550°CSF followed by bleaching with sodium hypochlorite to 80°ISO brightness. Kajaani coarseness equaled 12.2mg/100 meters and Kajaani weight average fiber length was 1.2mm.

(d) Bleached eucalyptus kraft pulp (BEK) (Santa Fe

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elemental chlorine free grade) was made from Eucalyptus Globulus pulped to Kappa# = 12 by the kraft process followed by ODE_D bleaching to 89°ISO brightness. Kajaani coarseness equaled 6.8 mg/100 meters and Kajaani weight average fiber length was 0.85mm.

(e) Bleached southern softwood kraft (SSWK) (Scott Mobile pine) was made from Loblolly and Slash pine and pulped to Kappa# = 26 followed by CEHED bleaching to 86°ISO brightness. Kajaani coarseness equaled 27.8 mg/100 meters and Kajaani weight average fiber length was 2.6mm.

(f) Bleached Hardwood Chemithermomechanical Pulp (HWCTMP) (Millar Western grade 450/83/100) having a Canadian Standard Freeness (CSF) of 450 and an ISO brightness of 83 was made from quaking aspen. Pulping was with alkaline peroxide pretreatment and pressurized refining followed by alkaline peroxide bleaching. Kajaani coarseness of the fibers equaled 13.8 mg/100 meters and the Kajaani weight average fiber length was 0.85mm.

APPARATUS

The test method requires no apparatus. The test method uses the procedures and materials described below to evaluate tissue samples using a panel of ten or more people and rank softness of the samples on the softness scale using the product standards of known softness scale values.

SAMPLE PREPARATION

1. Five samples to be tested by the panel of evaluators (judges) should be selected.

2. Calculate the number of sample pads and pads of standard samples needed for the test panel of judges for each product to be evaluated for softness using the following equation:

$$\text{Pads needed (each product)} = (x-1) \times (y)$$

x = number of products to be tested

y = number of persons on the test panel

3. Randomly select a roll of sample tissue for each product being evaluated and discard the first few sheets (to

get rid of the tail tying glue).

4. Prepare sample pads from each roll of product being tested. Each pad should be 4 sheets thick and made from a continuous sample of tissue that is four sheets long. Each
5 pad is made as follows: the four sheet long sample is first folded in half. This results in a double thickness sample that is 2 sheets long. The double thickness sample is then folded in half again to produce a 4 sheet thick, single sheet long sample pad. The folding should be done so that the outside
10 surface of the sheets when it was on the roll of tissue becomes the outside surfaces of the pad. If a product being tested is "two-sided", that is it has different surface characteristics on the outside surface of the sheet versus the surface facing the inside of the roll then the product should be tested twice,
15 once with the surface facing the outside of the roll as the outer surface of the sample pad and also tested with a separate sample pad prepared in which the folding results in the sheet surface facing the inside of the roll becoming the outer surface of the sample pad.

20 5. Make up the required number of pads from each product using the formula in paragraph 2 above. If more than one roll of a product is needed to prepare the required number of pads, then it is important that stacks of pads be randomized with product from each of the rolls. Code each pad with the
25 batch code in the top left hand corner (on the fold).

6. Select three standards to be used as references by the panel from among the standard tissues as follows:

- 30 ● Select the coarsest sample being evaluated and compare it to standard tissue sample pads and select a lower standard that is slightly coarser than the coarsest sample.
- Select the softest sample of product being evaluated and select a standard tissue pad that is slightly higher (softer) than the
35 softest sample being evaluated .
- Select a third standard which falls approximately in the middle of the lower

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and higher standards selected.

The three standard tissue pads selected become the handfeel references for the panel and define the softest, coarsest and midrange.

- 5 7. The handfeel references bracket the softness range of the products being evaluated by the panel. For greater accuracy, the highest and lowest references selected should be approximately 30 points apart on the Handfeel Softness Scale. The middle reference should be eight or more
10 points apart from the lower and higher references.

PANEL MEMBER SELECTION AND INSTRUCTION

1. Select a panel of about 10 people having about the same number of males and females and with age variations.
2. Ensure the panel members understand the
15 instructions and if necessary, give a "trial run".
3. Panels should be conducted in a quiet location.

Test Procedures

1. Begin the softness test by reading the following Standard Instructions.

20 STANDARD INSTRUCTIONS

These instructions are to be read to each panel participant before beginning the softness panel test procedure.

a. PURPOSE

25 "The purpose of this procedure is to compare the softness of toilet tissue samples."

b. METHOD

30 "You will be given two sample pads of toilet tissue at a time. Compare the two to each other using your dominant hand and make the comparison by feeling each sample with your dominant hand. You may stroke, bend, or crunch the samples as you see fit for making your judgment."

c. FIRST DECISION

35 "After feeling each of the two sample pads pair, you are asked to decide which sample is

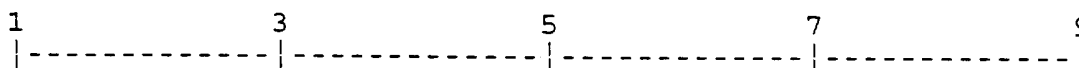
softer."

d. SECOND DECISION

5 "Rate the degree of difference in softness between the two pads using the following rating:

The scale uses odd numbers 1, 3, 5, 7, 9. You may use even numbers if you feel that the numbers listed do not fully represent the difference between two products."

10 PANEL RATING SCALE



The numbers on the rating scale are defined as:

- 1. No difference
- 15 3. Very small difference, not confident, someone could miss it
- 5. Small difference, confident about judgment
- 7. Moderate difference, easy to detect, confident
- 9. Very large difference, very easy to detect, memorable

20 e. CALIBRATION

25 "Before we start I will give you an example of the softest standard to be used for comparison and a sample pad of the least soft (coarsest standard) products. Please handle both. The difference in softness you feel between the two standard references you will rate on the definition scale as 9." (The 9 on the rating scale is the equivalent to the number of handfeel points on the softness scale between the higher and lower references selected for the panel in step 6.)

30

f. PARTICIPANT REACTION

"Do you have any questions about the test procedure?"

35 g. REASSURANCE

"Finally, don't agonize too long over each decision. Your opinion is as good as anybody else's. There are no right or wrong answers!"

2. Present every combination of sample pads and reference pads to each panel member and ask them to select the preferred sample and then rank the difference using the 1 to 9 rating scale of softness. Each panel member should receive the pairs in random order to avoid sequence errors.

3. Record the results of each pair as XYn. Where X is the preferred sample code, Y is the non-preferred sample code and n is the scale value (1 to 9).

Data Analysis

The paired comparison results are treated as if they belong to a ratio scale. The definition of a ratio scale is given as follows: A scale is a ratio scale if this scale is invariant under positive linear transformations of the form $y = ax, a > 0$.

The data pairs and ratio weights for "n" number of pads are loaded into a square matrix A of the following form.

20		O_1	O_2	...	O_n	
		W_1	W			1
25	O_1	W_1	W			2
		W_n^1				
		W_2	W			2
30	O_2	W_2	W			2
		W_n^1				
		W_n	W			n
35	O_n	W_n	W			2
		W_n^1				

Where O_i are the individual samples and W_i are the scale values (ratio weights) for each pair.

For square matrices of this type the following property exists

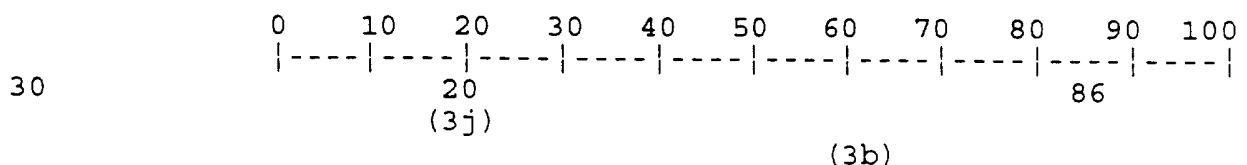
40 $AW = MW$

Where $W = (W_1, W_2, \dots, W_n)$. The weight vector W is

the eigne vector of the matrix A corresponding to its eigne value n. Saaty has shown (See, Saaty, T.L. "A Scaling Method for Priorities in Hierarchical Structures", Journal of Mathematical Psychology, 15, 234-281 (1977) and Saaty, T.L., "Measuring the Fuzziness of Sets", Journal of Cybernetics, 4(4), 53-61 (1974)) that to extract the eigne vector W from the estimated weights requires finding the largest eigne value of A (λ max). A computer program to solve for λ max and W is provided in McConnell, Wes, "Product Development Using Fuzzy Sets", INDA Tenth Technical Symposium, pp 55-72, November 17-19, 1982. The resulting eigne vector W is the best estimate ratio scale of the paired inputs. Taking the log of each element in this vector creates the more familiar equal interval scale in which the distances between objects are linear. The standard softness values are plotted versus the estimated equal interval scale values and the unknown samples are assigned numerical values by interpolation.

The mean and standard deviation of the standard softness values of each unknown sample are calculated from the calculated standard softness values for all panel members. If any individual panel member value falls outside of 2 standard deviations from the mean, that value is discarded and the mean and standard deviation are recalculated. The mean of the standard softness values with no values outside of 2 standard deviations from the mean is the standard handfeel softness value for that unknown sample.

HANDFEEL SOFTNESS SCALE



Tensile Strength

Tensile strength values given herein for tissue type

paper products are measured by a breaking length test (TAPPI Test Method No. T494om-88) using 5.08 cm. sample span and 5.08 cm/minute cross head speed. Typically, tissue strengths are different in the machine direction versus cross machine direction of the sheet. Also, the basis weight of tissue samples vary which affects tensile strength. In order to better compare tensile strengths from various tissue samples it is important to compensate for the differences in basis weight of the samples and for machine directional differences in tensile strength. This compensation is achieved by calculating a "Basis Weight and Directionally Normalized Tensile Strength" (hereinafter "Normalized Tensile Strength" or "NTS"). NTS is calculated as the quotient obtained by dividing the basis weight into the square root of the product of the machine direction and cross machine direction tensile strengths. Tensile strength calculations normalized for differences in basis weight and machine direction have been devised for better comparisons of tissue samples. Tensile strengths are measured in both the machine direction and cross machine direction and the basis weight for the tissue sample is measured in accordance with TAPPI Test Method No. T410om-88. When English units of measurement are used, tensile strength is measured in ounces per inch and basis weight in pounds per ream (2880 square feet). When calculated in metric units the tensile strength is measured in grams per 2.54 centimeters and the basis weight is measured in grams per square meter. It should be noted that the metric units are not pure metric units because the test apparatus used for testing tensile is set up to cut a sample in inches and accordingly the metric units comes out to be grams per 2.54 centimeters. Using the abbreviations MDT for machine direction tensile, CDT for cross machine direction tensile and BW for basis weight, the mathematical calculation of Basis Weight and Directionally Normalized Tensile strength (NTS) is:

$$\text{NTS} = (\text{MDT} \times \text{CDT})^*/\text{BW}$$

$$\text{NTS in English units} = 0.060 \times \text{the NTS in the above}$$

defined metric units.

TISSUE MAKING PROCESS

The oil containing enzyme modified fibers of the present invention may be used in any commonly known papermaking process for producing, soft, bulky, sanitary paper webs such as tissue, towel, napkins and facial tissue. Many different papermaking processes including those processes wherein the web is dried via can drying, through drying, thermal drying, and combinations thereof are suitable. Exemplary of the types of papermaking processes which might be used in conjunction with the present invention are those processes taught in U.S. Patent Nos. 3,301,746 to Sanford et al., 3,821,068 to Shaw, 3,812,000 to Salvucci et al., 3,994,771 to Morgan, Jr. et al., 4,102,737 to Morton, 4,158,594 to Becker et al., 4,440,597 to Wells et al., and 5,048,589 to Cook et al.

The preferred papermaking process is commonly known as the dry crepe process. Generally this involves using the paper furnish of the present invention to which dry strength chemicals are preferably added to generate tensile strength and other papermaking chemicals may be added. The paper furnish is then pumped from a machine chest and flows to a headbox and through a slice at 0.1 to 0.4% consistency onto a horizontal surface of a Fourdrinier wire through which water is withdrawn and web formation takes place. The wire cloth is entrained around a breast roll and several table rolls, then to a wire turning roll from which it is fed around a couch roll and several guide rolls back to the breast roll. One of the rolls is driven to propel the Fourdrinier wire. One or more vacuum boxes, deflectors or hydrofoils may be used between the table rolls to enhance water removal.

The wet web is formed on the upper surface of the Fourdrinier and transferred to a felt by pressing the web onto the felt by means of a couch roll or transferring the sheet to the felt by means of a pick-up shoe. The felt transports the web to a press assembly. The felt then moves around one or two press rolls, one of which may be a suction roll, and then is

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entrained around guide rolls and rotates back to the couch roll. Showers and guard boards can be used at various positions on the felt surface to assist in web pick-up, cleaning and conditioning the felt surface. The press assembly
5 comprises either a single press roll or an upper and lower press roll. Moisture is removed in the nip of the press assembly and transferred into the felt.

The formed and pressed web is transferred to the surface of a rotating drying cylinder, referred to as a yankee
10 dryer. The drying assembly may also include a hot air hood surrounding the upper portion of the yankee cylinder. The hood has hot air nozzles which impinge on the web and assist in moisture removal. The hood includes an exhaust to remove air from the hood chamber to control temperature. The web is
15 removed from the drying surface using a doctor blade to impart crepe to the web. To assist in removing the web from the drying surface in a controlled, uniform state, a creping adhesive is applied to yankee surface using a spray system. The spray system is a series of spray nozzles attached to a
20 header pipe extending across the width of the dryer surface. The creping adhesive can be any of the types commonly used in tissue papermaking technology.

The paper web creped from the drying cylinder is passed through a nip formed by a pair of rolls and wound into
25 a large roll referred to as a parent roll.

The tissue making process used in the examples can be generally characterized as a light weight, dry crepe process. A 14 inch wide pilot plant scale machine was operated as follows: Prior to web formation the paper furnish is
30 contained in a machine chest where dry strength additives, dyes or other chemical additives are incorporated. The paper furnish is delivered via a fan pump which flows from a headbox through a slice at 0.1% to 0.4% consistency onto the horizontal surface of a Fourdrinier wire through which water is withdrawn
35 and web formation takes place. The wire is entrained around a suction breast roll which aids in water removal and web

formation. The wire is entrained around several guide rolls and a wire turning roll and is fed back to the breast roll. One of these rolls is driven to propel the Fourdrinier wire.

The wet web is formed on the upper surface of the
5 Fourdrinier and transferred to a felt by means of a vacuum pick-up. The felt transports the sheet to a pressure roll assembly. The felt moves around one pressure roll, a solid rubber roll, and is entrained around guide rolls and rotates back to the vacuum pick-up. Moisture is removed in the nip of
10 the pressure roll and transferred into the felt.

The formed web is pressed and transferred to the surface of a rotating drying cylinder, commonly referred to as a Yankee Dryer. The web is removed from the surface of the Yankee at a web dryness between 95% and 96% using a doctor
15 blade. To assist in removing the web from the dryer surface in a controlled uniform state, a creping adhesive is applied to the Yankee surface using a spray nozzle. The adhesive mixture used in these examples was a 70/30 mixture of 70% polyvinyl alcohol and 30% of a starch based latex (National
20 Starch Latex 4441).

The paper web creped from the drying cylinder was passed through a nip formed by a pair of rolls and wound into a parent roll of desired size for testing. The paper machine formed a web 14 inches wide and ran at a reel speed of 40 to
25 50 feet/minute. All of the dry creped tissue samples in the examples were produced at a basis weight of 10 pounds/ream and 18-20% crepe. The samples were converted to 2-ply tissue (20 pounds/ream) for all testing.

The synergistic result from the combination of oils,
30 coarse fibers and surfactants is demonstrated in the following examples. All proportions used herein are by weight unless otherwise specified and fiber weight is based upon the air dried weight of the fiber unless otherwise indicated.

Example 1

35 Before the sample tests and results are described, it may be helpful to understand the data in Table I. The first

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column labeled, "SAMPLE/NUMBER", lists samples 1a through 1o. It should be noted that samples 1a through 1k are samples 3a through 3k, which come from Table III that were tested in of U.S. Patent application Serial Number 08/268,232, filed on June 5 29, 1994. Samples 1a through 1k are incorporated into Table I for comparison purposes.!!

A fiber mixture was prepared having 100% virgin fibers of the type used to make newsprint in the United States. The pulp sample contained 60% low freeness (about 250°CSF) 10 softwood chemi-thermomechanical pulp (SWCTMP), 30% softwood stoneground pulp and 10% northern softwood kraft pulp. This 60/30/10 blend of virgin fibers was selected to simulate the blends found in newsprint. Virgin fibers were employed because they do not contain any contaminants introduced in the 15 manufacture of newspapers. The pulp was formed into a sheet of flat paper and dried without any chemical additions. The flat paper was divided into representatives samples and, separate papermaking furnishes were prepared from each flat paper sample and dry creped tissue product was produced from 20 each flat paper furnish sample on a 14" wide dry crepe tissue machine as described above.

The samples of flat paper were designated samples 1N and 10. While sample 1N was left uncontaminated, sample 10 was 25 contaminated with surfactant and with enzymes, dewatered to a consistency of about 25-35%; crumbed to produce crumbed fiber; and passed through a fiber disperser and mixed with 0.50-2% soy bean oil while the fiber temperature was maintained at about 180°F. by injected steam, before being repulped and made into 30 lightweight dry crepe tissue. Lightweight dry crepe tissue products were made from each of the samples 1N and 10:

A separate pulp slurry was prepared with each tissue sample. A cationic starch dry strength resin Solvitose® - N (available from Nalco Chemical Co.) was added at a rate of 1% of the fiber weight. The pulp slurry at about 6% consistency was elevated 35 to a temperature of 180°F for 15 minutes. After being pulped and held at the elevated temperature for 15 minutes, the pulp

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slurry made from the sample (soy bean oil contaminated) was cooled to about 140°F, and diluted to a consistency of 5%, and the pH was reduced from ambient pH obtained with just pulp and tap water to a pH of 5 by the slight addition of sulfuric acid.

5 Surfactant and an enzyme mixture of cellulase enzyme (Celluclast 1.5L, Novo Nordisk Bioindustrials, Inc.), xylanase (Pulpzyme HA, Novo Nordisk Bioindustrials, Inc.) and resinase (Resinase A 2X, Novo Nordisk Bioindustrials, Inc.) was added to the 5% consistency furnishes of sample 10. The enzyme

10 addition uses 66.5 ml cellulase to 16.5 ml xylanase and 16.5 ml resinase per 100 lbs of air dried pulp. After addition of the enzymes, the pulp slurry of sample 10 was held at about 140°F for 30 minutes with mild agitation and then cooled and adjusted to pH 7 with sodium hydroxide raised.

15 The pulp was dewatered to a consistency of 25-35%. The dewatered fibers were then crumbed and then passed through a fiber disperser and mixed with the soy bean oil, while the fiber temperature was maintained at about 180°F. by injected steam.

20 The pulp slurry was then used to make lightweight dry crepe tissue as described above. Significant softness in terms of handfeel for this sample was observed. A substantial, synergistic improvement in softness was obtained by the combination of oil contamination of the fibers and a treatment

25 with the enzymes. Table I gives the results of the Normalized Tensile Strength (basis weight and directionally normalized) and handfeel for the tissue samples 1N and 10. It can be seen from the table that dramatic improvement in handfeel (perceived softness) is achieved by the combination of enzyme treatment

30 and oil contamination via dispersing treatment with steam on the tissue product.

Example 2

Three pulp furnishes made from different sources of high coarseness fibers were treated and made into lightweight

35 dry crepe tissue product using the same papermaking procedures as in Example 1. Two tissue samples designated 1K and 1L were

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made from a pulp obtained from repulping old newspaper (ONP). The other tissue sample, designated 1M was made from 70% ONP and 30% flexo newsprint. All tissue samples were first made by slurring the pulp with water at 6% consistency. The slurry
5 was raised to a temperature of 180°F and maintained at the elevated temperature for 30 minutes.

Each of the pulp slurries from samples 1K, 1L and 1M, were subjected to an additional treatment prior to being used in the papermaking process. This additional treatment was
10 comprised of reducing the temperature of the pulp slurry from 180°F to 140°F, adjusting the pH with sulfuric acid to 5.0 and adjusting the consistency to 5% adding surfactant and enzyme mixture. The enzyme addition was at a rate of 66.5 ml. for cellulase, 16.5 ml. xylanase and 16.5 ml. for lipase per 100
15 pounds of pulp, and the enzymes were added to the 140°F, 5% consistency slurries to be used for making these tissue samples. After the enzyme treatment, the slurry was dewatered to a consistency between about 25-35%. The dewatered pulped fiber was then crumbed to produce crumbed fibers. The crumbed
20 fibers from samples 1L and 1M were then passed through a micar.

Sample 1L was mixed with 1% of mineral oil while the fiber was maintained at a temperature of about 180°F. by injected steam.

Sample 1M, made from 70% oil/30% flexo newsprint was
25 mixed with 1.0% of mineral oil while the fibers were maintained at a temperature of about 180°F. by injected steam.

The 5% consistency slurries for all samples were then maintained at 140°F for 30 minutes, cooled, adjusted to pH 7 with sodium hydroxide and used as furnish for making dry crepe
30 tissue with the papermaking equipment and process described in Example 1. The cationic dry strength resin Solvitose® N was added to the furnish at a rate of 1% based on the weight of fibers. Tissue samples 1K, 1L, and 1M were then tested for handfeel, tensile in both machine and cross machine direction
35 and for basis weight. The results are shown in Table I. The tensile and basis weight data were used in the mathematical

calculation of the directionally normalized tensile strength (NTS). The results established the benefits of the addition of oils and steam to fibers of coarseness of 17 mg/100 meters and higher after enzyme treatment.

5 Example 3

Two tissue samples designated A and B were prepared from a pulp obtained by repulping old newspaper. Both tissue samples were first made by slurring the pulp with water at 6% consistency. The slurry was raised to a temperature of about
10 180°F and maintained at the elevated temperature for about 30 minutes.

Each of the pulp slurries were subjected to an additional treatment prior to being used in the papermaking process. This additional treatment was comprised of reducing
15 the temperature of the pulp slurry from 180°F. to 140°F., adjusting the pH with sulfuric acid to 5.0 and adjusting the consistency to about 5.0%. A surfactant and an enzyme mixture consisting of 66.5 ml. of cellulase 16.5 ml. xylanase and 16.5 ml. lipase was added per 100 pounds of pulp at 140°F. After
20 the enzyme treatment, the slurry was dewatered to a consistency between about 25% to 35%. The dewatered pulped fiber was then crumbed to produce crumbed fibers. The crumbed samples were then passed through a disperger.

Sample A was mixed with 1% of mineral oil while the
25 fiber was maintained at a temperature of about 180°F. by injected steam, whereas sample B was mixed with castor oil while the fiber was maintained at 180°F. by injected steam.

The dewatered samples were then adjusted to a consistency of 5% and used as furnish for making dry crepe
30 tissue with the papermaking equipment and process described in example 1. Tissue samples A and B were then tested for handfeel, tensile, both machine and cross direction and basis weight. The tensile and basis weight data were used in the mathematical calculation of directionally normalized tensile
35 strength. These results are listed in Table II. The results show that the type of vegetable oil used in the disperger has

a significant effect on the softness. The castor oil is preferred to the mineral oil.

Example 4

A fiber mixture was prepared using 100% virgin fibers
5 of the type used to make newsprint in the United States. The
pulp mixture contained 60% low freeness
(about 250 CSF) softwood chemi-thermomechanical pulp
(SWCTMP), 30% softwood stonegroundwood pulp and 10% northern
softwood kraft. This 60/30/10 blend of virgin fibers was
10 selected to simulate the blends found in newsprint. Virgin
fibers were employed because they do not contain any
contaminants introduced in the manufacture and printing of
newspapers. Dry creped tissue samples were prepared at two
different tensile levels on a 14" wide dry crepe tissue machine
15 as described above. These samples are designated A1 through
A4 in Table III.

Four pulp furnishes, Samples B, C, D and E, of old
newspaper were prepared at 5% consistency, dewatered to a
consistency of 25 to 25%, and then crumbed. Sample B was
20 passed through a fiber disperger and mixed with mineral oil but
was not subjected to steam treatment. Sample C was passed
through a disperger and subjected to steam treatment at 180°F.
but was not mixed with oil. Samples D and Sample E were passed
through a disperger and mixed with 0.5 to 2.0% of mineral oil
25 while the fiber treatment was maintained at 180°F. with steam.

The four treated furnishes were separately slurried
to a consistency of 5% and subjected to enzyme treatment as
described in Example I.

The enzyme treated samples were then used as furnish
30 for making dry crepe tissue and the papermaking equipment
described in example 1.

Sample D and Sample E were passed through the
disperger and mixed with oil and subjected to steam treatment
and prepared at two different tensile levels.

35 Tissue samples A through E were tested for handfeel,
tensile, both machine and cross direction and basis weight.

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The tensile and basis weight data were used in the mathematical calculation of directionally normalized tensile strength.

The results in Table III show that Sample B through D had better in softness levels than the controls at equivalent
5 NTS.

Claims:

We claim:

1. A method of making sanitary paper products from cellulosic fibers, comprising:

5 (a) pulping said cellulosic fibers in water with agitation to produce a pulp slurry, said slurry having a consistency between about 3% to about 18% and a pH below about 8.0;

10 (b) adding to the slurry a surfactant and at least one enzyme selected from the group consisting of cellulase, hemicellulose and lipase and maintaining said pulp slurry at a temperature above about 100°F for at least 15 minutes;

15 (c) dewatering the slurry to consistency of from about 25% to about 35%;

(d) crumbing the dewatered slurry, thereby producing crumbed fiber;

20 (e) passing the crumbed fiber through a fiber disperser and mixing oil selected from the group consisting of vegetable, mineral or lanolin oils or their derivatives with said fiber to produce treated pulp containing oily products; and

25 (f) using said treated pulp as a source of fibers in a paper making process to produce sanitary paper products.

2. The method of claim 1 wherein said cellulosic fiber is high coarse fiber.

3. The method of claim 1 wherein said cellulosic fiber is low coarse fiber.

30 4. The method of claim 1 wherein said hemicellulose is xylanase.

5. The method of claim 1 wherein the sanitary paper product is a tissue paper made at a basis weight between 7 and 35 pounds per ream.

35 6. The method of claim 1 wherein the sanitary paper product is a paper towel made at a basis weight between 20 and

40 pounds per ream.

7. The method of claim 1 wherein said pH of said pulp slurry is maintained between about 4 and 7.

8. The method of claim 1 wherein said pH and 5 chemical additions to the pulp slurry are insufficient to saponify said oily components.

9. A method of making sanitary paper products from cellulosic fibers comprising:

(a) pulping said fibers in water with agitation 10 to produce a pulp slurry, said slurry having a consistency between about 3% and 18% and a pH below about 8.0;

(b) maintaining said pulp slurry at a temperature above about 100°F for at least 15 minutes;

(c) adding to the slurry at a temperature below 15 140°F a surfactant and at least one enzyme selected from the group consisting of cellulase, hemicellulose and lipase;

(d) maintaining said pulp in contact with said enzyme for at least about 30 minutes;

(e) dewatering the slurry to a consistency from 20 about 25% to about 35%;

(f) crumbing the dewatered slurry, thereby producing crumbed fibers;

(g) passing the crumbed fibers through a fiber disperser, and mixing oil selected from the group consisting 25 of vegetable, mineral or lanolin oils or their derivatives with said fiber to produce treated pulp containing oily product, and maintaining said fibers at a temperature of about 180° F.; and

(h) using said treated pulp slurry as a source 30 of fibers in a papermaking process to produce sanitary paper products.

10. The method of claim 9 wherein said cellulosic fiber is high coarse fiber.

11. The method of claim 9 wherein said cellulosic 35 fiber is low coarse fiber.

12. The method of claim 9 wherein said temperature of said dispersed fibers in step (g) is maintained by injected steam.

13. The method of claim 9 wherein said hemicellulose
5 is xylanase.

14. The method of claim 9 wherein the sanitary paper product is a tissue paper made at a basis weight between 7 and 35 pounds per ream.

15. The method of claim 9 wherein the sanitary paper
10 product is a paper towel made at a basis weight between 20 and 40 pounds per ream and is a paper towel.

16. The method of claim 9 wherein said pH of said slurry is maintained between about 4 and 7.

17. A method of making sanitary paper from
15 cellulosic fibers of coarseness and comprising:

(a) slurring said cellulosic fibers with water at a consistency of between about 3% to about 18%;

(b) adding surfactant and at least one enzyme,
20 to said slurry, said enzyme selected from a group consisting of cellulase, hemicellulose and lipase, and maintaining said pulp in contact with said enzyme for at least about 15 minutes;

(c) dewatering the slurry to a consistency of from about 25% to about 35%;

(d) crumbing the dewatered fiber, thereby
25 producing a crumb fibers;

(e) passing the crumbed fibers through a fiber disperser and mixing oil selected from the group consisting of vegetable, mineral or lanolin oils or their derivatives with
30 said fiber, and maintaining said fibers at a temperature of about 180°F.;

(f) using said enzyme and surfactant treated pulp slurry as a source of fibers in a papermaking process to produce sanitary paper products; and,

(g) wherein said pulp, is maintained below
35 about pH 8 throughout said method.

18. The method of claim 17 wherein said cellulosic fiber is high coarse fiber.

19. The method of claim 17 wherein said cellulosic fiber is low coarse fiber.

5 20. The method of claim 17 wherein said temperature of said dispersed fibers in step (e) is maintained by injected steam.

21. The method of claim 17 wherein said hemicellulose is xylanase.

10 22. The method of claim 17 wherein the sanitary paper product is a tissue paper made at a basis weight between 7 and 35 pounds per ream.

23. The method of claim 17 wherein the sanitary paper product is a paper towel made at a basis weight between
15 20 and 40 pounds per ream.

24. The method of claim 17 wherein said pH of said pulp slurry is maintained between about 4 and 7.

25. The method of claim 17 further comprising adding
20 cationic dye to said enzyme treated pulp.

26. A method of making sanitary paper products from cellulosic fibers, comprising:

(a) pulping said cellulosic fibers in water with agitation to produce a pulp slurry, said slurry having a
25 consistency between about 3% and 18% and a pH below about 8.0;

(b) adding to the slurry a surfactant and at least one enzyme selected from the group consisting of cellulase, hemicellulose and lipase and maintaining said pulp slurry at a temperature above about 100°F for at least 15
30 minutes;

(c) dewatering the slurry to consistency of from about 25% to about 35%;

(d) crumbing the dewatered slurry, thereby producing crumbed fiber;

35 (e) passing the crumbed fiber through a fiber disperser and mixing oil selected from a group consisting of

vegetable, mineral or lanolin oils or their derivatives with said fiber to produce treated pulp containing oily products, and maintaining said fibers at a temperature of about 180°F.; and

5 (f) using said treated pulp as a source of fibers in a paper making process to produce sanitary paper products.

27. The method of claim 26 wherein said cellulosic fiber is high coarse fiber.

10 28. The method of claim 26 wherein said cellulosic fiber is low coarse fiber.

29. The method of claim 26 wherein said temperature of said dispersed fibers in step (e) is maintained by injected steam.

15 30. The method of claim 26 wherein said hemicellulose is xylanase.

31. The method of claim 26 wherein the sanitary paper product is a tissue paper made at a basis weight between 7 and 35 pounds per ream.

20 32. The method of claim 26 wherein the sanitary paper product is a paper towel made at a basis weight between 20 and 40 pounds per ream.

33. The method of claim 26 wherein said pH of said pulp slurry is maintained between about 4 and 7.

25 34. The method of claim 26 wherein said pH and chemical additions to the pulp slurry are insufficient to saponify said oily components.

35. The paper produced by the method of claim 1.

36. The paper produced by the method of claim 7.

30 37. The paper produced by the method of claim 13.

38. The paper produced by the method of claim 20.

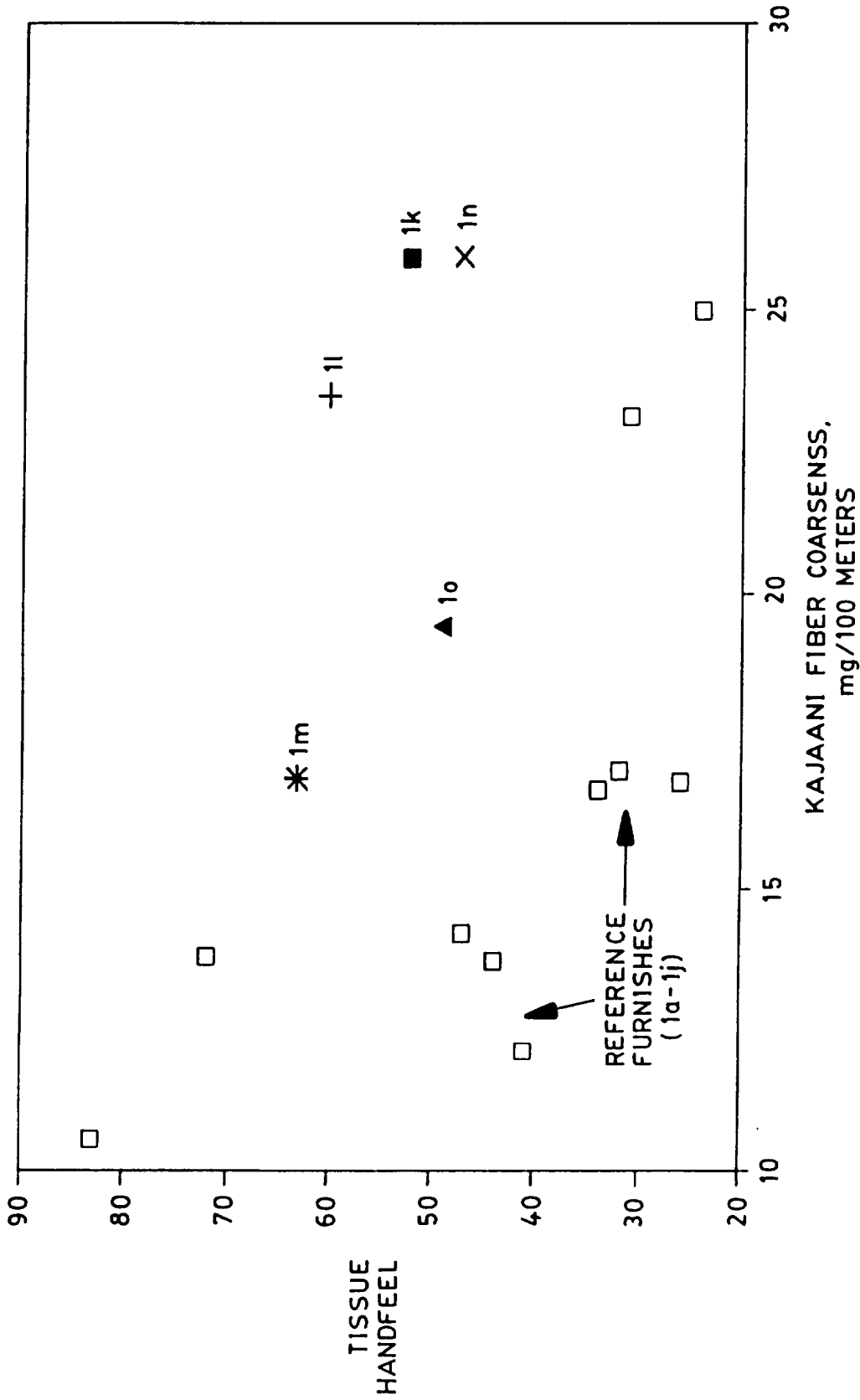


FIG.1

TABLE I
STRENGTH, HANDFEEL, AND COARSENESS DATA

SAMPLE NUMBER	FURNISH	SAMPLE #1		SAMPLE #2		HANDFEEL @ 10.4 NTS	COARSENESS (mg/100 meters)	HANDFEEL	COARSENESS (mg/100 meters)
		NTS	HANDFEEL	NTS	HANDFEEL				
1a	70% SSWK / 30% HWCTMP	9.7	33	10.4	31	31	23.2	31	23.2
1b	50% NSWK / 50% BEK	9.6	86	10.9	81	83	10.6	83	10.6
1c	25% NSWK / 25% SSWK / 50% BEK	8.9	75	10.2	71	72	13.9	72	13.9
1d	100% NSWK	9.9	48	13.7	44	47	14.3	47	14.3
1e	50% NSWK / 50% RF	10.5	44	16.0	39	44	13.8	44	13.8
1f	100% RF	12.2	40	14.8	37	41	12.2	41	12.2
1g	33% NSWK / 33% RF / 33% SWCTMP	11.8	33	14.7	31	34	16.7	34	16.7
1h	50% NSWK / 50% SWCTMP	8.9	34	15.0	28	32	17.0	32	17.0
1i	50% RF / 50% SWCTMP	10.0	26	15.0	24	26	16.8	26	16.8
1j	100% SWCTMP	9.4	26	12.8	20	24	25.0	24	25.0

FIG. 2A

TABLE I
STRENGTH, HANDFEEL, AND COARSENESS DATA

SAMPLE NUMBER	FURNISH	SAMPLE #1		SAMPLE #2		HANDFEEL @ 10.4 NTS	COARSENESS (mg/100 meters)	HANDFEEL	COARSENESS (mg/100 meters)
		NTS	HANDFEEL	NTS	HANDFEEL				
1k	ONP + enzyme/ surfactant	8.6	66	10.3	52	52	25.9	52	25.9
1l	ONP + enzyme/ surfactant + micar + steam + oil	11.3	56	7.0	71	60	23.6	60	23.6
1m	ONP/FLEXO + enzyme/ surfactant + micar + steam + oil	10.8	63	NO SAMPLE	NO SAMPLE	63	16.9	63	16.9
1n	CONTROL = 60% SWCTMP + 30% SWGWD + 10% NSWK	7.4	53	15.8	38	47	25.9	47	25.9
1o	CONTROL + enzyme/ surfactant + micar + steam + oil	10.4	49	NO SAMPLE	NO SAMPLE	49	19.5	49	19.5

FIG. 2B

TABLE II
STRENGTH, HANDFEEL, AND COARSENESS DATA

<u>SAMPLE NUMBER</u>	<u>SAMPLE DESCRIPTION</u>	<u>HANDFEEL</u>	<u>NTS (metric)</u>	<u>COARSENESS (mg/100 meters)</u>
A.	ONP + MICAR + STEAM + MINERAL OIL	71	7.04	19.3
B.	ONP + MICAR + STEAM + CASTOR OIL	85	6.63	19.3

FIG. 3

TABLE III
STRENGTH, HANDFEEL, AND COARSENESS DATA

<u>SAMPLE NUMBER</u>	<u>SAMPLE DESCRIPTION</u>	<u>HANDFEEL</u>	<u>NTS (metric)</u>	<u>COARSENESS (mg/100 meters)</u>
A1.	CONTROL	53	7.4	25.9
A2.	CONTROL	38	15.8	25.9
A3.	CONTROL (CALCULATED AT 11.4 NTS)	46	11.4	25.9
A4.	CONTROL (CALCULATED AT 10.6 NTS)	47	10.6	25.9
B.	ONP + MICAR + MINERAL OIL (NO STEAM)	47	11.4	25.6
C.	ONP + MICAR + STEAM (NO OIL)	50	10.6	25.6
D.	ONP + MICAR + STEAM + MINERAL OIL	56	11.3	23.6
E.	ONP + MICAR + STEAM + MINERAL OIL	60	10.4	23.6

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FIG. 4