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(54) Title: THICK AND SMOOTH MULTI-PLY TISSUE

(57) Abstract: The present invention relates to a paper tissue, and in particular to facial tissue, and disposable handkerchiefs. Claimed and described is a paper tissue comprising at least two plies, characterised in that the paper tissue has a physiological surface smoothness parameter of less than 700 microns, preferably from 650 microns to 50 microns, more preferably from 650 microns to 300 microns and in combination has a caliper per ply of more than 0.09 mm, preferably from 0.09 mm to 0.5 mm, more preferably from 0.1 mm to 0.2 mm. In one preferred embodiment a three-ply tissue with embossed middle ply is provided. Further is a related process claimed and described.

THICK AND SMOOTH MULTI-PLY TISSUE

Field of the invention

The present invention relates to paper tissue, and in particular to facial tissue, and disposable handkerchiefs. In one preferred embodiment a three-ply tissue with embossed middle ply is provided.

Background of the invention

Paper webs or sheets, sometimes called tissue or paper tissue webs or sheets, or herein called paper tissue, find extensive use in modern society. Such items as facial and toilet tissues are staple items of commerce, all of which are herein referred to as paper tissue. It has long been recognised that important physical attributes of these products are their strength and thickness/caliper, their softness and smoothness, their absorbency, and their lint resistance. Research and development efforts have been directed to the improvement of each of these attributes without seriously affecting the others as well as to the improvement of two or three attributes simultaneously.

Softness and smoothness relate to the tactile sensation perceived by the consumer when holding a particular product, rubbing it across the skin, or crumpling it within the hands. This tactile sensation is a combination of several physical properties. One of the more important physical properties related to the softness and smoothness is generally considered by those skilled in the art to be the surface structure of the paper tissue from which the tissue product is made and which is best captured by the physiological surface smoothness (PSS) parameter as known e.g. from US 5,855,738. As important for the tactile sensation of consumers is the thickness/caliper of a tissue product.

Strength is the ability of the product to maintain physical integrity and to resist tearing, bursting, and shredding under use conditions.

Absorbency is the measure of the ability of a product to absorb quantities of liquid, particularly aqueous solutions or dispersions. Overall absorbency as perceived by the consumer is generally considered to be a combination of the total quantity of a liquid a given mass of paper tissue will absorb at saturation as well as the rate at which the mass absorbs the liquid.

Lint resistance is the ability of the fibrous product, and its constituent webs, to bind together under use conditions, including when wet. In other words, the higher the lint resistance is, the lower the propensity of the web to lint will be.

WO97/44528 discloses a multi-ply tissue product with high absorbency. Example 4 discloses a product where a patterned, relatively textured ply is disposed between two substantially unpatterned, relatively untextured plies.

EP 0 264 676 discloses a process for the manufacture of multi-ply paper sheets. Example 3 discloses a three-ply product made from wet-formed paper, where the inner web is provided from embossed paper with a weight of 18g/m^2 and the outer webs are provided from calendered paper with a weight of 14g/m^2. The plies are assembled by a cellulose ether adhesive applied by nozzles.

US 5,855,738 discloses a process for making smooth paper tissue comprising a calendering step.

Relatively thick disposable paper products, namely in the form of paper handkerchiefs and facial tissues, are known. For example, Tempo™, sold by The Procter & Gamble Company, has a caliper of about 0.3 mm. A high caliper conveys the idea of high dry and wet strength to the consumer. A high wet strength, also referred to as wet burst strength, in particular prevents tearing or bursting which in turn results in contamination of the user's hand with mucus or other bodily fluids.

Even thicker disposable paper products are known and typically used as kitchen towels, such as Bounty™, sold by The Procter & Gamble Company, which has a caliper of about 0.7 mm and a wet burst strength which is greater than 200 g. However such kitchen towels to a considerable extent owe their caliper to embossing over the whole surface which results in a surface texture which is rough and does not provide a suitably smooth wiping surface for blowing the nose.

Other products with high wet burst strength and typically a relatively high caliper are those produced by through-air-drying. Though-air-drying facilities, however, are not available on conventional paper making machines and the provision of such equipment means a considerable financial investment.

In theory, the wet strength and caliper of a product can be increased by increasing the number of plies to 5, 6 or even more (instead of embossing or the like) and thereby maintaining a smooth outer surface. However, this approach would be very costly and also lead to a stiff product, hence compromising tactile perception.

In attempting to provide a very smooth surface it is common in the art to subject paper tissue to calendering. However, calendering always means a trade-off of caliper and softness for smoothness (as discussed e.g. in US 5,855,738).

In view of the prior art there remains a need for a tissue product, in particular a facial tissue, which:

- combines optimal strength, namely wet burst strength, absorbency and lint resistance
- further gives an ideal tactile sensation of softness, smoothness and thickness
- is cost effective to manufacture and preferably can be manufactured on conventional paper machines

– optionally provides skin care benefits

Summary of the Invention

The present invention relates to a paper tissue, and in particular to facial tissue, and disposable handkerchiefs. Claimed and described is a paper tissue comprising at least two plies, characterised in that the paper tissue has a physiological surface smoothness parameter of less than 700 microns, preferably from 650 microns to 50 microns, more preferably from 650 microns to 300 microns and in combination has a caliper per ply of more than 0.09 mm, preferably from 0.09 mm to 0.5 mm, more preferably from 0.1 mm to 0.2 mm. In one preferred embodiment a three-ply tissue with embossed middle ply is provided. Further is a related process claimed and described.

Detailed Description of the Invention

According to the present invention, a cellulosic fibrous structure is wet-laid using principles and machinery well-known in the art of paper-making. A suitable pulp furnish for the process of making the paper tissue substrate preferably contains papermaking fibres consisting essentially of cellulose fibres (commonly-known as wood pulp fibres) or cellulose-derived fibres (including, for example, rayon, viscose). Fibres derived from soft woods (gymnosperms or coniferous trees) and hard woods (angiosperms or deciduous trees) are contemplated for use in this invention. The particular species of tree from which the fibres are derived is immaterial. The wood pulp fibers can be produced from the native wood by any convenient pulping process. Chemical processes such as sulfite, sulphate (including the Kraft) and soda processes are suitable. Mechanical processes such as thermochemical (or Asplund) processes are also suitable. In addition, the various semi-chemical and chemi-mechanical processes can be used. Bleached as well as unbleached fibers are contemplated for use. Preferably no non-cellulosic fibres, such as latex, fibres are used.

The paper tissue according to the present invention may contain, as a highly preferred component a wet strength chemical agent. Preferably up to about 3.0%, preferably at least 0.5%, and more preferably at least 0.8% by weight, on a dry fiber weight basis, of wet strength chemical agent, such as water-soluble permanent and temporary wet strength resin, are contained.

Wet strength resins useful herein can be of several types. For example, Westfelt described a number of such materials and discussed their chemistry in Cellulose Chemistry and Technology, Volume 13, at pages 813-825 (1979).

Usually, the wet strength resins are water-soluble, cationic materials. That is to say, the resins are water-soluble at the time they are added to the papermaking furnish. It is quite possible, and even to be expected, that subsequent events such as cross-linking will render the resins insoluble in water. Further some resins are soluble only under specific conditions, such as over a limited pH range. Wet strength resins are generally believed to undergo a cross-linking or other curing reactions after they have been deposited on, within, or among the papermaking fibres. Cross-linking or curing does not normally occur so long as substantial amounts of water are present.

Of particular utility are the various polyamide-epichlorohydrin resins. These materials are low molecular weight polymers provided with reactive functional groups such as amino, epoxy, and azetidinium groups. The patent literature is replete with descriptions of processes for making such materials, including US-A-3 700 623, issued to Keim on October 24th 1972, and US-A-3 772 076, issued to Keim on November 13th 1973.

Polyamide-epihydrochlorin resins sold under the trademarks Kymene 557H and Kymene LX by Hercules Inc. of Wilmington, Delaware, are particularly useful in this invention. These resins are generally described in the aforementioned patents to Keim.

Base-activated polyamide-epichlorohydrin resins useful in the present invention are sold under the Santo Res trademark, such as Santo Re 31, by Monsanto Company of St. Louis, Missouri. These types of materials are generally described in US-A-3 855 158 issued to Petrovich on December 17th 1974; US-A-3 899 388 issued to Petrovich on August 12th 1975; US-A-4 129 528 issued to Petrovich on December 12 1978; US-A-4 147 586 issued to Petrovich on April 3rd 1979; and US-A-4 222 921 issued to Van Eenam on September 16th 1980.

Other water-soluble cationic resins useful hererin are the polyacrylamide resins such as those sold under the Parez trademark, such as Parez 631NC, by American Cyanamid Company of Sandford, Connecticut. These materials are generally described in US-A-3 556 932 issued to Coscia et al on January 19th 1971; and US-A3 556 933 issued to Williams et al on January 19th 1971.

Other types of water-soluble resins useful in the present invention include acrylic emulsions and anionic styrene-butadiene latexes. Numerous examples of these types of resins are provided in US-A3 844 880. Meisel Jr et al, issued October 29th 1974. Still other water-soluble cationic resins finding utility in this invention are the urea formaldehyde and melamine formaldehyde resins. These polyfunctional, reactive polymers have molecular weights on the order of a few thousand. The more common functional groups include nitrogen containing groups such as amino groups and methylol groups attached to the nitrogen. Although less preferred, polyethylenimine type resins find utility in the present invention.

More complete descriptions of the aforementioned water-soluble resins, including their manufacture, can be found in TAPPI Monograph Series No. 29, "Wet Strength in paper and Paperboard, Technical Association of the Pulp and Paper Industry (New York; 1965).

Temporary wet strength agents, such as modified starch may also, optionally, be used. Combinations of permanent and temporary wet strength agents may be used.

The present invention may contain dry strength chemical agents, preferably at levels up to 3% by weight, more preferably at least 0.1% by weight, on a dry fiber weight basis. A highly preferred dry strength chemical agent is carboxymethyl cellulose. Other suitable dry strength chemical agents include polyacrylamide (such as combinations of Cypro™ 514 and Accostrength™ 711 produced by American Cyanamid of Wayne, N.J.); starch (such as corn starch or potato starch); polyvinyl alcohol (such as Airvol™ 540 produced by Air Products Inc. of Allentown, PA); guar or locust bean gums; and polyacrylate latexes. Suitable starch materials may also include modified cationic starches such as those modified to have nitrogen containing groups such as amino groups and methylol groups attached to nitrogen, available from National Starch and Chemical Company (Bridgewater, NJ).

Chemical softening compositions, comprising chemical debonding agents are optional components of the present invention. US-A-3 821 068, issued June 28th, 1974 teaches that chemical debonding agents can be used to reduce the stiffness, and thus enhance the softness, of a paper tissue web. US-A-3 554 862, issued on January 12th 1971 discloses suitable chemical debonding agents. These chemical debonding agents include quaternary ammonium salts.

Preferred chemical softening compositions comprise from about 0.01% to about 3.0% of a quaternary ammonium compound, preferably a biodegradable quaternary ammonium compound; and from about 0.01% to about 3.0% of a

polyhydroxy compound; preferably selected from the group consisting of glycerol, sorbitols, polyglycerols having an average molecular weight of from about 150 to about 800 and polyoxyethylene glycols and polyoxypropylene glycols having a weight average molecular weight from about 200 to 4000. Preferably the weight ratio of the quaternary ammonium compound to the polyhydroxy compound ranges from about 1.0:0.1 to 0.1:1.0. It has been discovered that the chemical softening composition is more effective when the polyhydroxy compound and the quaternary ammonium compound are first premixed together, preferably at a temperature of at least 40°C, before being added to the papermaking furnish. Either additionally, or alternatively, chemical softening compositions may be applied to the substantially dry paper tissue web, for example by means of a printing process (N.B. all percentages herein are by weight of dry fibres, unless otherwise specified).

Examples of quaternary ammonium compounds suitable for use in the present invention include either unmodified, or mono- or di- ester variations of : well-known dialkyldimethylammonium salts and alkyltrimethyl ammonium salts. Examples include the di-ester variations of di(hydrogenated tallow)dimethyl ammonium methylsulphate and di-ester variations of di(hydrogenated tallow)dimethyl ammonium chloride. Without wishing to be bound by theory, it is believed that the ester moity(ies) lends biodegradability to these compounds. Commercially available materials are available from Witco Chemical Company Inc. of Dublin, Ohio, under the tradename "Rewoquat V3512". Details of analytical and testing procedures are given in WO95/11343, published on 27th April, 1995.

Examples of polyhydroxy compounds useful in the present invention include polyoxyethylene glycols having a weight average molecular weight of from about 200 to about 600, especially preferred is "PEG-400".

The paper tissue of the present invention may be made by common methods well-known to the person skilled in the art, such as by dewatering suitable pulp using, for example, one or more papermakers felts and/or belts. For the present invention conventional papermaking processes are preferred. Any process referred to herein as conventional is a paper-making process which does not comprise a step of through-air-drying. Alternatively, papermaking processes comprising a through-air-drying step can be utilised. Such processes are described in the patent literature referred to hereinafter with regard to through-air-dried tissue.

According to the present invention a paper tissue is provided from at least 2 plies which is thick but smooth and hence has a physiological surface smoothness parameter of less than 700 microns, preferably from 650 microns to 50 microns, more preferably from 650 microns to 300 microns and in combination has a caliper per ply of more than 0.09 mm, preferably from 0.09 mm to 0.5 mm, more preferably from 0.1 mm to 0.2 mm. According to the present invention it has been found that the caliper per ply is a relevant parameter in expressing how much caliper is provided in a cost effective way, i.e. per one ply. Any combination of ranges given above for the PSS parameter and the caliper per ply is within the scope of the present invention.

Preferably the paper tissue has a low ratio of caliper per ply over the PSS parameter, the ratio being lower than 6500 microns/mm, more preferably lower than 5000 microns/mm, yet more preferably lower than 3000 microns/mm.

A paper tissue according to the present invention has a first and a second surface, the surfaces being mutually opposed to each other, and a thickness

orthogonal to the first and second surface. The thickness is also referred to a caliper of the tissue.

The caliper of a tissue according to the present invention is preferably from 0.1 mm to1 mm, more preferably from 0.2 mm to 0.5 mm.

Moreover, a paper tissue according to the present invention has preferably a wet burst strength greater than 100 g, preferably from 150 g to 500 g, more preferably from 250 g to 400 g.

In one preferred embodiment of the present invention a paper tissue is provided from two plies. In one preferred two-ply embodiment of the present invention one ply is provided from a calendered paper tissue while the other ply is provided from a textured, preferably embossed paper tissue. Without wishing to be bound by theory, the following is believed: The embossing increases the overall caliper of the product and thereby also the caliper per ply. The calendering typically increases the smoothness of the respective ply and thereby a surface is provided with a low PSS parameter.

"Calendered", as used herein, comprises high pressure calendering, high pressure calendering denoting a calendering using a pressure per contact length of at least 3 kN/m, more preferably 5 kN/m to 50 kN/m, yet more preferably 10 kN/m to 25 kN/m. Calendering with higher pressure increases the smoothness of paper tissue and hence decrease the PSS parameter.

In accordance with the present invention preferably single plies are subjected to calendering, but alternatively several plies at a time or a whole multi-ply paper tissue may be calendered.

Alternatively other techniques known is the art to increase the smoothness of paper tissue can be used, such as the selection of appropriate Fourdrinier wires,

felts, and belt in the dewatering stages, further creping under the appropriate conditions (glue content, glue composition, blade impact angle, creping aides). Further surface treatments, for example with a lotion, as disclosed hereinafter, are within the scope of the present invention.

"Textured", as used herein, for a paper tissue refers to a paper tissue which is either through-air-dried, or bulk embossed, or comprises regions of different basis weights or is dried with a texture or creped under the appropriate conditions (glue content, glue composition, blade impact angle, creping aides), as explained hereinafter.

"Bulk embossed", as used herein, refers to an embossing which increases the caliper of the paper tissue by at least 5%, preferably 15%, more preferably 25% as compared to the caliper of the paper tissue before the bulk embossing. Preferably bulk embossing provides a pattern of embossed and unembossed areas, which is imparted to only a limited number of plies of the multi-ply paper tissues of the present invention in one process step, preferably only to one ply in one process steps. The outermost embossed areas of the pattern preferably extends over at least 75%, preferably 85%, more preferably 95% of the total surface area of the embossed paper plies. Knob to knob embossing is well known in the art as illustrated by commonly assigned U.S. Patent No. 3,414,459, issued Dec. 3, 1968 to Wells. The texture may also be imparted to the paper tissue by nested embossing as illustrated by U.S. Patent No. 4,320,162, issued Mar. 16, 1982 to Schulz et al. Alternatively, the texture may be imparted to the paper tissue by dual ply lamination embossing as illustrated by commonly assigned U.S. Patent No. 5,468,323, issued Nov. 21, 1995 to McNeil. Preferably such bulk embossing pattern is provided by steel-to-steel knob-to-knob embossing, the knobs preferably having an elliptical cross section and a height in the range of 0.5 mm to 3 mm, more preferably in the range of 1 mm to 2 mm. Preferably the bulk embossing provides a ratio of embossed areas to

unembossed areas of from 1:1 to 1:20, more preferably 1:2 to 1:15, yet more preferably a ratio of from 1:5 to 1:10.

To obtain the texture on either, or both, of the first and second opposed surfaces, the tissue may be alternatively through-air-dried. Through-air-dried tissue is disclosed in commonly assigned U.S. Patent Nos. 4,529,480, issued July 16, 1985 to Trokhan; 4,637,859, issued Jan. 20, 1987 to Trokhan; 5,364,504, issued Nov. 15, 1994 to Smurkoski et al.; 5,529,664, issued June 25, 1996 to Trokhan et al.; 5,679,222 issued Oct. 21, 1997 to Rasch et al.; 5,714,041 issued Feb. 3, 1998 to Ayers et al.; 5,906,710, issued May 25, 1999 to Trokhan. Alternatively, the paper tissue may be through-air-dried and made as disclosed in U.S. Patent Nos. 5,429,686 issued July 4, 1995 to Chiu et al. and 5,672,248 issued Sept. 30, 1997 to Wendt et al.

Alternatively, the paper tissue may be textured by providing various regions of differing basis weights, so that a multi-basis weight paper tissue is presented. Multi-basis weight paper tissue is disclosed in commonly assigned U.S. Patents Nos. 5,245,025, issued Sept. 14, 1993 to Trokhan et al.; 5,527,428 issued June 18, 1996 to Trokhan et al.; 5,534,326 issued July 9, 1996 to Trokhan et al.; 5,654,076, issued Aug. 5, 1997 to Trokhan et al.; 5,820,730, issued Oct. 13, 1998 to Phan et al.; 5,277,761, issued Jan. 11, 1994 to Phan et al.; 5,443,691, issued Aug. 22, 1995 to Phan et al.; 5,804,036 issued Sept. 8, 1998 to Phan et al.; 5,503,715, issued Apr. 2, 1996 to Trokhan et al.; 5,614,061, issued March 25, 1997 to Phan et al.; 5,804,281 issued Sept. 8, 1998 to Phan et al.; and 5,900,122 issued May 4, 1999 to Huston.

Alternatively, the paper may be conventionally dried with a texture, for example, according to commonly assigned U.S. Patent Nos. 5,549,790, issued Aug. 27, 1996 to Phan; 5,556,509, issued Sept. 17, 1996 to Trokhan et al.; 5,580,423, issued Dec. 3, 1996 to Ampulski et al.; 5,609,725, issued Mar. 11, 1997 to Phan; 5,629,052 issued May 13, 1997 to Trokhan et al.; 5,637,194, issued June 10,

1997 to Ampulski et al.; 5,674,663, issued Oct. 7, 1997 to McFarland et al.; 5,693,187 issued Dec. 2, 1997 to Ampulski et al.; 5,709,775 issued Jan. 20, 1998 to Trokhan et al.; 5,776,307 issued Jul. 7, 1998 to Ampulski et al.; 5,795,440 issued Aug. 18, 1998 to Ampulski et al.; 5,814,190 issued Sept. 29, 1998 to Phan; 5,817,377 issued October 6, 1998 to Trokhan et al.; 5,846,379 issued Dec. 8, 1998 to Ampulski et al.; 5,855,739 issued Jan. 5, 1999 to Ampulski et al.; 5,861,082 issued Jan. 19, 1999 to Ampulski et al., 5,871,887 issued Feb. 16, 1999 to Trokhan et al.; 5,897,745 issued April 27, 1999 to Ampulski, et al.; and 5,904,811 issued May 18, 1999 to Ampulski et al.

In a highly preferred embodiment of the present invention a paper tissue is provided from three plies. Preferably at least one ply is calendered and at least one ply is textured, preferably embossed. More preferably two plies are calendered and an embossed preferably is disposed there-between. This particular embodiment has the advantage of providing a smooth surface to the user on either side. Alternative embodiments of the present invention are for example those with any number of textured, preferably embossed plies disposed between two outer calendered plies, one of these being a four ply paper tissue with two embossed plies disposed between two calendered plies.

When two or more plies of paper tissue are combined to form the paper tissue, the plies may, optionally, be attached together by means, for example, of gluing or embossing, herein referred to as “attachment embossing”. Gluing is less preferred because it tends to result in a stiffer, less soft product.

“Attachment embosssing”, as used herein, refers to an embossing by which all plies of a multi-ply tissue according to the present invention are embossed in one process step. Preferably the attachment embossing does not or at least not to a large extent affect the smoothness of any calendered ply. Therefore, preferably the tissue has an unembossed surface over a major part of the surface area of the tissue, preferably on the first and the second surface. As used herein, this

means that the tissue has one or more regions not comprising an attachment embossing and, optionally, one or more regions comprising an attachment embossing, and that the region not comprising an attachment embossing is at least 50%, and as much as 99%, of the surface area of the tissue. Most commonly the regions comprising an attachment embossing lie close to the edge of the tissue (for example along two or four edges); and a regions comprising an attachment embossing may also be used for decorative purposes (for example to create a pattern or to spell out a logo or brand name). The region not comprising an attachment embossing is the continuous region between and/or around the region comprising an attachment embossing. Attachment embossing is preferably done by steel-to-steel pin-to-pin embossing.

If glue is to be used to attach the plies of a multi-ply paper tissue, according to the present invention the glue is preferably applied unevenly over the surfaces of the plies to be attached. Therefore the glue is preferably not applied by means such a spraying nozzle, since such nozzles apply the glue evenly with no preference for particular areas of the tissue, even when the glue is applied as to form a discontinuous net.

A textured, preferably embossed, paper tissue comprises raised portions. In one preferred embodiment of the present invention the glue to applied only to these raised portions of the paper tissue. Since primarily these raised portions are in context with adjacent plies, in particular with adjacent calendered plies, application of glue to these raised portions is sufficient as to ensure good attachment, but avoids the application of an amount of glue, which easily impart stiffness to the paper tissue.

One preferred method of applying glue to a tissue ply is to apply the glue by print rolling. Alternatively glue may be applied by melt blowing, so as to form areas of preferential glue applications, e.g. strips of glue.

The paper tissue and preferably one or both surfaces, most preferably both surfaces of the tissue may, optionally, be further treated with a lotion. A lotion can contribute to the smoothness of the paper tissue, and hence decrease its PSS parameter.

The lotion may comprise softening/debonding agents, emollients, immobilizing agents and mixtures thereof. Suitable softening/debonding agents include quaternary ammonium compounds, polysiloxanes, and mixtures thereof. Suitable emollients include propylene glycol, glycerine, triethylene glycol, spermaceti or other waxes, petrolatum, fatty acids, fatty alcohols and fatty alcohol ethers having from 12 to 28 carbon atoms in their fatty acid chain, and mixtures thereof. Suitable immobilizing agents include polyhydroxy fatty acid esters, polyhydroxy fatty acid amides and mixtures thereof. Other optional components include perfumes, antibacterial actives, antiviral actives, disinfectants, pharmaceutical actives, film formers, deodorants, opacifiers, astringents, solvents and the like. Particular examples of lotion components include camphor, thymol and menthol.

A process according to the present invention may utilise any paper tissue made by any method known in the art, preferred methods are disclosed herein.

The process comprises a step of supplying the paper tissue by unwinding at least two plies, preferably three plies, from a corresponding number of patent rolls. The process comprises a further step of applying a texture pattern to at least one ply, preferably by bulk embossing as disclosed herein. The process also comprises a step of high pressure calendering at least one ply using calendering pressures as disclosed herein. Further the process comprises a step of juxtaposing said plies to form a multi-ply tissue.

A more preferred process further comprises a step of applying lotion to the plies, which will form the outer plies of the mulit-ply paper tissue, most preferably the lotion is applied only to the surfaces which will form the outer surfaces of the

multi-ply tissue. Moreover a preferred process comprises a step of attaching the juxtaposed plies by embossing, referred to and described above as attachment embossing. Optionally the present process may also comprise the application of glue, preferably only to the raised portions of the textured plies.

Test Methods

Caliper is measured according to the following procedure: The tissue paper is preconditioned at 21° to 24°C and 48 to 52 percent relative humidity for two hours prior to the caliper measurement. If the caliper of toilet tissue is being measured, 15 to 20 sheets are first removed and discarded. If the caliper of facial tissue is being measured, the sample is taken from near the center of the package. The sample is selected and then conditioned for an additional 15 minutes.

Caliper of the multi-ply paper tissue, as used herein, is the thickness of the paper when subjected to a compressive load of 14.7 g/cm^2. Preferably, caliper is measured using a low load Thwing-Albert micrometer, Model 89-11, available from the Thwing-Albert Instrument Company of Philadelphia, Pa. The caliper per ply is the total caliper of the multi-ply paper tissue divided by the number of plies comprised. For a single ply tissue caliper per ply and caliper are identical. Decorated regions, perforations, edge effects, etc., of the tissue should be avoided if possible.

The wet burst strength is measured using an electronic burst tester and the following test conditions. The burst tester is a Thwing-Albert Burst Tester Cat. No. 177 equipped with a 2000 g load cell. The burst tester is supplied by Thwing-Albert Instrument Company, Philadelphia, PA 19154, USA.

Take eight paper tissues and stack them in pairs of two. Using scissors, cut the samples so that they are approximately 228 mm in the machine direction and

approximately 114 mm in the cross-machine direction, each two finished product units thick.

First age the samples for one to two hours by attaching the sample stack together with a small paper clip and "fan" the other end of the sample stack to separate the sheets, this allows circulation of air between them. Suspend each sample stack by a clamp in a 107°C (± 3°C) forced draft oven for 5 minutes (± 10 seconds). After the heating period, remove the sample stack from the oven and cool for a minimum of three minutes before testing.

Take one sample strip, holding the sample by the narrow cross direction edges, dipping the centre of the sample into a pan filled with about 25mm of distilled water. Leave the sample in the water four (4.0 ± 0.5) seconds. Remove and drain for three (3.0 ± 0.5) seconds holding the sample so the water runs off in the cross direction. Proceed with the test immediately after the drain step. Place the wet sample on the lower ring of the sample holding device with the outer surface of the product facing up, so that the wet part of the sample completely covers the open surface of the sample holding ring. If wrinkles are present, discard the sample and repeat with a new sample. After the sample is properly in place on the lower ring, turn the switch that lowers the upper ring. The sample to be tested is now securely gripped in the sample holding unit. Start the burst test immediately at this point by pressing the start button. The plunger will begin to rise. At the point when the sample tears or ruptures, report the maximum reading. The plunger will automatically reverse and return to its original starting position. Repeat this procedure on three more samples for a total of four tests, i.e., 4 replicates. Report the results, as an average of the four replicates, to the nearest gram.

For the physiological surface smoothness measurement, which reports the PSS parameter, a sample of the paper tissue is selected which avoids wrinkles, tears, perforations, or gross deviations from macroscopic monoplanarity. The sample is

conditioned at 22 to 24°C and 48 to 52% relative humidity for at least two hours prior to testing. The sample is placed on a motorised table and magnetically secured in place. Either face of the sample may be selected for the measurement, provided all traces are taken from the same face.

Physiological surface smoothness is obtained by scanning the paper tissue sample in any direction with a profilometer to obtain the Z-direction displacement as a function of distance. The Z-direction displacement is converted to an amplitude versus frequency spectrum by a Fourier Transform. The spectrum is then adjusted for human tactile response using a series of filters. The peak heights of the filtered amplitude frequency curve are summed from 0 to 10 cycles per millimetre to give the result.

The paper tissue sample is approximately 100 millimetres x 100 millimetres in size and mounted on a motorised table. While any suitable table will suffice, a table with surface tester model KES-FB-4NKES-SE, available from Kato Tech Company Limited of Koyota, Japan, or a CP3-22-01 DCI Mini Precision table using a NuStep 2C NuLogic Two Axis Stepper Motor Controller in the closed loop control mode have been found suitable. The table has a constant drive motor which travels at the rate of 1 millimetre per second. The sample is scanned 30 millimetres in the forward direction transversely indexed one millimetre, then reversed. Data are collected from the centre 26 millimetres of the scan in both the forward and reverse directions. The first and last 2 millimetres of each scan are ignored and not used in the calculations.

The profilometer has a probe with a tip radius of 2.54 microns and an applied force of 0.20 grams. The gauge range is calibrated for a total Z-direction displacement of 3.5 millimetres. Over the scan distance of the sample, the profilometer senses the Z-direction displacement of the stylus in millimetres. The output voltage from the gauge controller is digitised at a rate of at least 20 points per second. Over the entire 26 millimetre scan range, 512 pairs of time surface

height data points are obtained for both the forward and reverse directions of a scan. The profilometer is mounted above the sample table such that the surface topography can be measured. A suitable profilometer is a EMD 4320 WI Vertical Displacement Transducer, having an EPT 010409 stylus tip, and an EAS 2351 Analog Amplifier. This equipment is obtainable from Federal Products of Providence, Rhode Island.

The digitised data pairs are imported into a standard statistical analysis package for further analysis. Suitable software analysis packages included SAS of Cary, North Carolina, and preferably LabVIEW Instrument Control Software 3.1 available from National Instruments of Austin, Texas. When using the LabVIEW software, raw data pairs linking surface height and time from the individual scans are centered about the mean using the Mean.vi analysis tool in the LabVIEW software. The 512 data points from each of the 16 traces are converted to 16 amplitude spectra using the Amplitude and Phase Spectrum.vi tool. Each spectrum is then smoothed using the method described by the PROC Spectra Method of the SAS software. LabVIEW smoothing filter values of 0.000246, 0.000485, 0.00756, 0.062997, 0.00756, 0.000485, 0.000246 are utilized. The output from this tool is taken as the Amp Spectrum Mag (vrms).

The amplitude data are then adjusted for human tactile response using a series of frequency filters designed from Verrillo's data on vibrotactile thresholds as a function of vibration frequency as set forth in the Journal of Acoustical Society of America, in the article entitled "Effect Of Contactor Area On The Vibrotactile Threshold", Vol. 35, 1962 (1963). The aforementioned data are reported in a time domain as cycles per second and converted to the spatial domain in cycles per millimetre. The conversion factor and filter values are found in the procedure set forth in the 1991 International Paper Physics Conference, TAPPI Book 1, more particularly the article entitled "Methods For The Measurement Of The Mechanical Properties Of Paper tissue" by Ampulski, et al., and found at page 19, utilizing the specific procedure set forth at page 22 entitled "Physiological

Surface Smoothness". The response from the filters are set at 0 below the minimum threshold and above the maximum response frequencies and varies from 0 to 1 therebetween as described by the aforementioned Ampulski et al. article.

The physiologically adjusted frequency amplitude data are obtained by multiplying the amplitude spectra described above by the appropriate filter value at each frequency. A typical amplitude spectrum and filtered amplitude spectrum are illustrated in Fig. 5 of the aforementioned Ampulski et al. article. The Verrillo-adjusted frequency amplitude curve is summed point by point between 0 and 10 cycles per millimetre. This summation is considered to be the physiological surface smoothness. The eight forward and eight reverse physiological surface smoothness values thus obtained are then averaged and reported in microns.

Physiological surface smoothness measurements using the SAS software is described in commonly assigned U.S. Pat Nos. 4,959,125, issued Sept. 25, 1990 to Spendel; 5,059,282, issued Oct. 22, 1991 to Ampulski et al.; 5,855,738, issued Jan. 5, 1999 to Weisman et al., and 5,980,691, issued Nov. 9, 1999 to Weisman et al.

Either face of the tissue may be selected for the smoothness measurement, provided all traces are taken from the same face. If either face of the tissue meets any of the smoothness criteria set forth herein, the entire sample of the tissue is deemed to fall within that criterion. Preferably both faces of the tissue meet the above criteria.

Example

An aqueous slurry comprising 3% by weight of Nothern Softwood Kraft (NSK) fibres was prepared in a conventional re-pulper. The NSK slurry was refined

gently and a 2% solution of the permanent wet strength resin (Kymene™ 617) was added to the NSK stock pipe at a rate of 0.9% by weight of the total dry fibres. The absorption of the permanent wet strength resin onto the NSK fibres is enhanced by an in-line mixer. A 1% solution of the dry strength resin (carboxymethyl cellulose) is added to the NSK stock before the fan pump at a rate of 0.14% by weight of the total dry fibres. The NSK slurry was diluted to about 0.2% consistency at the fan pump.

A chemical softening composition was prepared comprising di-hard tallow diethyl ester dimethyl quaternary ammonium chloride and polyoxyethylene glycol, having an average molecular weight of 400 (PEG-400). The PEG-400 was heated to about 66°C, and the quat was dissolved into the molten PEG-400 so that a homogeneous mixture was formed.

An aqueous slurry comprising 3% by weight of eucalyptus fibres was prepared in a conventional re-pulper. A 1% solution of the chemical softening composition was added to the Eucalyptus stock pipe at a rate of 0.09% by weight of the total dry fibres. The Eucalyptus slurry was diluted to about 0.2% consistency at the fan pump. The 1% solution of the chemical softening composition was also added to the NSK slurry after post CMC addition and prior to dilution of the slurry to about 0.2% at the stock pump.

The two slurries were combined so that the ratio of NSK to eucalyptus fibres was 40:60 and the resulting slurry was deposited, by means of a single layer headbox onto a Fourdrinier wire to form an embryonic web. Dewatering occured through the Fourdrinier wire and was assisted by a deflector and vacuum boxes.

The embryonic web was transferred from the Fourdrinier wire, at a fibre consistency of about 20% at the point of transfer, to a conventional drying felt. The web was then transfered to the surface of a Yankee dryer with a sprayed creping adhesive comprising 0.25% aqueous solution of Polyvinyl Alcohol (PVA).

The fibre consistency was increased to an estimated 96% before dry creping the web with a doctor blade. The doctor blade had a bevel angle of about 25° and is positioned with respect to the Yankee dryer to provide an impact angle of about 81°. The Yankee dryer was operated at about 4 m/s and the dried, uncalendared paper was formed into 1ply rolls at a reel.

Three of these 1-ply rolls were taken to an off-line rewinding operation to form 3-ply rolls that were subsequently converted into a 3-ply tissue paper product, having overall dimension of about 210 mm square.

The 3-ply rolls were produced by simultaneously unwinding 3 of the 1-ply rolls, running the centre ply through a rubber to steel bulk embossing operation and rewinding the two unembossed outer plies with the embossed centre ply to form a 3-ply roll. For the centre ply embossing a smooth rubber roll was loaded against a patterned steel roll. The patterned steel roll has raised elliptical emboss knobs about 1.7mm deep having a major axis at the surface of about 2mm and a minor axis of about 1mm. The embossments are arranged in repeating pattern of concentric diamonds consisting of about 72 knobs in 900 square mm area.

The 3-ply roll was subsequently converted into a 3-ply tissue product. The three ply web was unwound and subjected to an embossing step before folding. The margin of the tissue paper product, extending about 15mm in from the edge was embossed following the process described in WO95/27429, published on 19th October 1995. The major part of the surface area of the tissue paper product (i.e. all of the surface area within the 15mm margin) was unembossed. The tissue was further decorated by embossing the brand name over a small area of the previously unembossed area and four decorative leaf patterns where embossed in the previously unembossed area was also added.

Lotion was printed on each of the outer surfaces of the 3-ply web via a two step application process before folding. The lotion was an aqueous solution of di-hard

tallow diethyl ester dimethyl quaternary ammonium chloride. The printing was accomplished by running the 3-ply web through two consecutive printing stations each consisting of an engraved anilox roll and a rubber backing roll pair.

The anilox roll was engraved to a cell volume of about 3 ml per square meter, and with supplied with lotion from a closed supply chamber designed to fill the engraved volume with lotion. A gap of 0.35mm was established between the anilox roll and backing roll, and the 3-ply web was run through this gap, transferring lotion to the surface touching the anilox roll. The web was then run through the second printing station with an identical anilox/rubber roll pair at a 0.35mm gap. The pairs were arranged such that the second anilox roll contacted the as yet unlotioned surface, transfering lotion to it. This arrangement transferred 0.45% active quat per dry weight of the finished 3- ply tissue.

The paper tissue obtained by the above described process had a basis weight of 54 g/m^2, a total caliper of 0.35 mm, a caliper per ply of 0.12 mm, a wet burst strength of 375 g and a PSS parameter of 620 micron.

A second example consists of substrate produced described above, in which the outer plies are run through a smoothing calendering roll. Calendering at 12 kN/m to 15 kN/m was found to further reduce the PSS parameter to about 500 to 450 microns.

WHAT IS CLAIMED IS:

1. A paper tissue, said paper tissue comprising at least two plies, said paper tissue having a caliper and a caliper per ply, said paper tissue further having a physiological surface smoothness parameter, characterised in that said physiological surface smoothness parameter is less than 700 microns and said caliper per ply is greater than 0.09 mm.

2. A paper tissue according to Claim 1, characterised in that said physiological surface smoothness parameter is from 650 microns to 100 microns.

3. A paper tissue according to any one of the preceding claims, characterised in that said caliper per ply is from 0.1 mm to 0.2 mm.

4. A paper tissue according to any one of the preceding claims, characterised in that the ratio of said physiological surface smoothness parameter to said caliper per ply is smaller than 5000 micron/mm.

5. A paper tissue according to any one of the preceding claims, said paper tissue having a wet burst strength, characterised in that said wet burst strength is from 150 g to 500 g.

6. A paper tissue according to any one of the preceding claims, said paper tissue having a wet burst strength, characterised in that said wet burst strength is from 250 g to 400 g.

7. A paper tissue according to any one of the preceding claims characterised in that it does not comprise through-air-dried paper.

8. A paper tissue according to any one of the preceding claims, characterised in that at least one of said plies comprises a bulk embossing and at least one of said plies does not comprise a bulk embossing.

9. A paper tissue according to Claim 8, characterised in that said at least one ply not comprising said bulk embossing is calendered.

10. A paper tissue according to any one of the preceding claims, characterised in that said paper tissue comprises at least three plies.

11. A paper tissue according to Claim 10, comprising two outer plies and at least one inner ply, characterised in that said outer plies do not comprise said bulk embossing and at least one of said inner plies comprises said bulk embossing.

12. A paper tissue according to Claim 11, characterised in that at least one of said outer plies is calendered.

13. A paper tissue according to any one of the preceding claims, characterised in that said bulk embossing is provided by a pattern of embossed and unembossed areas, characterised in that the ratio of said embossed areas to said unembossed areas is from 1:1 to 1:20.

14. A paper tissue according to any one of the preceding claims characterised in that at least one ply comprises a lotion.

15. A paper tissue according to any one of the preceding claims characterised in that said plies are not attached by adhesive.

16.A paper tissue according to any one of the preceding claims characterised in that said plies are attached by embossing.

17.A process for making the paper tissue according to any of the previous claims comprising the steps of :

- unwinding at least two plies, preferably three plies from a parent role
- applying a texture, preferably an embossing pattern, to at least one ply
- high pressure calendering at least one ply
- juxtaposing said plies to form a multi-ply tissue

18.A process according to Claim 15 which further comprises a step of applying a lotion to at least one of said calendered plies.

19.A process according to any one of the preceding claims which further comprises a step of embossing all of said plies.

20.A process of any one of the preceding claims which does comprise the application of a glue by print rolling.

INTERNATIONAL SEARCH REPORT

national Application No
PCT/US 00/32197

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 D21H27/30 D21F11/14

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

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 D21H D21F

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

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)
EPO-Internal

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
L	AMPULSKI R S ET AL: "METHODS FOR THE MEASUREMENT OF THE MECHANICAL PROPERTIES OF TISSUE PAPER" INTERNATIONAL PAPER PHYSICS CONFERENCE,XX,X, 1 January 1991 (1991-01-01), pages 19-30, XP000569858 cited in the application This document describes a way of measuring the "physiological surface smoothness parameter" to which it is referred in claim 1. page 22 -page 24 ---	
A	US 5 980 691 A (AMPULSKI ROBERT STANLEY ET AL) 9 November 1999 (1999-11-09) cited in the application --- -/--	

[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

° Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
13 March 2001	09. 04. 2001

Name and mailing address of the ISA	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Songy, O

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Form PCT/ISA/210 (second sheet) (July 1992)

INTERNATIONAL SEARCH REPORT

rnational Application No
PCT/US 00/32197

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication,where appropriate, of the relevant passages	Relevant to claim No.
A	US 5 855 738 A (LOUGHRAN SCOTT THOMAS ET AL) 5 January 1999 (1999-01-05) cited in the application	
A	US 5 728 268 A (LOUGHRAN SCOTT THOMAS ET AL) 17 March 1998 (1998-03-17)	
A	US 5 865 950 A (WEISMAN PAUL THOMAS ET AL) 2 February 1999 (1999-02-02)	
A	US 5 389 204 A (AMPULSKI ROBERT S) 14 February 1995 (1995-02-14) example 4	

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US 00/32197

Box I Observations where certain claims were found unsearchable (Continuation of item 1 of first sheet)

This International Search Report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☐ Claims Nos.:
because they relate to subject matter not required to be searched by this Authority, namely:

2. ☒ Claims Nos.: 1-20
because they relate to parts of the International Application that do not comply with the prescribed requirements to such an extent that no meaningful International Search can be carried out, specifically:
see FURTHER INFORMATION sheet PCT/ISA/210

3. ☐ Claims Nos.:
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

Box II Observations where unity of invention is lacking (Continuation of item 2 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

1. ☐ As all required additional search fees were timely paid by the applicant, this International Search Report covers all searchable claims.

2. ☐ As all searchable claims could be searched without effort justifying an additional fee, this Authority did not invite payment of any additional fee.

3. ☐ As only some of the required additional search fees were timely paid by the applicant, this International Search Report covers only those claims for which fees were paid, specifically claims Nos.:

4. ☐ No required additional search fees were timely paid by the applicant. Consequently, this International Search Report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:

Remark on Protest

☐ The additional search fees were accompanied by the applicant's protest.

☐ No protest accompanied the payment of additional search fees.

INTERNATIONAL SEARCH REPORT

Information on patent family members

rnational Application No
PCT/US 00/32197

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